

**Western Australian School of Mines**

**Efficiency of Coal Dust Suppression by Surfactants in Underground Mines:  
Investigation of the Role of Various Factors**

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**This thesis is presented for the Degree of  
Doctor of Philosophy – Mining & Metallurgy  
Of  
Curtin University**

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# Declaration

To the best of my knowledge and belief this thesis contains no material previously published by any other person except where due acknowledgment has been made.

This thesis contains no material which has been accepted for the award of any other degree or diploma in any university.

This research does not include any human and animal ethics issue.

Signature:

Date: 09/12/2019

*To my husband Zhao Fu and daughter Evelyn Fu*

*For their understanding, support and love*

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## **Abstract**

Coal dust is one of the primary health hazards that threaten coal workers in underground coal mines. Prolong exposure to high concentration coal dust can result in some fatal lung diseases. Coal workers pneumoconiosis (CWP), one of these lung diseases, which is irreversible and incurable, has killed thousands of coal miners since the fully mechanized mining method longwall mining was widely used around the world. Although the prevalence of CWP has been decreased substantially over the past decades because of many efforts on coal dust suppression, new CWP cases still have continued to occur and an increasing trend of CWP prevalence was observed in the recent years. For example, new CWP cases have been continuously reported in Australian since 2015, and the prevalence of CWP increased from 2.1% in 1990s to 3.2% in 2000s in the US. To minimize the coal dust hazard and provide a safety and health working for people in underground mines, regulations have been implemented to control the coal dust concentration limits in most of the mining nations, especially for respirable coal dust. Thus, an effective technology is required to lower the coal dust concentration to the standard limits to eliminate its health-related hazards.

It was considered that traditional coal dust suppression methods including ventilation and water only spray were not adequate to meet the regulated limits. Thus, considerable studies have been carried out to achieve an optimum method to reduce coal dust concentration. It was found that surfactants aided water spray can improve coal dust suppression efficiency compared with using plain water only although the improvements varied in different studies. Different research methods have been used to investigate the surfactant application on coal dust suppression, but some inconclusive or poorly reconcilable outcomes on these studies are still exist and consistent results are needed to provide effective coal dust control solutions for coal mining industry. To achieve this objective, this research applied experimental investigations and computational fluid dynamics modelling (CFD) to study the suppression by using surfactants aided water spray.

Providing a comprehensive study on coal dust suppression with surfactants aided water spray is the objective of this thesis. A literature review of identifying the health problems that caused by prolong exposure coal dust and the prevalence of CWP in the main mining countries, exploring the surfactant mechanisms in coal dust suppression process and summarising the commonly used static and dynamic methods for accessing surfactants was presented firstly. Then, a series of studies on determining the most cost-effective and easiest-operational static method and accessing the impacts of various factors including surfactant type, surfactant concentrations, the addition of salts, coal rank, coal particle size, coal sample size, temperature

and other factors on coal dust suppression by surfactants was carried out. Lastly, CFD simulation was applied to investigate the coal dust distribution characteristic in the wind tunnel and the dynamic method wind tunnel tests were selected to assess the effectiveness of surfactants on coal dust suppression. Experimental design method, for example, single comparative design, was employed in this study in order to obtain more reliable experimental data.

This research summarised both static and dynamic methods to evaluate the surfactants' effectiveness on coal dust suppression and determined the easiest-operational and most cost-effective method. This might help to provide a guidance for the coal mining industry to select the method for accessing surfactants. The performance of different type surfactants with various concentrations on coal dust control, and the impacts of various factors on surfactants' effectiveness were investigated and compared, the results might be valuable for the coal mining industry to make strategies on suppressing coal dust with surfactants aided water spray.

## List of publications included as part of the thesis

Four published SCI journal papers and two draft papers are incorporated in this thesis. The draft papers have been finished writing by Yinping Chen and currently are under reviewed by Dr. Guang Xu. The copyright of these published papers was checked, and they are appropriate to be reused in this thesis. Two conference papers were published but were not incorporated in this thesis. The details of these papers are listed as follows:

### Published Journal papers

1. Chen. Y, Xu, G., Albjanic B., (2017). Evaluation of SDBS surfactant on coal wetting performance with static methods: preliminary laboratory tests. *Energy Sources, Part A: Recovery Utilization and Environmental Effects*, 39(23), 2140-2150.  
<https://doi.org/10.1080/15567036.2017.1403503>
2. Xu, G., Chen. Y., Eksteen, J., Xu, J., (2018). Surfactant-aided coal dust suppression: A review of evaluation methods and influencing factors. *Science of The Total Environment*, 639, 1060-1076. <https://doi.org/10.1016/j.scitotenv.2018.05.182>
3. Chen. Y., Xu, G., Huang J., Eksteen J, Liu X, Zhao Z., (2019). Characterization of coal particles wettability in surfactant solution by using four laboratory static tests. *Colloids and Surface A: Physicochemical and Engineering Aspects*, 567, 304-312.  
<https://doi.org/10.1016/j.colsurfa.2019.01.068>
4. Chang, P., Chen, Y., Xu, G., Huang, J., Ghosh, A. and Liu, W.V., (2019). Numerical study of coal dust behaviours and experimental investigation on coal dust suppression efficiency of surfactant solution by using wind tunnel tests. *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects*, 1-16.  
<https://doi.org/10.1080/15567036.2019.1639855>

### Published conference papers

1. Chen. Y., Xu. G., Kuruppu. M., Albjanic. B., (2015). Application of Box-Wilson experimental design method for underground mine's respiratory coal dust studies. *The*

*15<sup>th</sup> North American Mine Ventilation Symposium. 21-25 June 2015. Blacksburg, Virginia, USA.*

2. Chen. Y., Xu. G., Wang. R., Albijanic. B., (2017). Study on coal wettability for dust suppression in the presence of surfactants by sink test. *The Australian Mine Ventilation Conference. 28 -30 August 2017. 105-109. Australia*

## **Draft papers**

1. Chen. Y., Xu. G., Huang. J., Eksteen, J., (2019). Characterized of coal wettability in presence of surfactants and electrolytes additions.
2. Chen. Y., Xu. G., Huang. J., Eksteen, J., Albijanic. B., (2019). Research on three type surfactants adsorption performance on different rank coal particles.



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## List of Acronyms

ANOVA	Analysis of Variance
ASTM	American Society for Testing and Materials
CFAD	Coconut Fatty Acid Diethanolamide
CFD	Computational Fluid Dynamics
CMC	Critical Micelle Concentration
CMDLD	Coal Mine Dust Lung Diseases
CTAB	Trimethylammonium Bromide
CWP	Coal Worker's Pneumoconiosis
HLB	Hydrophile-Liophile Balance
MSHA	Mine Safety and Health Administration
NIOSH	National Institute for Occupational Safety and Health
OEL	Occupational Exposure Limit
PEL	Permissible Exposure Limit
REL	Recommended Exposure Limit
SDBS	Sodium Dodecyl Benzenesulfonate
SDS	Sodium Dodecyl Sulphate
SD2ES	Sodium Di (2-ethylhexyl) Sulfosuccinate
XPS	X-ray photoelectron spectroscopy

# **1 Chapter 1**

## **1.1 Research background**

## **1.2 Research objectives**

## **1.3 Thesis outline**

## 1.1 Research background

Some lung diseases have been a safety threat for mining workers since mining started. The fully mechanized coal mining method of longwall mining has been used widely in the industry since 1970s due to its higher production rates and more cost-effective in operation (Peng, 2006). The high concentration of coal dust in underground coal mines caused by the mechanized mining method resulted in the high prevalence of these lung diseases. Studies have indicated that coal miners at the risk of some potentially fatal coal mine dust lung diseases (CMDLD) including coal worker's pneumoconiosis (CWP), tuberculosis, emphysema, chronic bronchitis, chronic obstructive pulmonary disease and other lung disease due to prolong the exposure to excessive amounts of coal dust particles (Ayoglu, Acikgoz, Tutkun, & Gebedek, 2014b; DE Pollock, Potts, & Joy, 2010; Ross & Murray, 2004).

Among these lung diseases, CWP was commonly observed in coal miners and it contributed to a high mortality rate in last decades. CWP was first recognized as a disease of British coal mines in the 1600s. (Joy, Colinet, & Landen, 2012; Laney & Attfield, 2010; Reynolds, Blackley, Laney, & Haldin, 2017). Although many efforts have been made on coal dust suppression and the prevalence of CWP has decreased substantially over the past decades, new CWP cases still continue to occur and the prevalence of CWP in some mining countries even shown a rise in last few years. For example, it was believed that CWP was considered has been eradicated in Australia until ten coal miners were diagnosed with CWP in 2016, and it was also reported that among the 150,000 unprocessed chest X-ray cases, 1,000 coal miners could have CWP (Zosky et al., 2016). In the U.S., the prevalence of CWP dropped from 6.5% in the 1970s to 2.1% in the 1990s, but it increased to 3.2% in the 2000s. China as the largest coal production and consumption country, the prevalence of CWP was 6.02% in the 2010s and still have thousands of coal miners diagnosed with CWP every year in last decade (G. Xu, Chen, Eksteen, & Xu, 2018). All these numbers confirmed the necessity of lowering coal dust concentration and providing a health and safety working conditions for coal miners in underground mines.

To reduce the risk of developing these lung diseases for coal miners, most mining nations have implemented regulations to control coal dust concentration under a permissible exposure limit (PEL) or occupational exposure limit (OEL). For example, the regulated limit of exposure respirable coal dust was reduced from 3 mg/m<sup>3</sup> to 2.5 mg/m<sup>3</sup> in Australia in 2018. In the U.S. a PEL of 2 mg/m<sup>3</sup> for respirable coal dust was set up in 1972 and it was further reduced to 1.5 mg/m<sup>3</sup> in 2016 by the Mine Safety and Health Administration (MSHA), and the National Institute for Occupational Safety and Health (NIOSH) has adopted a Recommended Exposure Limit (REL) of 1 mg/m<sup>3</sup> since 1995 (G. Xu, Chen, et al., 2018).

It was believed that ventilation and plain water spray as two traditional methods for suppressing coal dust in underground coal mines were not adequate to lower the coal dust concentration to meet the limits (G. Xu, Chen, et al., 2018). Thus, the research focus on surfactant aided water spray coal dust suppression technology has received increasing attention in last decades. Studies have indicated that surfactants enhanced the coal dust suppression efficiency with various improvement in different research. Although a large number of investigations have been conducted with different research methods, some results are still poorly reconcilable, and some inconsistent outcomes needed to be demonstrated to provide the coal mining industry with effective coal dust suppression solutions. Thus, a simple operation and cost effective method to evaluate the performance of surfactants on lower coal dust concentration is required to be determined, and an optimum surfactant with optimum concentration is needed as a guidance for providing a safety working environment for the coal mining industry.

## **1.2 Research objectives**

It is challenging for the mining industry to lower the coal dust concentration to meet the Permissible Exposure Limit (PEL) thus put the coal miners under the threat of several lung diseases especially CWP. To reduce the coal dust concentration to the recommended exposure limit and provide a less health-related hazards working environment is a necessity for mining industry. The objectives of this research are stated as follows:

The first purpose is to identify health issues that caused by coal dust and systematically review and analysis the considerable research work that have been carried out in the last decades. The specific aims are listed as follows:

- (1) To identify the problems of lung diseases that caused by coal dust, describe the prevalence of CWP, and state the regulations implemented for coal dust concentrations.
- (2) To explore the surfactant mechanisms in coal dust suppression process.
- (3) To summarise the commonly used static and dynamic methods for accessing surfactants.
- (4) To determine the factors that affect the surfactants' effectiveness.

The second objective is to access the methods and determine the most cost-effective and easiest-operational static method, and determine the optimum surfactant with optimum concentration.

- (1) To determine the most cost-effective and easiest-operational static method in terms of accessing coal wettability.

- (2) To assess the impacts of surfactant type, surfactant concentration, the addition of salts on the surfactant effectiveness.
- (3) To evaluate the impacts of the coal rank, coal particle size, coal sample size, temperature and other factors on coal dust suppression by surfactants.
- (4) To study the adsorption of different type surfactants on coal dust particles with various properties.

The third objective is to use CFD simulation to investigate the coal dust movement in the wind tunnel, and to assess the effectiveness of surfactants on coal dust suppression.

- (1) To investigate the coal dust distribution characteristic in the wind tunnel by using the CFD modelling.
- (2) To study the improvement of coal dust suppression in presence of surfactants by employing the wind tunnel test.

### **1.3 Thesis outline**

This thesis consists of eight chapters, the relationship of these chapters and the description of each chapter are as follows:

Chapter 1 states the background and objectives of this research. The threat of coal dust hazard to coal miners, the prevalence of CWP, the standards or limits of coal dust concentration in coal mines, the limitations of traditional coal dust control methods and the development of the surfactant-aided coal dust suppression method are presented. The research objectives of this thesis are described as well as its structure.

Chapters from chapter 2 to chapter 7 are presented by papers that have been published, submitted or prepared for submission. The status of each paper can be found at the start of each chapter.

Chapter 2 provides a comprehensive review literature review on previous research about coal dust suppression by using surfactants. The commonly used static and dynamic test methods to evaluate surfactants' effectiveness were summarized, and the factors affect surfactants' effectiveness and the applications of surfactants on coal dust control on mining industry are critically reviewed.

Chapter 3 presents a preliminary experimental study with using a wide surfactant concentration range for the static measurements, which helps to determine the potential optimal surfactant concentration range for the followed research. Chapter 4 systematically analyse the reliability of four static tests includes the contact angle measurement, the sink test, the capillary rise test

and the drop penetration test to characterize the coal wettability. Based on the results of Chapter 4, Chapter 5 used the most reliable measurement to study the impacts of surfactant type, surfactant concentration, coal dust particle properties and the electrolytes addition to surfactants on improving the coal wettability.

Chapter 6 demonstrates the surfactant adsorption on the coal dust particles via the zeta potential test and the adsorption measurement. The factors includes surfactants properties and coal dust properties that affect surfactant adsorption on coal dust particles were both investigated.

In Chapter 7, coal dust distribution characteristics and air flow behaviours in the wind tunnel were investigated by employing CFD modelling. Based on the CFD simulation results, a series of the wind tunnel experiments were conducted to evaluate the effectiveness of surfactants on reducing coal dust concentrations.

Chapter 8 refers as a conclusion section that highlighted the achievements and summarised the main research findings. Also, the limitations of this research and the recommendations are stated to guide the future work.

## 2 Chapter 2

### **Surfactant-aided coal dust suppression: a review of evaluation methods and influencing factors**

The work in this chapter has been published on the journal of Science of the Total Environment. It was entirely written by Yinping Chen and reviewed by Dr. Guang Xu, Professor Jacques Eksteen and Professor Jailin Xu.

Please cite this paper as:

Xu, G., Chen. Y., Eksteen, J., Xu, J., (2018). Surfactant-aided coal dust suppression: A review of evaluation methods and influencing factors. *Science of The Total Environment*, 639, 1060-1076. <https://doi.org/10.1016/j.scitotenv.2018.05.182>



This chapter provides a comprehensive review literature review on previous research about coal dust suppression by using surfactants. In this chapter, a detailed summary on the commonly used static and dynamic test methods to evaluate surfactants' effectiveness is presented. Also, the factors affect surfactants' effectiveness and the applications of surfactants on coal dust control in mining industry is critically reviewed.

## **Abstract**

There is an increasing trend in the occurrence of coal worker's pneumoconiosis even in developed countries such as the US and Australia who have believed such an issue have been well controlled in the past. Water spray is one of the most commonly applied methods for underground coal mines dust control, and research have shown the dust suppression efficiency can be greatly improved by adding surfactants. However, the literature appears to show inconsistent results that do not provide the coal mining industry with a clearly effective solution. The breakthrough in this field relies on the achievements in prior work, but an up-to-date critical review was not found. By critically reviewing prior studies, this paper highlights the advances in the surfactant-aided coal dust suppression technology. Firstly, the surfactant chemical structure, surfactant type and mechanism of surfactant adsorption were explained. Secondly, the commonly used surfactant efficiency evaluation methods were described. This is important for producing comparable and reproducible results. After that, key aspects of the influencing factors were discussed, which are essential for developing effective and robust dust suppression products. In the discussion on the challenges and further research directions, we suggest more focus should be on the dynamic interaction between the coal particle and water droplet in wind tunnels or well controlled onsite conditions.

**Key words:** coal worker's pneumoconiosis, surfactant, coal dust suppression, coal wettability, wind tunnel

## **2.1 Introduction**

Coal dust is a primary health hazard for underground coal workers. Studies have shown that prolonged exposure to excessive quantities of respirable coal dust can lead to irreversible and potentially fatal lung diseases namely Coal Worker's Pneumoconiosis (CWP), chronic obstructive pulmonary disease, tuberculosis, chronic bronchitis, emphysema, and other lung diseases. These lung diseases are commonly observed in coal mine workers and contribute to a higher mortality rate (Ayoglu et al., 2014b; DE Pollock et al., 2010; Ross & Murray, 2004; Weeks, 1991).

Although the prevalence of CWP has substantially and systematically decreased over the past few decades due to many efforts on coal dust control, new cases have continued to occur, and the prevalence of CWP have shown an increasing trend in the last few years. For example, the prevalence of CWP in the US decreased from 6.5% in the 1970s to 2.1% in the 1990s, while it increased to 3.2% in the 2000s (Joy et al., 2012; Laney & Attfield, 2010; Mo, Wang, Au, & Su, 2014). During 2005-2015, 1.5% coal miners were found had CWP in the US and the highest prevalence of 2.4% was found in the eastern region (Reynolds et al., 2017). In Australia, it was suggested CWP was absent for the last 30 years until more than 10 coal miners were diagnosed with CWP in 2016. It was projected that up to 1,000 coal miners in Queensland could have CWP among the 150,000 unprocessed x-rays (Zosky et al., 2016). Glass's study showed that 7.3% of the sample of 248 coal miners in Australia have simple CWP, simple CWP is the early stage of the CWP (Glass et al., 2018). The prevalence of CWP is also high in China, which was 6.02% in the 2010s, and 13,955 and 13,846 miners were diagnosed with CWP in 2013 and 2014, respectively. (Han et al., 2017; Harrison, 2015a; Zosky et al., 2016).

The increase in CWP prevalence may be due to the rapidly increasing mine productivity as the level of mining mechanization has been raised (Ross & Murray, 2004). In order to provide a safety working environment for coal miners, a Permissible Exposure Limit (PEL) for respirable coal dust has been set up in most mining nations (Darling, 2011; Wu, 2003). For example, the PEL for respirable coal dust of 2 mg/m<sup>3</sup> was enacted by the Mine Safety and Health Administration (MSHA) in 1972 in the US, and this was further reduced to 1.5 mg/m<sup>3</sup> in August 2016 (Mine Safety and Health Administration, 2014; NIOSH, 1995). In addition, a Recommended Exposure Limit (REL) of 1 mg/m<sup>3</sup> was adopted by the National Institute for Occupational Safety and Health (NIOSH) in September 1995 (NIOSH, 2003a). During 1995-2003, MSHA coal mine inspectors and operators collected 657,419 coal dust samples and the results indicated that 6.5% of them exceeded the 2.0 mg/m<sup>3</sup> limit and 24.6% were greater than 1.0 mg/m<sup>3</sup> (Safety & Studies, 2008). Moreover, during 2004-2008, 6,600 valid compliance samples were collected from longwall designated occupations in longwall coal mines in the US, and it was found that 11% exceeded the 2.0 mg/m<sup>3</sup> limit (Niewiadomski, 2009). In Australia, 10% of coal mines exposed longwall operators to the coal dust level that equals to or greater than the exposure limit in 2012, and it was further increased to 60% in 2014 (Harrison, 2015b). These existing issues indicate that an effective technology is needed to reduce coal dust levels to the acceptable limit in order to eliminate its health-related hazards.

Considerable research has been carried out in developing coal dust control technologies, and water suppression with added surfactant is one of the effective approaches. The laboratory and

field studies started in 1924 in Great Britain. Later, the use of surfactants for coal dust control was suggested in the US around 1940, and it was considered that surfactants could be applied as an aid based on preliminary laboratory test results (Hartmann & Greenwald, 1940). After 1970, a fully mechanized longwall mining method was widely used by the coal mining industry in the US, which exacerbated the coal dust problem due to a high production rate (Peng, 2006). Traditional coal dust suppression methods, such as the use of ventilation and water spray with plain water, were considered not adequate to meet the standard coal dust PEL (Harrold, 1979). Therefore, increasing attention was paid to research related to surfactant aided water spray coal dust suppression technology. This can be a function of two main phenomena; firstly the efficiency of droplet-particle contact in a gaseous phase in the macroscopic sense and, secondly, the microscopic physico-chemical adhesion phenomena once a liquid droplet has come into contact with a coal particle. Compared with using plain water, the coal dust suppression efficiency improvement varied in different studies. It ranged from 0% to 93% (Chander, Alaboyun, & Aplan, 1990; Howard W Kilau, Lantto, Olson, Myren, & Volt, 1996; Kost, Shirey, & Ford, 1980; Meets & Neethling, 1987; Tien & Kim, 1997).

Despite numerous studies have been conducted with different research methods, some outcomes of these studies are still inconclusive or poorly reconcilable. Some results lack the consistency in providing the coal mining industry with effective coal dust suppression solutions. And in some areas, further research is needed in regard to the surfactant function mechanisms. Because of increased occupational health awareness and the recent CWP prevalence increasing trend, the research on this topic is attracting more attention and therefore a critical review would be appropriate. This paper aims at reviewing and identifying the surfactant mechanisms and key factors that influence their effectiveness, and proposes areas of focuses for further research. Commonly used standard static and dynamic test methods are summarized, and affecting factors for coal dust removal efficiency are identified and discussed. The challenges and further study directions are proposed in the discussion section.

## **2.2 Characteristics of surfactant**

Surfactant is an abbreviation for surface active agent. It is a class of chemicals that when in solution, the concentration is higher at the surface than in the bulk of the liquid. This means the surfactant concentrates at the surface where it needs to function, which makes it very economical in use because most properties of water can be retained and the wetting is improved at a small cost. This phenomenon is known as adsorption and occurs at the liquid-solid, liquid-liquid and liquid-air interfaces (Porter, 1994). Since surfactant has the ability of modifying interfacial properties at low concentrations, it is widely used in numerous applications, such as

the mineral, environment, biological, agricultural, pharmaceutical, textile and cosmetic industries (Musselman & Chander, 2002; Rocha et al., 2005; Rosen & Kunjappu, 2012). In the minerals industry, it can be used to increase the coal dust suppression efficiency when added to the water spray system.

### 2.2.1 Surfactant chemical structure

Surfactants are organic compounds and their molecules consist of polar head groups and non-polar tail groups. In general, the head groups can be hydrophilic, lipophobic or oleophobic, and the tail groups can be hydrophobic, lipophilic or oleophilic (Farn, 2008). The types of surfactants applied to coal dust suppression are usually made up of hydrophilic head groups and hydrophobic tail groups. This sub-class of surfactants will be the focus in this paper. The hydrophilic head groups make the surfactants soluble in polar solvents such as water, and these groups determine the main characteristics of surfactants. The hydrophobic tail groups are usually made up of hydrocarbon chains, which make the surfactant insoluble in polar solvent (Farn, 2008; Rosen & Kunjappu, 2012). The surfactant may contain one pair of hydrophilic head and hydrophobic tail, or multiple numbers of pairs in various combinations. The relative sizes and shapes of the hydrophilic and hydrophobic parts determine the properties of surfactants (Porter, 1994).

The properties of the hydrophilic group categorise the surfactant types into four classes: anionic, non-ionic, cationic and amphoteric surfactant. The simplified schematic structures of the four classes are shown in Figure 1. The anionic surfactant possesses a negative charge on the hydrophilic head, and it can be dissociated in water into an amphiphilic anion (hydrophilic group) and a cation. The cationic surfactant, which has a positively charged hydrophilic head, can be dissociated in water into an amphiphilic cation and an anion. However, the non-ionic surfactant has no charge on their hydrophilic head and therefore cannot be dissociated in water. This is because the hydrophilic group is of a non-dissociable type with a neutral charge (Bergh, 1999; Mobius, Miller, & Fainerman, 2001; Salager, 2002). The amphoteric surfactant has both a positive and a negative group, and its charge is dependent on the pH of the solution where it exists (Farn, 2008). Because the amphoteric surfactant are hardly used in the coal dust suppression both in laboratory and field studies, this type is not discussed further in this review.

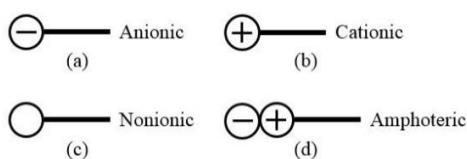


Figure 1. Schematic diagram of simplified surfactant structure. Circle stands for the hydrophilic-polar head (water liking), straight line stands for the hydrophobic-nonpolar head tail (water hating)

### **2.2.2 Adsorption of surfactant on coal**

The adsorption phenomena occur at the interface between the surfactant solution and the coal surface. Coal is composed of coal macerals and mineral matter, and the coal macerals are mainly hydrophobic while the mineral matter are hydrophilic (Gosiewska, Drelich, Laskowski, & Pawlik, 2002). The coal surface is primarily composed of coal macerals, which makes it naturally hydrophobic. Surfactants improve coal wetting performance in two ways: through reducing the surface tension of water and converting the coal surface to hydrophilic by adsorbing onto the hydrophobic sites (Howard W Kilau, 1990; Howard W. Kilau & Pahlman, 1987).

Due to the surface adsorption phenomenon, some pronounced physical changes happen to the solution, and one of them is the surface tension. The surface tension of water is reduced when surfactants are added, and continue to decrease with increased concentration until reaches to a point where it remains relatively constant or changes with a lower slope. This point of concentration is named the Critical Micelle Concentration (CMC), and at this concentration the surfactant molecules form a unimolecular layer on the surface. The CMC value varies from one to another surfactant. At concentration above the CMC, more than one layer of surfactant molecules can form ordered structures on the surface, but this has less effect to the further reduction of the surface tension. A typical representation of the changes of the surface tension with surfactant (Sodium Dodecyl Benzenesulfonate (SDBS)) concentrations is shown in Figure 2.

The adsorption phenomena of surfactants occur after adding surfactants into water, when the hydrophilic head groups of surfactants are in a polar environment, and the hydrophobic tails of surfactants tend toward the hydrophobic coal surface. The hydrophobic tail groups attach themselves to the hydrophobic sites on the coal surface through dipole forces, leaving the hydrophilic heads orientate toward the surrounding water phase. This process converts the hydrophobic sites on the coal surface to hydrophilic sites, which improves the coal wetting performance (Howard W Kilau, 1990; Howard W Kilau & Voltz, 1991; Tien & Kim, 1997). The schematic diagram at the molecular level of adsorption is shown in Figure 3.

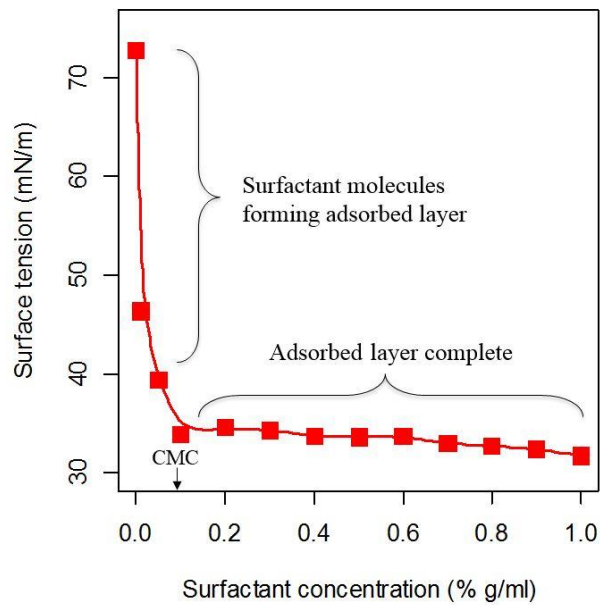


Figure 2. Surface tension versus surfactant (SDBS) concentration

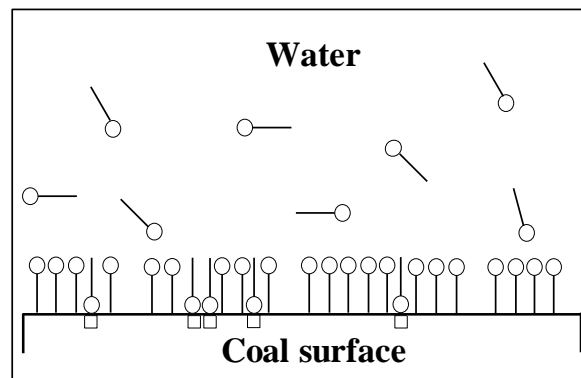


Figure 3. Schematic of adsorption on coal surface by surfactants. (Circle: hydrophilic head group; Short line: hydrophobic tail group; Square: hydrophilic sites on the coal surface)

## 2.3 Evaluation methods for surfactant effectiveness

Two categories of methods, which are static and dynamic methods, are generally employed to evaluate the surfactant effectiveness on coal dust suppression (Figure 4). Static methods test the interactions between coal particles and surfactant bulk solution when coal particles and the solution are in firm contact for a period of time, whereas the dynamic methods measure the phenomena and effectiveness when the coal particles and spray droplets are flowing in air with certain motions.

As can be seen in Figure 4, among the static methods, the surface tension test, the adsorption test, and the zeta potential test are usually used to describe the surfactant adsorption; and the sink test, the contact angle measurement, the capillary rise test, and the drop penetration test are commonly employed to express coal wettability. The dynamic methods consist of the wind

tunnel test and field test. These tests investigate the dynamic interaction between coal particles and water droplets when they are suspended and flow in air. It takes consideration of air flow, contact and collision of coal particles and water droplets, and the wetting and envelopment of dust particles by water droplets. Thus, they are more reliable than those of static tests. However, dynamic tests are more expensive and time consuming, and the results are greatly affected by operating errors. The static tests express the surfactants effectiveness indirectly through parameters related to coal wetting performance such as surfactant adsorption and coal wettability. In general, the higher the coal wettability is, the more easily coal particles can be captured by surfactant solution droplets. Compared to dynamic tests, more studies are based on static tests due to their ease to operate and convenience in controlling the variables. However, they are not a direct reflection of the coal dust suppression effectiveness. This is due to the contact time between solution droplets and coal particles in the spray system being much shorter, and the surface to volume ratio of the water droplet is much higher than that of bulk solutions. The alteration of the surface characteristics and the wetting behaviour in droplets is different compared to that of the bulk volumes.

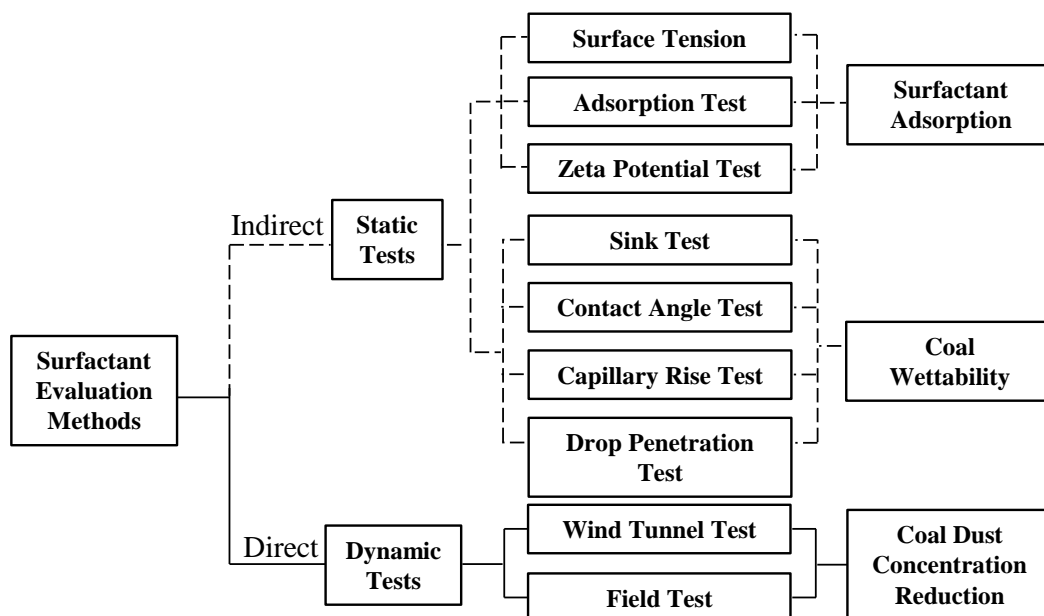


Figure 4. Surfactant effectiveness evaluation methods

## 2.3.1 Static methods

### 2.3.1.1 Surfactant adsorption tests

#### 2.3.1.1.1 Surface tension test

Pure water has a surface tension of 72.8 mN/m at 20 °C (Cheng, Urata, Yagihashi, & Hozumi, 2012a), such a high surface tension makes it hard for coal particles to penetrate its surface,

resulting a low efficiency for water to capture coal particulars. Surfactants can lower the water surface tension with a small amount of dosage. Figure 2 shows that adding only 0.05% surfactant results of up to 50% surface tension reduction.

Among many other test methods, the equipment developed based on the Wilhelmy plate method is the most widely used one (Yildirim, 2006). In it, a thin rectangular plate made of glass or platinum is vertically attached to a microbalance or a tensiometer through a thin metal wire. The plate is partially immersed into the test liquid causing a meniscus formed around the plate (Rolo, Caco, Queimada, Marrucho, & Coutinho, 2002), the capillary rise force on the plate due to wetting is determined by the microbalance or tensiometer (Yildirim, 2006). The surface tension then can be calculated by using the Wilhelmy equation show in Equation 1.

$$\gamma = \frac{F}{(2w + 2d) \cos \theta} \quad \text{Equation 1}$$

Where  $w$  and  $d$  are the plate width and thickness, respectively;  $\gamma$  is the surface tension;  $F$  is the capillary force on the plate, and  $\theta$  is the contact angle between the liquid and plate. In practice, the contact angle is rarely measured or complete wetting is assumed ( $\theta = 0$ ).

The critical surface tension, firstly proposed by Zisman (Zisman, 1964), as the surface tension of liquid needed for spontaneous spreading of the liquid over a certain solid surface. For coal particles to be wetted effectively, the critical surface tension is around 45 mN/m (Parekh & Aplan, 1978). The surface tension is the most significant factor affecting coal wetting performance, and only until after it reaches the critical surface tension, other factors begin to dominate (Howard W Kilau, 1990).

#### **2.3.1.1.2 Adsorption test**

The behaviour of surfactant adsorption on coal particle surface is another indicator to evaluate surfactants effectiveness. The more adsorbed surfactant on the coal particle surface, the better wetting performance it has. Surfactant adsorption can be determined from the difference between the initial and final surfactant concentrations according to Equation 2 (Roman Marsalek, Pospisil, & Taraba, 2011):

$$a = \frac{(C_0 - C_e)V}{m} \quad \text{Equation 2}$$

Where  $a$  is the amount of the adsorbed surfactant, mmol/g;  $c_0$  is the initial concentration of the surfactant, mmol/l;  $c_e$  is the concentration of the surfactant at the adsorption equilibrium, mmol/l;  $V$  is the volume of the surfactant solution, l; and the  $m$  is the mass of the coal, g.



In the adsorption test, the amount of coal particles used lies in the range of 0.1 g to 0.4 g, and the surfactant solution ranges from 50 ml to 100 ml. The dust particle-surfactant solution suspension usually is shaken constantly for 24 hours to reach equilibrium, and then separated by centrifugation to obtain the supernatant liquid for measuring the solution's final concentration (Roman Marsalek et al., 2011; Mishra & Kanungo, 2003; Tang, Zhao, Sun, He, & Hai, 2016).

### **2.3.1.1.3 Zeta potential measurement**

Zeta potential is a physical property that exists when solid coal particles are suspended in liquid, and it is defined as the electrical potential at the surface of hydrodynamic shear in the diffuse region of the double layer of the particles (Hunter, 2013). In aqueous media, both the presented nature ions in the solution and the solid surface determine the zeta potential (Martí et al., 2003). Also, the pH of the solution, as one of the most important factors, impacts the zeta potential to a larger degree. Zeta potential of the solution without defining the pH is a meaningless number (Berg, Romoser, Banerjee, Zebda, & Sayes, 2009; Hunter, 2013). Different alkali or acid, for example NaCl, HCl, or KNO<sub>3</sub>, can be added to adjust the solution pH (Aktas, 2000; R. J. Crawford & D. E. Mainwaring, 2001; R Marsalek, 2008). Zeta potential provides a measurement of the surface charge characteristics of the coal particles in surfactant solution as a function of pH (R. J. Crawford & D. E. Mainwaring, 2001). It can be calculated through the Helmholtz-Smoluchowsky equation as follows (Gallardo-Moreno, González-García, Gonzalez-Martin, & Bruque, 2004):

$$\zeta = \frac{\eta}{\varepsilon} \mu_e \quad \text{Equation 3}$$

where  $\zeta$  is the zeta potential;  $\eta$  is the viscosity of the suspending liquid;  $\varepsilon$  is the dielectric conductivity of the suspending liquid; and  $\mu_e$  is the electrophoretic mobility of particles, which is defined as the ratio between the particle velocity in an applied electric field and the electric field magnitude.

Commonly used commercial measurement equipment include the Zetasizer Nano Series, the Coulter DELSA 440 SX zeta potential analyser, and the PHOTAL CSA-600 micro-electrophoresis instrument with a microprocessor (Gungor, Alemdar, Atici, & Ece, 2001; R Marsalek, 2008; Roman Marsalek et al., 2011; Ozdemir, Taran, Hampton, Karakashev, & Nguyen, 2009). The tested sample needs to be prepared according to the instrument specific instruction.

It was demonstrated that zeta potential should be measured after the solution reaches equilibrium, and the time required for equilibrium varies for different researchers. For example, Marsalek (R Marsalek, 2008) suggested the adsorption of three surfactants (Trimethylammonium Bromide (CTAB), Sodium Dodecyl Sulphate (SDS) and SLOVAFOL) arrived equilibrium after 240 mins, but Crawford and co-workers (R. J. Crawford & D. E. Mainwaring, 2001) equilibrated the suspension in 10 mins for three surfactants (SDS, CTAB and Teric G12A8 (a polyethylene oxide derivative with a 12 carbon length chain incorporating 8 ethylene oxide units)), Ozdemir (Ozdemir et al., 2009) used 1 hour to shake the suspension before zeta potential measurement.

The zeta potential of coal particles in water is negative, which means the coal surface consists of more negative charges than positive charges (Yang, Tan, Wang, Shang, & Zhao, 2007). The addition of anionic surfactant decreases zeta potential as the overall negative charge is increased; cationic surfactant increases zeta potential and makes it less negative or even positive; and there is no remarkable change by adding non-ionic surfactant (Aktas, 2000; R. J. Crawford & D. E. Mainwaring, 2001; R Marsalek, 2008; Yang et al., 2007). Zeta potential can be used to study the surfactant adsorption on the coal particle surface and the stability of the coal particle in surfactant solution. A large difference between the zeta potential of coal in water and the coal in the surfactant solution can be treated as one aspect of improved effectiveness, and this test is usually applied to only cationic and anionic surfactant (Kost et al., 1980).

### **2.3.1.2 Coal wettability tests**

#### **2.3.1.2.1 Sink test**

The sink test measures the immersion rate of coal particles when dropped and totally immersed into the surfactant solution. It has been traditionally employed and regarded as one of the most conventional single measurement methods to analyse the impact of surfactants on coal wettability (Glanville & Haley, 1982). It was initially developed by Draves and Clarkson (Draves & Clarkson, 1931) to determine and compare the efficiency of wetting agents by using skein test, and became a test standard (International, 2010). It was later modified by Walker to determine the wettability of certain surfactant solutions on coal particles, hence, it is also referred to as the Walker test (Walker, Petersen, & Wright, 1952).

The test surfactant solution prepared in distilled or deionized water is placed in a container, and a predetermined amount of coal particles is dropped at a fixed height to the solution surface. The rate at which the dust passes into the solution is recorded. The container used for the sink

test can be a beaker or Petri dish of various sizes, for example, a 250 mL beaker was used by Walker (Walker, Petersen, et al., 1952), and a 1 L Petri dish was applied by Glanville (Glanville & Wightman, 1979). In the Draves test, it was found a less sink time is required when using a container with greater diameter (Fowkes, 1953). This phenomena was also confirmed for the sink test (Glanville & Haley, 1982).

Half a gram of coal particles is traditionally used in the test, although different masses are utilized. For example, in the study conducted by Glanville (Glanville & Wightman, 1979), 0.1 g and 0.5 g coal particle samples were used, but 0.7 g was used by Tien (Tien & Kim, 1997) , and 0.04 g was used by Chander (S. Chander, B. R. Mohal, & F. F. Aplan, 1987). It was pointed out that the wetting rate is independent of the amount of coal samples in the range of 0.02-0.1 g (S. Chander et al., 1987). However, in the range of 0.25-1.5 g, the sink rate is increased twice for a six times mass increase (Glanville & Haley, 1982). For precise/repeatable evaluations, the test should maintain the solution in a water bath of  $25.0 \pm 0.2$  °C. Four to five replications are needed for improved accuracy (Copeland, Eisele, & Kawatra, 2009; Glanville & Haley, 1982; Glanville & Wightman, 1979, 1980; Kawatra, 2006).

The wetting rate can be measured by recording the time taken for the coal particle sample to break through the solution surface by visual observation or record the weight change measured by a microbalance. The latter method is referred to as the modified Walker test (Figure 5). As shown in Figure 6, there are different variations of how the time of wetting is measured. Some investigators measure the time for the vast majority of coal particles to disappear (the vast majority wetting rate), whereas others measure the time for the last trace of coal particle to sink (the last practical wetting rate). While in the modified Walker test, the wetting rate is determined by measuring the weight of the coal particle wetted as a function of time (the initial wetting rate). The wetting rates decrease with time so these three different ways of measurement can result in different wetting rates (S. Chander et al., 1987). The initial wetting rate is much more reliable than the other two. This is because the determination of “the vast majority” and “the last trace” has strong subjectivity which makes it challenging to repeat and compare results.

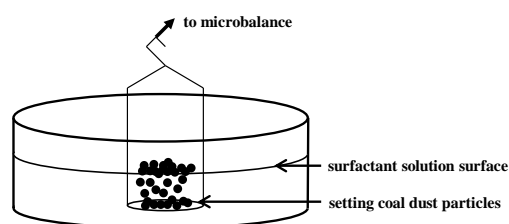


Figure 5. Schematic diagram of the sink test apparatus

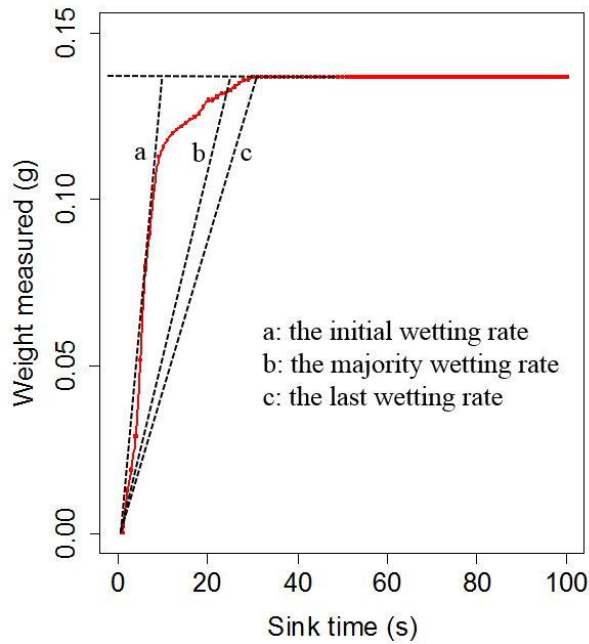


Figure 6. Different wetting rates demonstrated by measurement of 0.40% SDBS surfactant on a subbituminous coal

### 2.3.1.2.2 Contact angle measurement

The contact angle is an indicator of the interaction between the liquid-solid interface and the liquid-vapour interface as shown in Figure 7. It is geometrically acquired by applying a tangent line from the contact point along the liquid-vapour interface in the droplet profile (Yuan & Lee, 2013a). The interface where solid, liquid, and vapour meet is called the “three-phase contact line” or “three-phase contact perimeter”. In general, a small contact angle ( $\theta < 90^\circ$ ) means high wettability, or mostly wetting, whereas a larger contact angle ( $\theta > 90^\circ$ ) corresponds to low wettability, or mostly non-wetting.

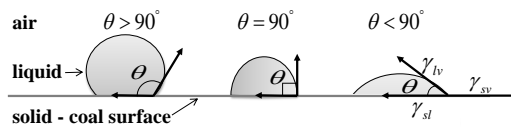


Figure 7. Schematic illustration of contact angle

For a flat, smooth, homogenous solid surface (an idealised surface), the Young’s equation (Equation 4) is satisfied when the horizontal force reaches equilibrium. In this equation,  $\theta_Y$  is the Young’s contact angle, and  $\gamma_{lv}$ ,  $\gamma_{sv}$  and  $\gamma_{sl}$  represent the interfacial tension of liquid-vapour, solid-vapour and solid-liquid, respectively (Young, 1805).

$$\gamma_{lv} \cos \theta_Y = \gamma_{sv} - \gamma_{sl} \quad \text{Equation 4}$$

In practice, due to surface roughness and chemical heterogeneity, the observed contact angles are usually not equal to the Young's contact angle  $\theta_Y$ , and it is not adequate to measure and use the single and unique contact angle  $\theta_Y$  to characterize wetting behaviour. The contact angle is dynamic if the three-phase contact line is in motion. These angles fall within the range of advancing and receding contact angles. The advancing contact angle ( $\theta_A$ ) is measured when the volume of the drop is increasing and the three-phase contact line is just about to advance, and the receding contact angle ( $\theta_R$ ) is measured when the volume of the drop is decreasing and just before the three-phase contact line recedes. The difference  $\theta_A - \theta_R$  is called contact angle hysteresis ( $H$ ). The experimental advancing contact angle  $\theta_A$  is a good approximation of  $\theta_Y$ . The measured receding contact angle  $\theta_R$  is found to have less reproducibility due to liquid adsorption and solid swelling. The contact angle formed by a drop wetting a rough and heterogeneous surface is not equivalent to Young's contact angle, and they are called the Wenzel angle and Cassie-Baxter angle, respectively. Due to the lack of guidelines regarding the degree of smoothness required for the measurement, it is recommended that the solid surface to be made as smooth as possible during measurement (Chau, 2009; Yuan & Lee, 2013a).

Coal samples need to be prepared to be suitable for contact angle measurement. Polishing and cleaning are the two main steps for preparing briquette coal samples (Arnold & Aplan, 1989; Gutierrez-Rodriguez, Purcell, & Aplan, 1984). A detailed sample preparation procedure is given in (J Drelich, Laskowski, Pawlik, & Veeramasoneni, 1997). For coal particles, pressing the particles into plates to create a smooth and flat surface is suggested (R. J. Crawford & D. E. Mainwaring, 2001; Guy, Crawford, & Mainwaring, 1992). Generally 25 to 35 Mpa are used to press the coal powders for 2 to 5 min (He & Laskowski, 1992; J. Li, Zhou, & Liu, 2016; G. Zhou et al., 2016). It should be recognized that contact angle values obtained on compressed pellets are different from that of individual particles at fluid interfaces. This is because of roughness and porosity of the pellet is different from an individual particle, and the mechanical treatment can modify the particle surface to a large extent.

Captive bubble and sessile drop techniques are the two most commonly used contact angle measurement methods. In the captive bubble technique as shown in Figure 8, a surfactant solution is placed in a rectangular glass chamber, and the coal sample is mounted on the chamber with the measured coal sample surface facing downward. An air bubble is introduced by using a U-shaped micro syringe and attached to the coal surface from below. The advancing and receding contact angles can be measured by increasing or decreasing the bubble size by adjusting the syringe. During the measurement, the needle should be remained in contact with

the bubble to prevent bubble distortion (Bariar, 2013; J Drelich et al., 1997; Gosiewska et al., 2002). In the sessile drop technique as shown in Figure 9, a drop of surfactant solution is placed on a prepared coal sample surface by using a micro syringe (Jaroslaw Drelich, Miller, & Good, 1996). The profile of the captive bubble or sessile drop is photographed for contact angle measurement using a computer program (Bigelow, Pickett, & Zisman, 1946; He & Laskowski, 1992; Leja & Poling, 1960).

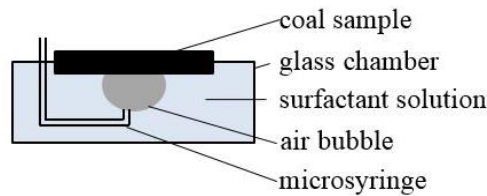


Figure 8. Schematic illustration of captive bubble technique

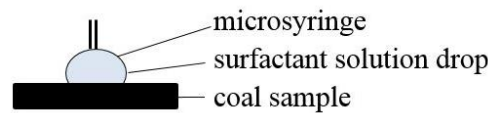


Figure 9. Schematic illustration of sessile drop technique.

Apart from the above described methods, the method named interface partitioning method was also used by some researchers (Wei, Chander, & Hogg, 1992). In this method, a coal particle is placed at the water-air interface and it will be partitioned into two parts, one part penetrates into water and the other part is left in air. The position of coal particles at the interface is determined by coal wettability. For small spherical coal particles, gravity is neglected, and the portioning can be described as Equation 5:

$$R = 1 - \frac{1}{\pi(\theta - \cos \theta \sin \theta)} \quad \text{Equation 5}$$

Where  $R$  is the ratio of the cross-sectional area of the particle in water to its total area of cross section determined by the projected image; and  $\theta$  is the contact angle. This method applies to small coal particles, and at least 30 coal particles are required to be measured to obtain each data.

### 2.3.1.2.3 Capillary rise test

The capillary rise test was developed by Crowl and Wooldridge for assessing the wettability of powders. Due to the capillary action, liquid rises unopposed in a bed of compressed coal powders contained in a glass tube. This test indirectly measures the contact angle of a liquid to fine powder particles (Crowl & Wooldridge, 1967; Zeller, 1983).

A typical capillary rise test apparatus is shown in Figure 10. The height and the internal diameter of the glass tube is 120 mm and 8 mm, respectively. A glass fibre filter is attached to the bottom of the glass tube by water-insoluble glue. Usually 2 g of coal particles are tapped lightly into the glass tube, and then the bottom of the coal column is immersed to the surfactant solution for 2 mm to 4 mm (Howard W. Kilau & Pahlman, 1987; Kost et al., 1980; Zeller, 1983). Burt and Fewtrell (1970) proposed a centrifugal compaction technique to obtain reproducible packing for this test. This method fills the powder into tubes manually, spin the samples by a laboratory centrifuge, which assists achieving a uniform compaction. Study have tested various spinning speeds and found that the porosity of the packing powder bed decreases with the increase of speed (Galet, Patry, & Dodds, 2010).

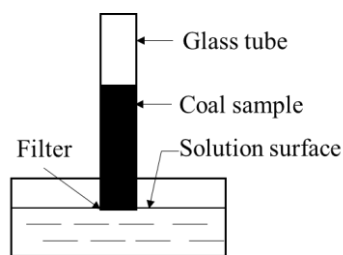


Figure 10. Schematic diagram of capillary rise test apparatus

There are two indicators for coal wettability in this test: the absorbed liquid rise front height and the absorbed liquid weight gain. The liquid front height change can be measured and monitor it over time, or monitor the time taken for the liquid front to reach to a certain height. If weight change is measured, the initial total mass of the prepared sample is measured before the test begin, and monitor its weight change over time (Kost et al., 1980; Wu, 2003; Zeller, 1983). Measuring the gained weight is more accurate than measuring the risen liquid front height. This is because only the liquid front at the wall can be observed, but this does not necessarily reflect the interior progress (or the average progress) of the liquid front.

Capillarity is related to contact angle, and their functional relationship can be described by Equation 6 (Washburn, 1921), where,  $h$  is liquid front rise height, cm;  $k$  is a constant term to modify the mean radius;  $r$  is the mean radius, cm;  $\gamma$  is the liquid surface tension, dynes  $\text{cm}^{-1}$ ;  $\theta$  is the contact angle,  $\eta$  the liquid viscosity, poise; and  $t$  is time, s. If the capillary height and time is measured, the contact angle can be calculated by Equation 7 derived from Equation 6. If weight change is measured, and the porosity of the prepared sample is known, the relationship between weight and height can be expressed as Equation 8, where  $W$  is the weight gain in gram;  $h$  is the liquid front rise height, cm;  $P$  is the porosity of packed coal column, %;  $\rho$  is the density of the wetting solution,  $\text{g cm}^{-3}$ ;  $R$  is the inner radius of the glass tube, cm. Substitute Equation 6 to Equation 8, we can get the relationship between the weight gain and

contact angle (Equation 9). Rearranging Equation 9, the contact angle can be expressed as a function of the weight gain (Equation 10).

$$h^2 = \frac{k\gamma \cos \theta}{2\eta} t \quad \text{Equation 6}$$

$$\cos \theta = \frac{2\eta}{k\gamma} \frac{h^2}{t} \quad \text{Equation 7}$$

$$W = \frac{hP\rho\pi R^2}{100} \quad \text{Equation 8}$$

$$W^2 = \left(\frac{P\rho\pi R^2}{100}\right)^2 \left(\frac{k\gamma \cos \theta}{2\eta}\right) t \quad \text{Equation 9}$$

$$\cos \theta = \left(\frac{100}{P\rho\pi R^2}\right)^2 \left(\frac{2\eta}{k\gamma}\right) \frac{W^2}{t} \quad \text{Equation 10}$$

As can be seen in Equation 6 and Equation 9, the height rise square ( $h^2$ ) or the weight gain squared ( $W^2$ ) have a linear relationship to the measured time ( $t$ ). And the slope of the linear relationship is directly proportional to the contact angle. Thus, a larger slope equates to a smaller contact angle, and therefore, a better wettability.

Although the above theoretical relationship holds, it is not verified in the study of coal powders. It was found that capillary rise test result for aqueous surfactant solutions had poor correlation to sink test and contact angle test, and a much higher concentration of surfactants were required to yield some discernible weight gain. One conceivable explanation is that surfactant concentration on the interface between the coal powders and the wetting solution is reduced due to adsorption, thus a significantly higher concentrations of surfactants are required in order to observe discernible differences (Feldstein, 1981; Glanville & Haley, 1982).

#### 2.3.1.2.4 Drop penetration test

Drop penetration test, also named particle bed soak test, measures the penetration rate of surfactant solutions into powders due to gravity. It was extensively applied by Wu and his co-workers (Wu, Chen, Zhou, & Wang, 1998; Wu & Gu, 2001; Wu, Ou, & Zhou, 2005; Wu, Peng, & Wu, 2007). The test can be performed in two ways. In the first method (Figure 11 (a)), an end tape sealed glass tube (5 mm ID) is filled with a tight bed of coal particles up to 5 cm. 40  $\mu$ L (H. Kilau, 1993; Howard W Kilau & Voltz, 1991) or 200  $\mu$ L (Wu et al., 2007) surfactant solution is gently deposited on the surface of the powder column by using a micropipette. To



analyse the penetration ability, time is recorded for the solution front face to reach a certain depth. In the second method, one droplet of surfactant solution is gently deposited on the flat surface of the coal powder bed (Figure 11 (b)). The time required for the droplet to penetrate completely into the coal powder is recorded (Chan, Cooke, Horvath, & Aziz, 2012; H. Kilau, 1993; Susana, Campaci, & Santomaso, 2012). The wetted area on the powder bed was also used as a parameter to evaluate the wetting performance (Wu, Ou, et al., 2005; Wu et al., 2007; Wu, Zuo, Ou, Zhou, & Li, 2005). In general, the less penetration time and the larger wetted area, the better the wetting performance surfactants possess.

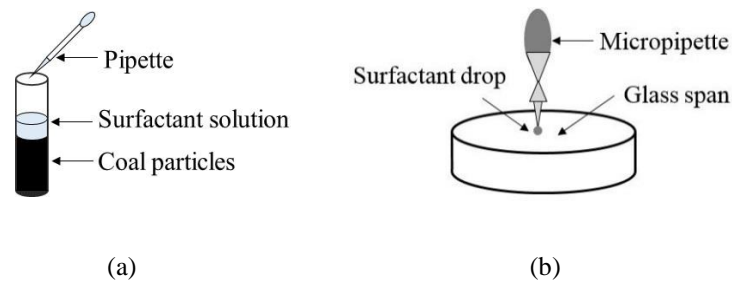


Figure 11. Schematic diagram of the drop penetration test apparatus

## 2.3.2 Dynamic methods

### 2.3.2.1 Laboratory wind tunnel tests

The coal dust suppression efficiency is a complex function of many factors that are not easy to control in the underground working conditions. Simulated laboratory wind tunnel tests provide a controlled environment where the tests can be performed easily and economically. The wind tunnel apparatus is usually designed by the investigators for specific research objectives. Generally, such system includes a wind tunnel to confine the spread of dust, a blowing or exhausting fan to provide air flow, a dust feeder to introduce dust particles into the wind tunnel, the water spray apparatus, the dust monitoring equipment, and a tail dust collection unit.

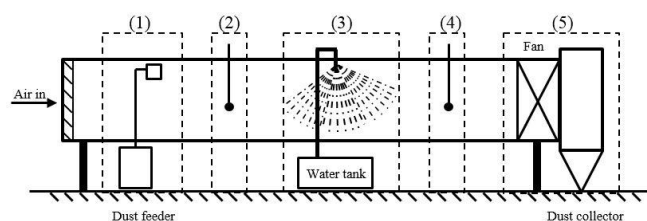


Figure 12. Schematic diagram of a wind tunnel. (1) dust feeder; (2) dust monitor before water spray; (3) water spray nozzles; (4) dust monitor after water spray; (5) exhaust fan and a dust collection unit.

The basic structure of wind tunnel used in the laboratory is shown in Figure 12. The size (length×width×height) of the wind tunnel varies in different laboratory tests. Wang (YP Wang,

Tien, Wilson, & Erten, 1991) designed a wooden wind tunnel with 4.1 m×0.61 m×0.796 m, but in the experiment performed by Chugh (Hirschi, Chugh, Sana, & Mohanty, 2002), used a 7.6 m×3.7 m×2.7 m wind tunnel. Tessum (Tessum, Raynor, & Keating-Klika, 2014) applied a wind tunnel with an unknown length × 0.30 m×0.30 m, Zhou (Q. Zhou, Qin, Ma, & Jiang, 2017) performed tests in a 6 m×1.2 m×1.2 m wind tunnel that constructed of glass and steel, and a larger wind tunnel with size of 22 m×1.3 m×1.45 m was utilized in the study conducted by Zhang (Zhang, Shao, & Huang, 2014).

Air is drawn through the inlet of the wind tunnel by an exhaust fan, and the supplied air velocity is generally in the range of 0.60 m/s to 1.1 m/s (Tessum et al., 2014; YP Wang et al., 1991). A dust feeder is placed at the beginning section where coal particles are injected. It usually takes around 3 minutes for air velocity and dust concentration to reach a homogeneous and stable state (Douglas Pollock & Organiscak, 2007; Zhang et al., 2014). A real-time aerosol monitor is usually used for measuring the dust concentration before and after the coal particles pass the water spray area, based on which the coal dust removal efficiency is determined. In the spray system, nozzles are chosen according to different application scenarios. For example, full cone spray nozzles are mostly employed at the location far away from the coal dust source or at the transfer points, while the hollow cone spray nozzles are usually applied on the coal dust source. Air atomising spray nozzles are considered as the most effective, but its high maintenance requirement limits their utilisation (Colinet, Rider, Listak, Organiscak, & Wolfe, 2010; Kissell, 2003; T. Ren, Wang, & Cooper, 2014). The optimum operating nozzle pressure is usually in the range of 550 kPa and 590 kPa (Hirschi et al., 2002; Tessum et al., 2014). The flow of the spray is around 2 L/min to 8 L/min (Hirschi et al., 2002; Tessum et al., 2014; Q. Zhou et al., 2017). Finally, at the end of the wind tunnel, a dust collector is required for collecting the coal dust and preventing the pollution of the laboratory and the environment.

### **2.3.2.2 Field tests**

Field test is the most direct way of evaluating the effectiveness of coal dust control because it is conducted under the real working conditions where coal dust need to be suppressed. However, it is challenging due to the convoluted nature of multiple interacting phenomena and it is difficult to draw reliable and generalizable conclusions from the results due to large fluctuation of the underground mining working conditions and the lack of accurate and reproducible measurements. Therefore, only a few comprehensive field studies are available in the literature.

We have listed eight representative studies in Table 1. They all demonstrate that the addition of surfactants improves coal dust reduction efficiency, but the level of improvement varies significantly. These can be contributed to several factors. First, different surfactants were applied to different mine sites with various coal properties leading to various efficiency improvement. Second, different methods were used for coal dust monitoring. For example, Anderson (Anderson, Evans, & Peluso, 1964) used “midget impinge sampling devices”, which counts coal dust by drawing air through the impinge and taps the coal particles in the liquid filled in a container. Kost (Kost et al., 1980) applied “MSA Model G personal samplers” that determine dust concentration by weighing the respirable dusts collected by filters that attached to a cyclone. Other factors, such as the amount of surfactant solution, the quality of airflow, and the mining practices (quantity of explosives, mining method, mining machines, loading technique, etc.) can all contribute to the discrepancies between studies. These make field tests not comparable if the test conditions are not identical, and only conclusive results can be obtained for a specific test condition. The correlation between wind tunnel test and then field test has not been well established. Few studies have done the wind tunnel test and applied the findings to the field with the aim of comparing the results. This may be due to the challenge of the complexity of the actual working conditions which can hardly be simulated in the laboratory.

Due to the dynamic environment in the field, measured dust levels need to be adjusted to compare the situation with and without the use of surfactants. For this purpose, the adjusted dust concentration and the normalized dust concentration are proposed (Kost et al., 1980). They are shown Equation 11 and Equation 12:

$$C_A = C \frac{Q_T P_A}{Q_A P_T} \quad \text{Equation 11}$$

$$C_N = \frac{CTQ_T}{P_A} \quad \text{Equation 12}$$

Where  $C_A$  is the adjusted dust concentration,  $\text{mg}/\text{m}^3$ ;  $C_N$  is the normalized dust emission,  $\text{mg}/\text{ton}$ ,  $C$  is the measured dust concentration,  $\text{mg}/\text{m}^3$ ,  $Q_T$  is air flow when measuring dust concentration,  $\text{m}^3/\text{s}$ ;  $Q_A$  is the average air flow during entire tests period,  $\text{m}^3/\text{s}$ ;  $P_A$  is the production when measuring dust concentration,  $\text{ton}/\text{s}$ ;  $P_T$  is the average production during the entire test period,  $\text{ton}/\text{s}$ ;  $T$  is sampling time,  $\text{s}$ .

Table 1. Summary of representative field tests

Reference	Country	Mine sites	Surfactants and concentration	Efficiency improvement
(Hargraves & McKinnon, 1961)	Australia	Metropolitan Colliery Coal Mine	Three un-named surfactants with unknown concentration	One surfactant improved 25%, the second type improved 30%, and the last one had no significant reduction
(Anderson et al., 1964)	USA	Unknown	Unknown surfactant with 1% and 2%	28~33%
(Kost et al., 1980)	USA	Kittanning Seam, Indiana Country, Pennsylvania	Surfynol 465 (non-ionic), Aerosol ma-80, DC-13 (anionic)	27% respirable dust reduction, 36% total dust reduction
(Hirschi et al., 2002)	USA	Illinois Basin #5 seam coal	JFP95Blue with 0.01%	1~40%
(K. Wang, Jiang, Wu, Shao, & Pei, 2014)	China	Da ning Mine	Alcohol polyoxyethylene ether (non-ionic) with 0 ~ 0.30%	57.7~84.9%
(J. Li et al., 2016)	China	Yuejin Coal Mine	0.01% SDS (anionic) with 0.05% NaAc	67.97% respirable dust reduction, 70.74 % total dust reduction
(Q. Zhou et al., 2017)	Chian	Luwa Mine	0.03% SDBS (anionic) + 0.03% Acid methyl ester ethoxylate (nonionic) with ratio of 5:1	44.94% respirable dust reduction, 31.79 % total dust reduction
(Q. Zhou, Qin, Wang, Wang, & Wang, 2018a)	China	Luwa Mine	0.03% SDBS (anionic) with 0.05% NaAc	75.15% respirable dust reduction, 78.32 % total dust reduction

## 2.4 Influencing factors

Previous investigations have proved that a variety of factors affect the efficiency of surfactants on coal dust suppression. In this paper, factors are grouped into three categories: factors related to surfactant type, factors related to coal dust properties, and other factors. Their influence will be discussed in detail in this section.

### 2.4.1 Factors related to surfactants

Table 2 summarizes published studies on surfactants for the improvement of coal wettability or coal dust remove efficiency. A greater proportion of these studies focus on coal wettability enhancement, whereas only few studies are applicable to the coal dust concentration changes by dynamic test measurement. The focus area of surfactants used for coal particles has been on how surfactants themselves as well as coal particles properties affect the surfactant efficiency. Non-ionic, anionic and cationic surfactants with various concentrations, and surfactant with additives were investigated to coal particles with different sizes or different rank under various

test methods. A number of important findings were summarized in Table 2, which can help provide a guideline for choosing suitable surfactant.

#### **2.4.1.1 Surfactant type**

Different surfactant types act differently in modifying the characteristic of water solution, thus perform differently when applied to water spray systems for coal dust control. The choice of the surfactant type is the primary factor to consider for improved dust suppression. A wide variety of surfactants were investigated in order to determine the most effective ones in terms of efficiency and cost. SDS (anionic), CTAB (cationic) and Octylphenol Ethoxylate (Triton X-100) (non-ionic) are the most studied surfactants in coal wettability. The degree of wettability changes mainly depend on the type and adsorption characteristic of surfactants (Bera, Kumar, Ojha, & Mandal, 2013).

Generally, non-ionic surfactant is considered to be the ideal candidate because it shows the highest efficiency on coal wettability. A total of 120 non-ionic surfactants (most have a general formula of  $RX(CH_2CH_2O)_nH$ ) with the Hydrophile-Liophile Balance (HLB) number in the range of 7 to 9 were investigated and found that the surfactant Surfynol family performed the best both in the sink test and wind tunnel test (Tien & Kim, 1997). Its coal dust collection efficiency in the wind tunnel for particles below 3  $\mu m$  achieved more than 80% improvement compared to using water only. In another study that tested three types of surfactants (SDS, CTAB, Teric G12A8), the non-ionic surfactant was found to reduce contact angle the most, display the highest adsorption density, and cause the zeta potential-pH dependence of the coals to become less negative (R. J. Crawford & D. E. Mainwaring, 2001). In a wind tunnel study (Tessum & Raynor, 2017a), non-ionic surfactant was proved to have the same or higher coal dust collection efficiency than cationic or anionic surfactant sprays. The superior performance of non-ionic surfactants may be due to two reasons. The first one is that the CMC for non-ionic surfactants is lower than other types. This means micelles can be formed at a lower concentration, which makes it more efficient. The second reason is the aggregation number, which is known as the number of surfactant molecules in a micelle, is considerably higher for non-ionic surfactant than other types. However, an inconsistent result were observed by Zhou (G. Zhou et al., 2016), which shows an non-ionic surfactant Coconut Fatty Acid Diethanolamide (CFAD) has less effect on reducing the contact angle compared to an anionic surfactant (SDS) and an cationic surfactant (CTAB).

Anionic surfactants are also preferred by researchers in studies where coal wettability is modified due to their ease of use and low cost of manufacture. Most studies show the

improvement on coal wettability caused by anionic surfactants are satisfactory, although not as good as non-ionic surfactants but better than cationic surfactants. SDS and SDBS are the two most popular used surfactants. Larger reductions of surface tension and sink time were observed when a 0.10% applied to subbituminous coal particles by Chen (Chen, Xu, & Albijanic, 2017). It was found SDS increased the hydrophilicity of low rank coals (S. Wang, Tang, & Tao, 2018), and 0.20% to 0.40% resulted in more than half reduction of contact angles tested on anthracite and bituminous coals (Q. Li, Lin, Zhao, & Dai, 2013). Zhou (Q. Zhou et al., 2017; Q. Zhou et al., 2018a) applied wind tunnel tests and field tests and both results indicated that SDS helps to remove coal dust efficiently. Compared with non-ionic surfactant, Crawford (R. J. Crawford & D. E. Mainwaring, 2001) noticed that SDS is less effective for reducing the contact angle and adsorbing on coal surface. Anionic surfactant has a higher CMC than non-ionic surfactant. This means, at the same concentration, less micelles are formed when using anionic surfactant, thus it is less effective.

In aqueous solutions, coal particles possess a negative charge, thus cationic surfactants are not preferred. This is because, when absorbed to coal, the hydrophobic end of the surfactant is directed away from the coal, which leads to poor wettability (Feldstein, 1981). However, this assumption is not supported by studies that measure the electrostatic charge on freshly generated airborne coal particles (Q. Hu, Polat, & Chander, 1992; Polat, Polat, Chander, & Hogg, 2002). The coal particles from anthracite, bituminous, and sub-bituminous coal possess overall charge of both signs with relatively equal chances, and the magnitude increases with coal rank and air humidity. They have also found that the droplets generated from anionic surfactant solutions have a greater percentage of negative charge, and such charges significantly increase at low concentrations but reach to a constant beyond the CMC. This trend also applies to cationic surfactant, but the dominant charges are positive with higher magnitude. Surprisingly, for non-ionic surfactant, more droplets possess positive charges and the magnitude increases linearly with surfactant concentration. For cationic surfactants, the dust collection efficiency increases with the droplet charge, which is affected by surfactant concentration. This relationship was not observed with anionic and non-ionic surfactants. The higher amount of charge on cationic surfactants impact the droplet-particle collision process, but for anionic and non-ionic surfactant droplets with less charges, the dust collection efficiency is only dominated by the thermodynamic relationship. This means reducing the surface tension for improved dust suppression efficiency is only possible for anionic and non-ionic surfactants which impart low charges on droplets. The same group of authors found that the cationic surfactants provided the highest dust collection efficiency when using bituminous

coal dust in a dust chamber (Q. Hu et al., 1992). They also found that the dust collection efficiency did not correlate to the change of surface tension and contact angle measurements. Rather, in the concentration range of 0.001% to 0.1%, dust collection efficiency first increased and then decreased. The higher collection efficiency range for non-ionic and anionic surfactant is narrow, whereas cationic surfactant had the highest collection efficiency and a broader effective range.

#### **2.4.1.2 Surfactant concentration**

Surfactant concentration is a key factor that reduces the surface tension of the solution, increases the coal wettability, and therefore causing a key effect on increasing dust suppression efficiency. As mentioned in Section 2.2, the surfactant concentration is higher at the surface than that in the bulk of a liquid due to adsorption. Porter (Porter, 1994) proposed a simplified model for this phenomenon as four stages: (1) At very low concentration, the surfactant molecule lies flat on the surface without specific orientation. (2) The number of surfactant molecules increases with the increase of concentration, so they start to orientate to fit in the confined surface space. (3) With surfactant concentration continue increasing, surfactant molecules are sufficient to form a unimolecular layer on the surface, and this concentration is known as the CMC. At this stage, some surface properties are at an optimum, such as the surface tension and the surface adsorption. (4) At the concentration above the CMC, no apparent change in the adsorption at the hydrophobic surface, and an ordered structure known as micelle is formed as long as the concentration is above the CMC.

There are two opposing forces of interaction that determine the CMC. The 1<sup>st</sup> force (Force 1) exists between the polar groups repelling one ionic to another due to the mutual charge repulsion. A larger charge leads to a greater repulsion and less tendency to form micelles. A 2<sup>nd</sup> force (Force 2) exists between the hydrophobic groups to attract the groups together. When Force 2 is larger than Force 1, the molecules will aggregate together (Porter, 1994). Thus, the CMC is determined by the relative strength of these two forces. Knowing the CMC of a certain surfactant to a given coal sample is of practical interest as this might be the lowest concentration needed to get the maximum coal dust reduction benefit. The optimum surfactant concentration varies with surfactant types and coal particles. In addition, no apparent change in the wetting performance of coal particles when the surfactant concentration is lower than the critical wetting concentration, which is the minimum concentration required for improved wetting phenomena (Glanville & Haley, 1982).

### 2.4.1.3 Synergistic effects

The combination use of surfactants and the addition of some chemical additives can substantially increase surfactant efficiency, thus reducing the solution cost without compromising coal dust reduction efficiency. The addition of a specific chemical additive should be based on the applied surfactant type and quantity. Surfactant mixtures and the addition of electrolytes are commonly ways to reach the synergistic effect.

#### 2.4.1.3.1 Surfactant mixtures

When surfactants are used as a mixture, the properties of the mixed surfactants will deviate from those of each individual surfactant when it is used by itself. The CMC of the two mixed surfactants system could be lower than that of each individual surfactant, which is the main cause achieving the synergistic effect.

The CMC of two mixed surfactants system can be determined by the following equations (Clint, 1975, 1992; Porter, 1994):

$$\frac{1}{CMC_M} = \frac{\alpha}{CMC_A} + \frac{(1 - \alpha)}{CMC_B} \quad \text{Equation 13}$$

$$C_A = x f_A CMC_A = \alpha CMC_M \quad \text{Equation 14}$$

$$C_B = (1 - x) f_B CMC_B = \alpha CMC_M \quad \text{Equation 15}$$

Where  $CMC_M$ ,  $CMC_A$  and  $CMC_B$  are the CMC of surfactant mixture, surfactant A and surfactant B, respectively.  $C_A$  and  $C_B$  are the monomer concentration of surfactant A and B,  $x$  is the mole fraction of surfactant A in the mixed micelles,  $\alpha$  is the mole fraction of surfactant A in the mixture, and  $f_A$  and  $f_B$  are known as the activity coefficient in the expression of  $\exp[\beta(1 - x)^2]$ ,  $\beta$  is the molecular interaction parameter.

It can be observed that CMC of the mixture was largely determined by the surfactant with the lower CMC. This means the properties of some commercial surfactant mixtures are greatly impacted by the longer hydrophobic chains rather than short chains. In Equation 14 and Equation 15, when  $\beta=0$ , then the mixed surfactant system is considered as an ideal mixture, there is no interaction of the two surfactants. A synergistic effect occurs when  $\beta<0$ , and the  $CMC_M$  will be lower than that of an ideal mixture. However, when  $\beta>0$ , the  $CMC_M$  will be higher than that of an ideal mixture, which is rare in practice.

The combination of non-ionic surfactant and anionic surfactant can produce a synergistic effect on improving the coal wettability (Feldstein, 1981). The combination also has the advantage



of reducing the solution dependency on pH and electrolyte concentrations. Results from drop penetration tests have shown a 40~60% improvement when polymers is added to non-ionic or anionic surfactants (Zinkan & Koenig Jr, 1988). In a different study, capillary rise and drop penetration tests were used to investigate the synergistic effect between surfactants (Sodium Di (2-ethylhexyl) Sulfosuccinate (SD2ES), SDS and SDBS) and polymers (low and high molecular weight) (Howard W Kilau & Voltz, 1991). Coal samples with different hydrophobicities were tested, and the results showed that the combination of the anionic surfactant and a polymer showed a synergistic enhancement on the higher hydrophobic coal sample, but negligible improvement on the lower hydrophobic coal sample. The combination use of the non-ionic surfactant and the polymer showed no apparent wetting performance improvement. Both low molecular weight polymers and high molecular weight polymers showed similar wetting enhancement, however, the excessive addition of the high molecular weight polymers could lower the wetting performance of the surfactant.

#### **2.4.1.3.2 Addition of electrolytes**

The addition of electrolyte is another way of enhancing surfactant performance. It can significantly decrease the CMC of anionic and cationic surfactants, while less so for non-ionic surfactants. This is because the addition of electrolytes can decrease the charge in the solution, reduce the repulsion between ionic hydrophilic groups and weaken Force 1 (as described in Section 4.1.2), resulting in increased tendency of micelles forming (Porter, 1994). Due to the accessibility and cost effectiveness of the electrolytes, their use can lead to a considerable practical interest in the mining industry.

By using the sink test, the addition of electrolytes were shown to reduce the sink time dramatically. However, such wetting enhancement was only shown by many dipositive ions, especially calcium or magnesium ion, but not monopositive or tripositive ions. The amount required to have apparent effects was as low as 0.06 mmol/l, which is commonly see in any natural water (Glanville & Wightman, 1979). By adding sodium salt ( $\text{Na}_2\text{SO}_4$ ) and potassium salts ( $\text{K}_4\text{Fe}(\text{CN})_6$ ) to a surfactant (SD<sub>2</sub>ES) solution (Howard W. Kilau & Pahlman, 1987), the wetting performance was increased remarkably as demonstrated by results from both the capillary rise and drop penetration test. Potassium hydroxide and sodium hydroxide were also shown effective in this regard. Adsorption capacity of SDBS surfactant on coals was remarkably increased when in presence of  $\text{KNO}_3$  (Mishra & Kanungo, 2003). A possible reason is that the addition of electrolytes can cause the oxygen in the hydrophilic head groups of the surfactant molecules to repel from the coal surface, resulting the surfactant molecule to “stand out” and making the coal surface become hydrophilic (Kim & Tien, 1994).  $\text{Na}_2\text{SO}_4$  was also

shown to largely decrease the wetting time in the drop penetration test. But this synergistic effect decreases as the anionic surfactant concentration increases (Wu & Gu, 2001).

To overcome the shortage of dust particle being re-raised after the covering surfactant solution is dried, Xi (Xi, Feng, & Li, 2017) developed a product that works synergistically for both improved wettability and longer effect. It mixed polyethylene into the SDS surfactant solution. This product although showed reduced wettability than using only SDS, but can form a thin layer of armor on the coal particle surface. It can hold the particles together after drying, thus potentially provide a longer effect. Dynamic tests, such as wind tunnel tests, should be carried out to further confirm the finding.

## **2.4.2 Factors related to coal properties**

Coal natural wettability is determined by the surface hydrophilic sites and hydrophobic sites, which can be modified by the adsorption of surfactants. Surfactants perform differently on improving the wettability of different coals, and this section will discuss how coal properties, such as coal rank, coal dust particle size and coal pore structure influence the surfactants performance.

### **2.4.2.1 Coal rank**

Coal rank is the primary factor determines its hydrophobicity. Properties include the carbon content, moisture content, ash content, and volatile matter content varies among coal ranks. High rank coals contain more carbon, less moisture and less volatile matter content, and they tend to be more hydrophobic than low rank coals (Speight, 2015; J. Yu, Tahmasebi, Han, Yin, & Li, 2013). It should be noted that coal is an extremely heterogeneous material with physical and chemical properties variations even for the same coal sample. Past studies have shown that the performances of surfactants are highly dependent on the types of coal used (R. J. Crawford & D. E. Mainwaring, 2001; Feldstein, 1981; S. Liu et al., 2016; Tien & Kim, 1997).

Laboratory studies indicate that a surfactant solution is more effective on the wettability of higher rank coal. For example, a larger contact angle decrease of up to 20°, a larger variation in zeta potential, and a higher surfactant adsorption density for high rank coal samples were observed in surfactant solutions (R. J. Crawford & D. E. Mainwaring, 2001; Q. Li et al., 2013; Mohal, 1988). Similarly, when different rank coal samples were tested using sink tests, the wetting rate of lower rank coal was observed worse than those of higher ranks (S. Chander et al., 1987; Tien & Kim, 1997).

### **2.4.2.2 Coal particle size**

Coal particles that can be suspended in air or settled in the mine airway are generally 1 $\mu$ m to 100  $\mu$ m (Hartman, Mutmansky, Ramani, & Wang, 2012). Agglomerates are always formed in air, which are affected by both particle electrostatic charges and air humidity (Polat et al., 2002).

Numerous studies have indicated that finer coal particles are more difficult to be wetted. Such phenomenon is believed to be attributed to finer coal particle's large pore volume, higher degree of surface roughness and surface energy (Q. Li et al., 2013). For example, the sink time for 30  $\mu$ m coal particles was 56 s, whereas for 450  $\mu$ m particles was only 1.2 s (Glanville & Haley, 1982). In the penetration test, the time required for finer coal particles to penetrate to certain heights are much greater than that of coarser particles (Wu et al., 2007). For example, when using 0.4 % SDS surfactant, it took 150 s for particles in the range of 177 to 250  $\mu$ m to penetrate 4 cm, but it used more than four times longer for particles in the range of 125 to 150  $\mu$ m. Finer coal particles tend to have larger contact angles than those of coarser ones, regardless of coal ranks (Q. Li et al., 2013; Yang, Wu, Gao, & Li, 2010). Significantly lower dust collection efficiency for smaller coal particles was also confirmed in wind tunnel studies (Tessum & Raynor, 2017a).

The mechanisms of the above-mentioned phenomena is not well established. Yang and Li (Q. Li et al., 2013; Yang et al., 2010) have proposed several sound possibilities. The first one is that smaller coal particles have large specific area and stronger adsorption of air, which reduces its wettability. The second possible mechanism is smaller coal particles have smaller average pore radius but larger specific pore volume. This leads to a rougher surface that is not easily wetted. The last mechanism is attributed to surface atoms composition and structure change. This was investigated by the X-ray photoelectron spectroscopy (XPS) spectrum analyses, and found that the carbon content is higher in smaller coal particles, but the oxygen content is lower. This causes an increase of hydrophobicity in smaller coal particles. A constant spreading wetting hypothesis was also proposed (Glanville & Haley, 1982), in which the wetting rate per unit coal surface area is assumed to be constant, and smaller particles have large surface area thus require more time to be wetted.

### **2.4.2.3 Coal particle pore structure**

Coal particles are porous material and their porosity is closely related to their chemical characteristics. The pores in coal particles are generally classified into three categories: macro-pore (> 50 nm), meso-pore (between 2 to 50 nm), and micro-pore (< 2 nm) (Speight, 2013).

The adsorption of surfactant solution by coal particles is a complex process because it involves coal surface-surfactant solution-gas interactional effects, which is a solid-liquid-gas interaction. Li (Q. Li et al., 2013) proposed that coal rank is one of the reasons that contribute to various pore diameter distributions, for example, compared to anthracite coal sample, lignite and bituminous coal sample have more meso pores and less macro pores. He also pointed out that finer coal particles have more developed pore structures, which maybe a reason leads to its poor wettability. Coals with larger pore parameters (surface area and pore volume) were found to have larger contact angles, which means poor coal wettability. This is because the more developed and complicated pore structures lead to poor wettability as they have stronger gas adsorption capacities, and gas film can be easily formed around the coal particles which weaken the wetting process. Using a fractal dimension to describe pore structures, he concluded that the larger the fractal dimensions are, the more developed the pore structures, and the poorer the coal wettability is. Therefore, compared to larger size coal particles, the respirable coal dust has a more developed pore structure and larger fractal dimension, thus possesses stronger capacity of gas adsorption. Therefore, they are more hydrophobic and hard to be wetted. This conclusion also was supported by Zhou (Q. Zhou, Qin, Wang, Wang, & Wang, 2018b).

### **2.4.3 Other factors**

Aside from the factors mentioned above, other factors, such as temperature, air humidity, and water magnetization, can also influence coal wettability. For dust control purposes, temperature and ambient humidity are rarely treated as variables in underground practice. The pH of water also influences surfactant performance, but it is usually defined by the nature of the native water used in the mine. Magnetization of water is widely studied as an addition or alternative method for surfactant, which is not the focus of this paper. These factors are only briefly discussed in this section.

The surfactant CMC decreases with increased temperature, which leads to increased surfactant effectiveness. Study has shown a linear increase of sink test wetting rate with temperature for both anionic and non-ionic surfactants (Glanville & Haley, 1982). The adsorption capacity was also found obviously increased with temperature when the surfactant concentration was above CMC (Lyu et al., 2018; Roman Marsalek et al., 2011; Mishra & Kanungo, 2003).

Studies uses air humidity as a variable is not common due to the challenge to control it both in the laboratory and mine sites. By using a water spray system to suppress dust that emitted from an enclosed dust chamber, Grindnig (Grundnig et al., 2006) found that to reach equal dust

suppression, less water consumption is required for a relatively higher humidity. This means a higher humidity environment is preferred for increased dust suppression efficiency.

Ash is the mineral residual in coal after combustion, its impact on coal wettability in surfactant solutions was inconsistent although the strong hydrophilic property of ash should result in preferred wettability. Xu (C. Xu et al., 2017) used the sink test to measure coal samples with ash contents various between 3.87% and 49.34%, and found that it did not influence the results. Hu's sink tests results (F. Hu, 2014) also supported this finding. However, in a study for coal flotation (J. Wang et al., 2018), zeta potential and adsorption measurements were conducted to coal samples with ash content varied from 8.2% to 63.5%, and the biggest zeta potential change and the most amount of adsorption were observed for the highest ash content. Compared to a low ash content (0.1%) coal sample, a four times more surfactant adsorption was also observed for samples with 10% ash content (X. Liu, Liu, Fan, & Zhang, 2017).

pH variations are found to affect the wetting, and the level of influence is dependent on the coal and the effect is more obvious under lower calcium ion concentration (Feldstein, 1981). Surfactant magnetization is also viewed as a factor that can change coal wetting performance by some researchers. Yan (Yan & Haike, 1998) found that the surface tension of solution was greatly reduced after it was magnetized. Cui (Cui et al., 2011) measured contact angles of coal samples both in un-magnetized and magnetized surfactant solutions, and noticed that magnetized surfactant solution contributed to smaller contact angles. Zhou (Q. Zhou et al., 2018a) also observed that surface tension and contact angle are both reduced when surfactant solution was magnetized, and field tests further demonstrated almost 40% improvement on dust suppression by using magnetized surfactants. In Zhou's another research, surfactants dust suppression efficiency was increased by 9.29% after magnetized in a field test (Q. Zhou et al., 2017), which confirmed magnetization can produce a synergy effect on surfactant solution to capture coal dust. .

Table 2. Summary on representative surfactant aided coal dust suppression research

	References	Surfactants or Additives	Concentration	Coal dust	Test method	Major findings
Non-ionic surfactant	(Glanville & Haley, 1982)	Triton X-100	0 ~20 wt%	Sink test 0.5 g Capillary rise test 2.0 g	Sink test Capillary rise test	1. Larger coal particles have better wettability than finer ones 2. When using non-ionic surfactant, coal wettability increases with temperature
	(S. Chander et al., 1987)	Ethoxylated nonyl and octyl phenols	$0 \sim 10^{-3}$ mol/l	Subbituminous Anthracite	Sink test Contact angle test	1. High rank coal has a better wetting rate than low rank in presence of non-ionic surfactant 2. Sink test results correlate well with contact angle measurement
	(Tien & Kim, 1997)	Surfynol 440, Macol 30, Plurafac RA 43, Mindust 293, and Neodol 92	0.05% 0.10% 0.20%	Bituminous, Subbituminous 0.07 g coal sample	Sink test Wind tunnel test	1. Coal wettability increases with surfactant concentration 2. Surfynol surfactant family works best at coal dust capturing 3. Coal with higher sulfur content shows better wettability when volatility remains unchanged
	(Aktas, 2000)	Diacetone alcohol, DAA, 4-hydroxy-4-methyl-2-pentanon, 2-ethyl hexanl-ol, 2ETH	$0 \sim 8080$ $\mu\text{M}/\text{dm}^3$	Low rank coals, Size: $< 5\mu\text{m}$	Adsorption test Zeta potential test	1. The value of zeta potential of coal particles in non-ionic surfactant solution is determined by the PH of the solution 2. Longer contact time of surfactant and coal particles leads to larger surfactant adsorption
	(R. J. Crawford & D. E. Mainwaring, 2001)	Teric G12A8	$0 \sim 40 \times 10^{-5}\text{M}$	Medium volatile subbituminous coal; Low volatile bituminous coal	Contact angle test Adsorption test Zeta potential test	1. The biggest decrease in contact angle and the highest surfactant adsorption were observed with non-ionic surfactant 2. Surfactant does not change contact angle of the low rank coal 3. Teric G12A8 reduce the zeta potential negativity of coal with coal rank increases and surface functionality decreases

	(G. Zhou et al., 2016)	Cocoonut fatty acid diethanolamide (CFAD)	0.005% 0.05% 0.5%	Size: 70, 100, 160, 200, 320 mesh	Contact angle test	<ol style="list-style-type: none"> <li>1. Compressed coal particles have larger contact angel than polished coal surfaces</li> <li>2. Non-ionic surfactant has worse wetting ability than anionic and cationic surfactants based on the contact angle test</li> <li>3. Contact angle increases with coal particle size which indicates larger coal particles have better wettability</li> </ol>
	(Lyu et al., 2018)	Nonylpheonl ethoxylate with 12 ethylene oxide groups (NPEO-12)	0 ~ 2.0 g/l	Subbituminous coal Size: - 74 $\mu\text{m}$	Adsorption test	<ol style="list-style-type: none"> <li>1. The adsorption process of NPEO-12 on coal surface is spontaneous</li> <li>2. The adsorption amount of NPEO-12 on coals increases with temperature when the concentration is over 0.5 g/l</li> </ol>
Anionic surfactant	(Glanville & Haley, 1982)	Sodium dodecyl sulfate (SDS)	0 ~ 20 g/l	-200 +350 mesh	Sink test	<ol style="list-style-type: none"> <li>1. Wettability increases with surfactant concentration and temperature</li> <li>2. Larger coal particles have higher wettability</li> </ol>
	(R. J. Crawford & D. E. Mainwaring, 2001)	Sodium dodecyl sulfate (SDS)	0 ~ $10 \times 10^{-5} \text{M}$	Medium volatile subbituminous, Low volatile bituminous	Contact angle Adsorption test Zeta potential test	<ol style="list-style-type: none"> <li>1. SDS decreases the zeta potential of higher rank coals, but has little effect on lower rank coals</li> <li>2. The adsorption of SDS on coals is less than the other tested non-ionic surfactant</li> </ol>
	(Q. Li et al., 2013)	Sodium dodecyl sulfate (SDS)	0.05% 0.2% 0.4%	Anthracite, bituminous and lignite coal sample. Size: < 100 $\mu\text{m}$	Contact angle test	<ol style="list-style-type: none"> <li>1. Finer coal particles have poor wettability because of the complex micro structure</li> <li>2. Coal particles with higher volatile content have poor wettability</li> <li>3. Based on contact angle results, 0.20% of SDS was recommended for field application</li> </ol>

	(Chen et al., 2017)	Sodium dodecylbenzene sulfonate (SDBS)	0.05% to 1.0%	Subbituminous	Surface tension Sink test Zeta potential test Capillary rise test Drop penetration test	1. Coal wettability increases rapidly until concentration reaches 0.4%, and levels off afterward 2. Results from the sink test and surface tension are closed related
Cationic surfactant	(R. J. Crawford & D. E. Mainwaring, 2001)	Cetyl trimethylammonium bromide (CTAB)	$0 \sim 6 \times 10^{-5} M$	Medium volatile subbituminous coal, Low volatile bituminous	Contact angle Adsorption test Zeta potential test	1. CTAB makes the zeta potential of coals less negative, for higher rank coals, a charge reversal was observed 2. Adsorption value is higher for low rank coals 3. Less reduction on contact angle compared to non-ionic surfactants
	(G. Zhou et al., 2016)	Cetyl trimethylammonium bromide (CTAB)	0.005% 0.05% 0.5%	Size: 70, 100, 160, 200, 320 mesh	Contact angle test	1. Coal dust with larger porosity and smaller volume percentage of ultramicropore is easier to be wetted 2. Cationic surfactant (CTAB) works better than non-ionic surfactant (CFAD)
	(J. Wang et al., 2018)	Cetyl trimethylammonium bromide (CTAB)	0.10%	Lignite with three densities: <1.45, 1.45~1.80, >1.80g/cm <sup>3</sup>	Adsorption test Zeta potential test	Higher density coals adsorb slightly more CTAB surfactant
Surfactant with	(Howard W. Kilau & Pahlman, 1987)	Sodium di (2-ethylhexyl) sulfosuccinate + NaCl, Na <sub>2</sub> SO <sub>4</sub> and K <sub>4</sub> Fe(CN) <sub>6</sub>	Surfactant =0.0041% ; Additives =2.5 to 30 mmol/l	<74 μm for drop penetration test; <74 μm, 2.00~2.38 mm for capillary rise test	Capillary rise test Drop penetration test	Na <sub>2</sub> SO <sub>4</sub> and K <sub>4</sub> Fe(CN) <sub>6</sub> remarkably improve coal wettability, but NaCl is not effective



(Howard W Kilau & Voltz, 1991)	SD2ES, SDS, SDBS, EAG(10), LEA (12), LEA (12) + polymer	Surfactant = 0 ~ 0.10 wt%; Polymer = 0~0.20 wt%	Bituminous with medium volatile, Bituminous with high volatile, Size: < 100 μm	Drop penetration test Capillary rise test Zeta potential test	1. Adding polymer to non-ionic surfactant solution does not have obvious synergistic effect 2. For the tested two coal samples, the wettability of anionic surfactant with concentration range of 0.13 ~ 75% was all improved by the addition of polymer, and 0.15% of polymer improved the greatest
(Tien & Kim, 1997)	Surfynol TG, Biosoft TD 400 (non-ionic surfactant + Sodium hydroxide (NaOH), Potassium hydroxide (KOH))	Surfactant = 0.05%, 0.10%; Additives = 6.8%	Bituminous, subbituminous	Sink test	1. For Biosoft TD 400, wetting performance indicated by sink tests was improved about 130% when 6.8 % of KOH was added 2. When adding 6.8% NaOH to 0.05% and 0.1% Surfynol TG, sink time improvement (70%) was only observed for the 0.1% Surfynol TG
(Wu & Gu, 2001)	SDS, SDBS, SDHS + Na <sub>2</sub> SO <sub>4</sub>	Surfactant = 0 ~1 %; Na <sub>2</sub> SO <sub>4</sub> = 0 ~30 mmol/l	Lignite with size < 0.09mm	Surface tension Drop penetration test	1. For 0.74% SDS, apparent effect was observed only when the Na <sub>2</sub> SO <sub>4</sub> concentration is greater than 5 mmol/l 2. For 0.99% SDS, the coal wettability increased dramatically with the addition of Na <sub>2</sub> SO <sub>4</sub> when its concentration less than 3 mmol/l, after that, the improvement slowly increased 3. Increased Na <sub>2</sub> SO <sub>4</sub> concentration contribute to the reduction of surface tension for SDS and SDBS
(Mishra & Kanungo, 2003)	SDBS + KNO <sub>3</sub>	Surfactant =0.002 mol/l ; Additives =0.001M	Subbituminous coal Size: <100 μm	Adsorption test	1. Adsorption capacity increases with temperature 2. KNO <sub>3</sub> improved the adsorption of SDBS on coals because more positive sites were generated on the coal surface by the adsorption of K <sup>+</sup> , which helps the attraction of anionic surfactants

(R Marsalek, 2008)	CTAB + SDS	CTAB and SDS were mixed to 0.1 mmol/l	Bituminous Subbituminous Commercial activated coal	Zeta potential test	The zeta potential increases with the ratio of CTAB and SDS when the mixture time is 5 minutes. But when the mixture time is 240 minutes, the zeta potential only increases with the mix ratio less than 1:3
(J. Li et al., 2016)	Lauryl sodium sulfate + Glycerin, NaAc, Urea, NaCl, and Ethanediol	Surfactant = 0.10 wt% ; Additives = 0.05 wt%	60 ~ 100 mesh	Surface tension test Contact angle test Capillary rise test	1. Results from three different test methods were inconsistent 2. The capillary rise method was chosen as the primary evaluation method, which showed adding NaAC to surfactant solution was the most effective
(Q. Zhou et al., 2017)	SDBS + Acid methyl ester ethoxylate (nonionic)	SDBS= 0.03%; Acid methyl ester ethoxylate = 0.03%;	Size: < 75 $\mu$ m	Surface tension test Wind tunnel test Field test	1. The mass ratio of 5:1 for anionic and non-ionic surfactant resulted the lowest solution surface tension 2. Dynamic tests demonstrated up to 9% more effective for the magnetized surfactant solution compared to the un-magnetized one

## 2.5 Discussion

Studies have shown that the CWP occurrence rate increases in recent years in some of the major coal producing countries. Together with greater restrictions on coal dust exposure limits, the coal mining industry is challenged to meet such limits and need a more effective dust management strategy. Surfactant aided water spray has been demonstrated effectiveness in prior work. However, findings are still lack of consistency mainly due to the use of different test methods, disparity in operational procedures, and variation in water quality and coal property. It is believed a breakthrough in this field relies on the achievements in prior work, but an up-to-date critical review was not found. Thus, this paper uses the opportunity to highlight past advances and considers how this knowledge enable achievements for the current challenges.

Choosing the suitable method to evaluate the effect of surfactant is one of the most important steps to obtain the correct and reliable experimental data. Nine commonly used surfactant effectiveness evaluation methods are discussed in detail in Section 2.3. They reflect different aspects including surfactant adsorption, coal wettability, and coal dust concentration reduction. So far, there are no conclusive findings that indicate which method is superior to others. In most cases the researchers either addressed the physico-chemical adsorption aspect, or performed macroscopic studies where too many variables made problem deconvolution difficult. The value and relevance of the methods depend on the focus of the study. For example, for mechanistic and quantitative studies, static tests are more suitable; while for practical and applicable studies, dynamic tests are more intuitive. The results from some of the test methods are closely related while others are not. Also, due to non-standardized test procedure, the same test with different procedures may lead to different results. This is one of the challenges for some inconsistent conclusions in the past studies. There is a demand for identifying a most suitable static test method that can be used to select a surfactant and determine its dust suppression effectiveness without the expensive wind tunnel tests or field tests.

The study of surfactant aided coal dust suppression have many similarities in the study of flotation in mineral processing. Some of the research methods used for flotation study can be integrated to the field of coal dust suppression. Many studies have devoted to the investigation of macroscopic parameters, such as the chemical properties, equipment and operational properties. However, understanding the microscopic perspective of the particle-droplet

interaction is essential for understanding the underlying mechanisms. This microscopic interaction can be classified into the particle-droplet collision, attachment, and stability processes. In flotation, the understanding of these sub-processes are mostly investigated by studying the induction time (Verrelli & Albijanic, 2015) and the particle- bubble encounter (Hassanzadeh, Firouzi, Albijanic, & Celik, 2018). Applying the same principles to coal dust, the induction time can be understood as the time required for the particle and droplet to form an attachment. It is useful to quantify the effectiveness of a dust suppression solution. The goal should be increasing the time available for particle-droplet attachment by optimizing the suppression system, and reducing the induction time by the modification of droplet surface chemistry, size and its velocity. Similarly, the particle-droplet encounter, which has the most significant direct impact on dust capture rate, can be studied by using corresponding methods of experimental measurements, numerical and analytical modelling discussed in (Hassanzadeh et al., 2018).

After the test method is decided, a well-designed experiment is essential because the drawn results and conclusions largely depend on the collected data. Scientific experimental design method plays a crucial role in obtaining more accurate and reliable experimental data. In addition, the application of the design of experiments (DOE) method can help investigators reduce the unnecessary tests and avoid running some blind experiments, which leads to experimental cost-effectiveness. A poorly-designed experiment may result in the neglect or loss of important data or relationships, which generates false or partial conclusions. Some well-known experimental design methods, such as factorial design, fractional factorial design and response surface methods should be more widely employed in this field for finding the robust surfactant formulas.

Surfactant effectiveness is a combination of the factors including that discussed in Section 2.4. Some factors influence the ultimate effectiveness independently, while other factors have correlation effects. Most of the previous studies focused on the independent effects. However, the coal dust suppression is a complicated process, and it is necessary to investigate the combined effect of factors in a systematic way. There is no doubt that the field studies reflect the true effectiveness for the aimed surfactant. However, field studies can hardly reproduce and generate universal conclusions. On the other hand, laboratory tests can overcome the shortages of field studies, but a clear link between these two remains lacking. For example, by comparing the sink test, contact angle test, and the capillary rise test, a study has indicated that good performance in the former two tests would also show good behaviour in the field (Feldstein,

1981). However, this statement was not validated. In another study (Q. Hu et al., 1992), dust collection efficiency was found to not correlate with surface tension or contact angle, but rather correlate well with the work of adhesion for non-ionic and anionic surfactants. Thus, further study should focus on the dynamic interaction between the coal particle and water droplet in wind tunnels or well controlled onsite conditions.

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### 3 Chapter 3

#### **Evaluation of SDBS surfactant on coal wetting performance with static methods: preliminary laboratory tests**

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This chapter presents a preliminary experimental study with using a wide surfactant concentration range for the static measurement, which helps to determine the potential optimal surfactant concentration range for the followed research.

## **Abstract**

Coal dust contributes to lung diseases in coal miners. Spraying water mixed with surfactants is a method to suppress high concentration coal dust. This study presents the evaluation of the effect of SDBS surfactant on coals. Surfactant concentrations ranging from 0% to 1.0% were analysed through the characterization of coal wettability and surfactant adsorption by using sink test, capillary penetration test, drop penetration test, surface tension test and zeta potential test. Results show that SDBS surfactant greatly increased the coal wetting performance and the 0.02%-0.40% concentration was suggested as the optimum concentration range to improve coal wettability of coals.

**Keywords:** coal dust, surfactant, coal wettability, sink test

## **3.1 Introduction**

Coal dust is defined as a fine powdered form of coal, which is produced by drilling, crushing and grinding when coal is mined, processed or transported. It can usually be suspended in air or settle in mine airways and its size ranges from 1  $\mu\text{m}$  to 100  $\mu\text{m}$  (Hartman et al., 2012). High concentration coal dust contributes to two hazards: coal dust explosions and coal dust related lung diseases such as coal workers' pneumoconiosis (CWP), chronic obstructive pulmonary disease, silicosis, tuberculosis, chronic bronchitis, emphysema (Ayoglu, Acikgoz, Tutkun, & Gebedek, 2014c; Ross & Murray, 2004). These lung diseases are caused by continued exposure to high concentration of respirable coal dust with sizes finer than 10  $\mu\text{m}$  (Darling, 2011). These two hazards in underground coal mines still pose a serious risk for safety in mining and coal miners requiring comprehensive management. For example, in the USA, the prevalence of CWP increased from 2.1% in the 1990s to 3.2% in the 2000s (Joy et al., 2012; Laney & Attfield, 2010). In Queensland, Australia, more than 10 coal miners were diagnosed with CWP in 2016 and it was estimated that more than 1000 coal miners could have CWP (Peacock, 2016, March 4).

To minimize the coal dust concentration, especially for respirable coal dust, Permissible Exposure Limits (PEL) were introduced in the mining industry. For example, in the USA, a 2  $\text{mg}/\text{m}^3$  PEL of respirable coal dust was implemented by the Mine Safety and Health

Administration (MSHA) in 1972, and it was further reduced to 1.5 mg/m<sup>3</sup> in January 2015, effective from August 2016. Meanwhile, a Recommended Exposure Limit (REL) of 1 mg/m<sup>3</sup> of respirable coal dust was developed by the National Institute for Occupational Safety and Health (NIOSH) (NIOSH, 2003b; Ross & Murray, 2004). Although these exposure limits have been put in place, there are still some mine sites that cannot meet the limits (Laney & Attfield, 2010). What's worse, the research results point out that even exposed to the recommended exposure limit of 1 mg/m<sup>3</sup>, there is still a chance that coal miners can contract these lung diseases. Thus, a holistic approach is still necessary for reducing coal dust in underground coal mines.

Surfactant-containing water spray is the most common and effective way for coal dust suppression. Previous research results show that an improvement, ranging from 0% to 60%, was obtained by applying surfactant solutions compared to using water only (Hirschi et al., 2002; Douglas Pollock & Organiscak, 2007; Tien & Kim, 1997). To evaluate the effect of surfactants on coal dust suppression, dynamic and static methods, are commonly used. Dynamic methods, including the wind tunnel test and the field test, evaluate surfactants by monitoring the change in coal dust concentration before and after surfactants' addition. Static methods, comprising coal wettability tests and surfactant adsorption measurements, estimate the surfactants' effect by measuring coal wettability and surfactant adsorption. Coal wettability of coal dust describes the ability of coal surfaces to be wetted by a liquid, and it can be characterized by some parameters such as contact angle, sink time and penetration time. Coal wettability tests comprise the sink test, the contact angle test, the capillary penetration test and the drop penetration test. Surfactant adsorption refers to the ability of water of coal surfaces to adsorb surfactant molecules, and it can be measured through the surface tension test, the adsorption test and the zeta potential test. The results obtained from dynamic tests are more reliable because they are accomplished under real or simulated working conditions encountered. But it is expensive and time consuming, and the results are greatly affected by the working conditions and operating errors. Static methods are more commonly used as they are simple to conduct and cost-effective, and they evaluate surfactants indirectly through coal wettability and surfactant adsorption. Theoretically, the greater coal wettability and surfactant adsorption, the more easily coal dust can be suppressed.

The objectives of this study are as follows: firstly, to evaluate the impact of Sodium dodecyl benzenesulfonate (SDBS) surfactant on coal dust wetting performance by applying some static tests including the sink test, the capillary penetration test, the drop penetration test, the surface



tension test and the zeta potential test, and secondly, to study the correlation of those different static measurements on predicting changes of coal particle wetting characteristics by surfactant solutions with various concentrations.

## 3.2 Experimental

### 3.2.1 Materials

The coal selected for this study is a medium volatile sub-bituminous coal, which was obtained from the Premier Coal Mine in Collie, Western Australia. The coal was firstly crushed and then pulverised to finer particles by a pulveriser. Particle size distribution is shown in Figure 13 and the proximate and ultimate analysis are given in Table 3.

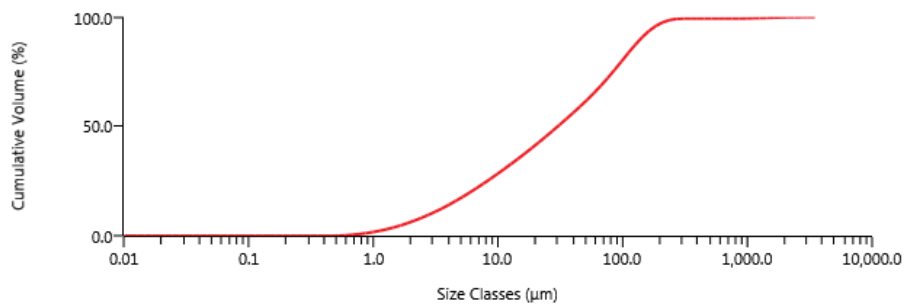


Figure 13. Coal sample particle size distribution-average result

Table 3. Proximate and ultimate analysis of Collie coal

Proximate analysis (%)					Ultimate analysis (%)			
$M_{ad}$	$V_{ad}$	$A_{ad}$	$FC_{ad}$	$C_{ad}$	$H_{ad}$	$O_{ad}$	$N_{ad}$	$S_{ad}$
19.5	35.0	4.3	41.2	56.9	3.44	14.5	0.91	0.44

SDBS, an anionic surfactant, was prepared in deionized water with various concentrations (0.05, 0.10, 0.20, 0.30, 0.40, 0.50, 0.60, 0.70, 0.80, 0.90 and 1.00%). These concentrations were selected because in previous research the range of surfactant solution selected was narrow and the number of solution concentrations tested were limited. For example, Li (Q. Li et al., 2013) applied only three concentrations of sodium dodecyl sulfate (0.05, 0.20 and 0.40%). However, a sufficient surfactant concentration is necessary before any wetting phenomenon can occur between coal particles and surfactant solution (Glanville & Haley, 1982). After the concentration reaches a certain value, known as critical micelle concentration (CMC), the adsorption of surfactant solution reaches a maximum (Porter, 1994). After CMC, higher

concentration will not apparently change the wetting phenomenon. Thus, the range from 0.05% to 1.00% was initially chosen.

## 3.2.2 Coal wettability tests

### 3.2.2.1 Sink test

The sink test was initially developed by Draves and Clarkson to describe the effect of surfactants on the wettability of cotton fibres, and later modified by Walker to determine coal wettability (Walker, JR, Petersen, & Wright, 1952). Coal wettability is characterized by the sink wetting rate that is determined by the time needed for a measured amount of coal particles to sink beneath the surfactant solution surface. Usually, the greater the sink wetting rates, the better the wettability of the coal particles.

The apparatus used for the sink test was a 150 ml beaker of 80 mm diameter, as shown in Figure 14, 0.5 g of coal particles was gently placed on the surface of 100 ml SDBS solution by using a flour sifter. The time that was required for all coal particles to sink beneath the surfactant solution surface was recorded by a stopwatch. Three sink tests were conducted for each SDBS concentration.

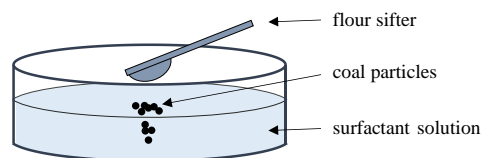


Figure 14. Schematic diagram of the apparatus for the sink test

### 3.2.2.2 Capillary penetration test

The capillary penetration test performed in this study followed the procedures recommended by Zeller (Zeller, 1983). A glass tube of height 120 mm, with an internal diameter of 8 mm and an external diameter of 10 mm, was used. A glass microfiber filter with a diameter of 10 mm was attached to the bottom of the glass tube with water resistant glue. 2 g of coal particles were loaded gently in the glass tube and then the glass tube was tapped for around 2 minutes. The final consolidated column height was approximately 60 mm. Then the glass tube was placed vertically in a beaker with the SDBS solution, and the bottom of the glass tube was immersed by around 2 mm below the solution surface as shown in Figure 15. In each run (three tests were conducted for each SDBS concentration), the glass tube was removed from the liquid and the weight gain was measured after immersion time intervals of 10, 40 and 70 minutes.

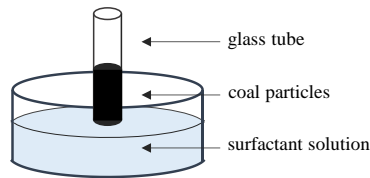


Figure 15. Schematic diagram of the apparatus for the capillary penetration test

### 3.2.2.3 Drop penetration test

The drop penetration test conducted was adapted from the procedures suggested by Chao (Chao, Peng, & Wu, 2007). The apparatus used in this test was the same as that used for the capillary penetration test. In this test, surfactant solution was injected into the top of the coal column as shown in Figure 16. The glass tube was filled with 3 g of coal particles, and the coal particles were consolidated by tapping for about 2 minutes. For most coal samples, the height of the coal particle column was around 90 mm after consolidation. The glass tube was fixed vertically, and a ruler was placed against the glass tube. Then, a certain volume of the SDBS solution was gently dropped on the top of the coal particle column by using a pipette. The penetration height from the coal column top surface to liquid front face was recorded by time intervals of 3, 5 and 10 minutes. Three penetration tests were conducted for each SDBS concentration.

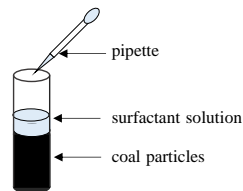


Figure 16. Schematic diagram of the apparatus for the drop penetration test

## 3.2.3 Surfactant adsorption tests

### 3.2.3.1 Surface tension

Surfactant adsorption can occur at a liquid-solid, liquid-liquid or liquid-air interface. When considering surfactant solution applied to coal particles, the adsorption occurs at the interface between the surfactant solution and coal particles. Surface tension is the elastic tendency of a fluid surface that makes it acquire the least surface area possible (Porter, 1994). Therefore, surface tension could be used to infer the surfactant adsorption. In this study, an Analite Surface Tension Meter, Model 2141 was employed to measure the surface tension of each concentration. This meter was calibrated and the test for each concentration was conducted three times.

### 3.2.3.2 Zeta potential test

Zeta potential measurements were carried out in the presence and absence of SDBS surfactant by using a Zetasizer (Zetasizer Nano Z). 0.025 g coal particles were added into a 50 mL SDBS solution then stirred for a certain time, around 15 minutes, until the particle suspension reached an equilibrium state. The Zetasizer automatically tested the zeta potential three times for each sample.

## 3.3 Results and discussion

### 3.3.1 Coal wettability

#### 3.3.1.1 Sink wetting rate

The sink time for coal particles is obtained as a function of SDBS concentration as shown in Figure 17. Coal particles did not disappear from the surface of plain water over 24 hours, however, they sank beneath the surface of 0.05% SDBS solution surface in 943 s. The sink time kept decreasing greatly to 158 s and 38 s as the SDBS concentration went up to 0.10% and 0.2%, respectively. For an SDBS concentration above 0.3% and up to 1.0%, a slight drop in the sink time was observed and the sink time remained around 10 s. The wetting rates were calculated based on the sink time, as shown in Figure 18. An increase in the sink wetting rate with increasing SDBS concentration was obtained, indicating that coal wettability was enhanced as the SDBS concentration increased in the selected range.

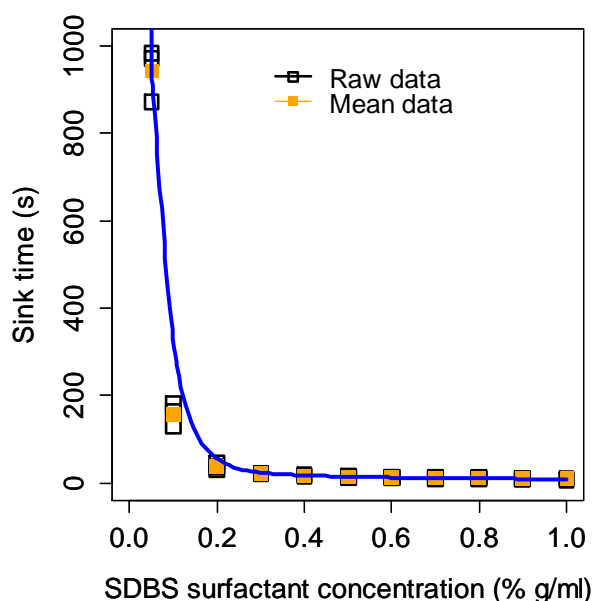


Figure 17. Sink time for Collie coal with various SDBS concentrations by the sink test

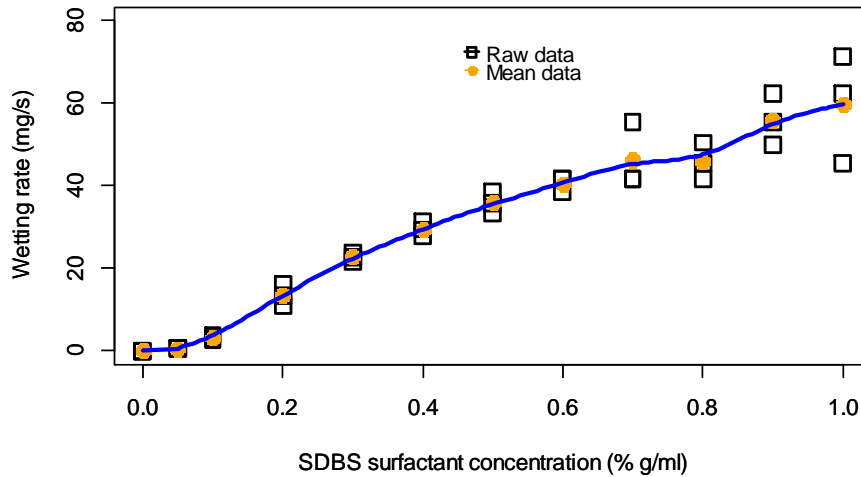


Figure 18. Sink wetting rates for Collie coal with various SDBS concentrations by the sink test

### 3.3.1.2 Capillary penetration wetting rate

The results as shown in Figure 19 indicate the weight gain of Collie coals increased in the SDBS surfactant solutions compared to in plain water. It should be noted that the greatest weight gain occurred when the SDBS surfactant concentration is 0.90%. Figure 20 demonstrates the capillary penetration wetting rates of coals in various SDBS concentrations, and that the greatest wetting rate was obtained in 0.40% SDBS concentration. The capillary penetration wetting rates rose as the SDBS concentration increased from 0.00% to 0.40%, however, it decreased slightly after the SDBS concentration increased from 0.40% to 1.00%. In addition to the SDBS concentration, test duration is another factor affecting the capillary penetration wetting rate. For all the concentrations, the initial wetting rates in the first 10 mins are much greater than those in the ranges of 10 mins to 40 mins and 40 mins to 70 mins.

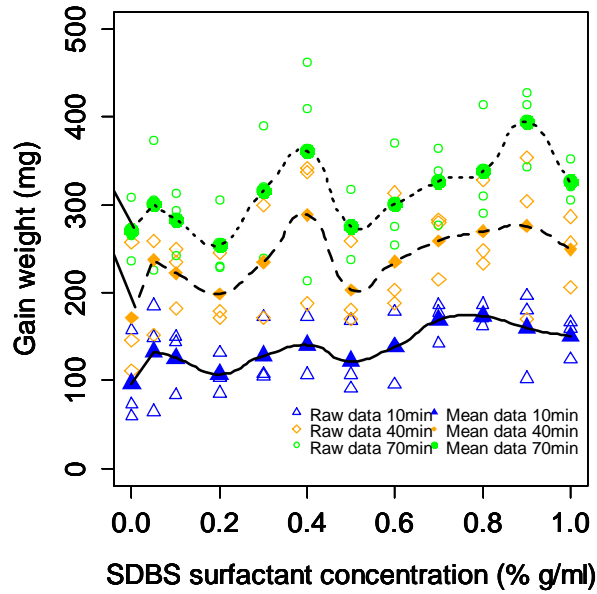


Figure 19. Weight gain for Collie coal with various SDBS concentrations

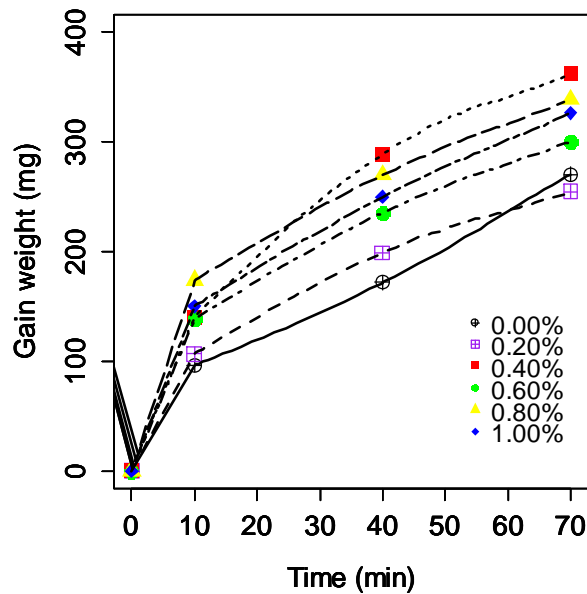


Figure 20. Capillary penetration wetting rates for Collie coal with various SDBS concentration solutions

### 3.3.1.3 Drop penetration wetting rate

Surfactant concentration has a more obvious effect on penetration height in the drop penetration tests. As shown in Figure 21, the drop penetrated heights greatly increased when the SDBS concentration increased. The drop height penetrated was only around 1 mm in first 3 minutes when the SBDS concentration was less than 0.10%. However, it increased strongly to approximately 4.5 mm after the SDBS concentration reached 0.20%, and when the concentration reached 0.30%, the drop height further increased to about 13.5 mm. Over 0.3%,

the drop height remained unchanged. Figure 22 illustrates that the wetting rate reached a maximum when the SDBS concentration was 0.4%. In the first 3 mins, the wetting rates increased greatly when the SDBS concentration increased, after that, they all increased slowly and the wetting rate of 0.4% SDBS concentration exceed those of 0.6, 0.8 and 1.0% SDBS concentrations. This result suggested that the optimum concentration may be around 0.40%; increasing the SDBS concentration greater than 0.40% results in no obvious increase in the wetting rate. A similar trend of wetting rates was observed as with that of the capillary penetration test - the initial wetting rates in the first 3 mins were much greater than the wetting rates later on in the test.

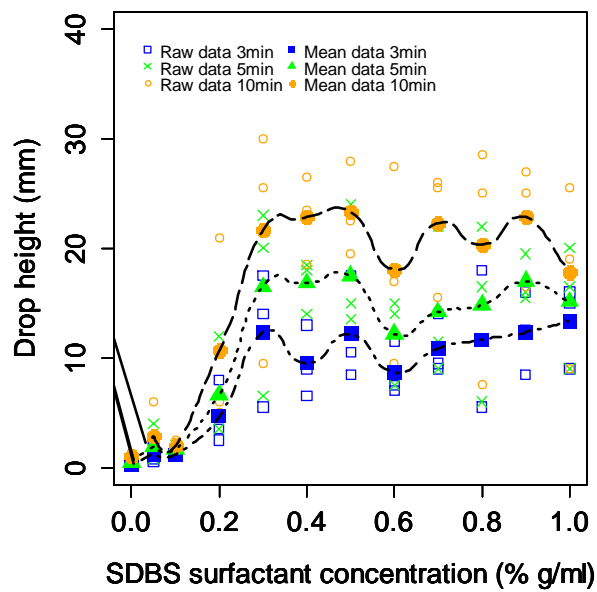


Figure 21. Effect of various concentration of SDBS on the drop penetration height

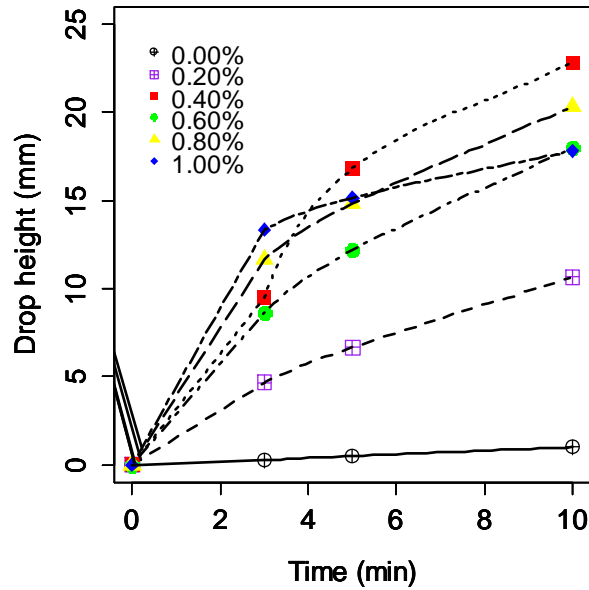


Figure 22. Drop penetration wetting rates for Collie coal with various SDBS surfactant solutions

### 3.3.2 Surfactant adsorption

#### 3.3.2.1 Surface tension

As shown in Figure 23, the surface tension of the deionised water decreases strongly after adding the SDBS surfactant. The greatest reduction occurred from the test with pure deionized water (60.6 mN/m) to the test with 0.05% SDBS solution (39.4 mN/m). Another significant observation was that as the SDBS concentration increased above 0.2%, the surface tension decreased rather slowly and remained almost unchanged.

The shape of the surface tension curve is rather similar to that of the sink time of coal particles. The Spearman's rank correlation coefficient between the surface tension and the sink time was 0.96 according to the data in Table 4. This result indicates that the surface tension and the sink time is quite strongly correlated to the SDBS concentration. Greater surface tension at the air-liquid interface prevents coal particles sinking into the solution, and the addition of the SDBS surfactant helps coal particles to sink by decreasing the surface tension. Thus, the surface tension at the air-liquid interface is one of the factors affecting surfactant adsorption when coal particles interact with surfactant solutions.



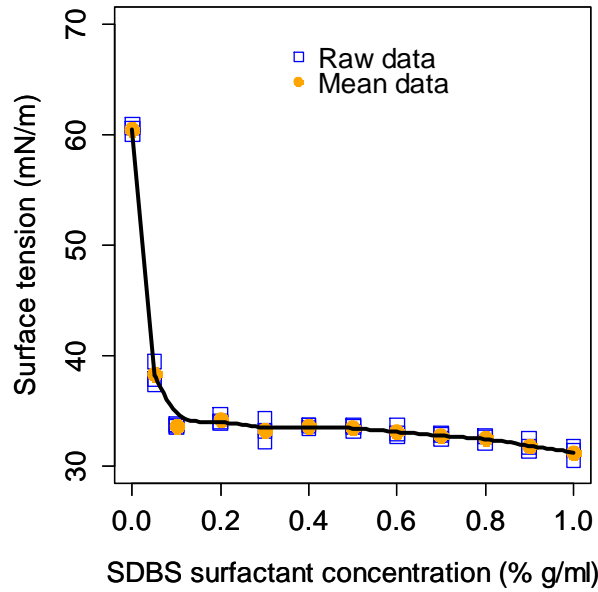


Figure 23. Effects of various concentration of SDBS on surface tension

Table 4. The surface tension and the sink time of 11 sets of SDBS concentrations

SDBS concentration %	0.05	0.10	0.20	0.30	0.40	0.50	0.60	0.70	0.80	0.90	1.00
Surface tension (mN/m)	39.4	33.8	34.6	34.3	33.7	33.6	33.7	32.9	32.7	32.4	31.7
Sink time (s)	943	158	38	22	17	14	12	11	11	9	9

### 3.3.2.2 Zeta potential

As shown in Figure 24, the values of zeta potential of coal particle suspension in the deionised water are all negative, indicating that coal particles are negatively charged in the deionized water. After adding the anionic SDBS surfactant, there is electrostatic repulsion between the coal particles' surface and the anionic heads of the SDBS surfactant molecules. Therefore, the addition of the SDBS surfactant decreased the zeta potential of coal particle suspension. The greatest fall occurred over the range from 0.00% to 0.4%; after that, the zeta potential remained almost unchanged.

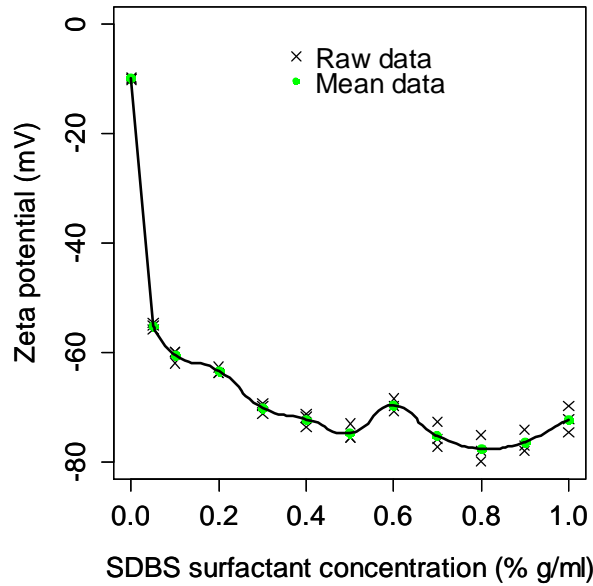


Figure 24. Effect of various SDBS concentration on the zeta potential of Collie coal

### 3.4 Conclusions

Five static tests were employed to evaluate the effect of SBDS surfactant on the wetting performance of Collie coals through coal wettability measurement and surfactant adsorption characterization. By measuring three wetting rates, experiment results suggested the addition of SDBS surfactant improved coal wettability substantially. All the wetting rates increased greatly initially with increasing SDBS concentrations, and then slight raises were observed after the concentration reached an optimum. However, the optimum concentration varies for these three tests. 0.2%, 0.40% and 0.40% were obtained from the sink test, the capillary penetration test and the drop penetration test, respectively. Thus, the range from 0.20% to 0.40% was suggested to achieve the greatest coal wettability by SDBS surfactant for Collie coals. A similar optimum concentration was noted by the surface tension test and the zeta potential test. The greatest drops of the surface tension and the zeta potential were observed when the SDBS concentration was 0.20% and 0.40%, respectively.

Among the five static tests, the results from the sink test and the surface tension test show a rather close correlation with the Spearman's rank correlation coefficient of 0.96. Thus, the surface tension between surfactant solution and coal particles is an important factor that affects the sink time. The sink time goes down with decreasing surface tension. Both the sink time and the surface tension remain almost unchanged after the SDBS concentration reaches about

0.20%. Results of the capillary and drop penetration tests show that both test duration and surfactant concentration have an impact on the coal wetting phenomenon. The longer the test duration, the more weight gained and the larger the height penetrated, but the initial wetting rates are greater than those later on in the test.

Among these static test methods, the sink test is the simplest and easy to perform, and the results are more reliable than other tests. For the capillary penetration test and the drop penetration test, it is difficult to prepare uniform coal beds in glass tubes, resulting in different porosities of the coal beds. However, the porosity of the coal bed is one of the important factors determining the penetration wetting rates. The surface tension test only involves the surfactant solution, thus, the results are not relevant enough when considering coal particles' properties.

This study presents some preliminary results of these static tests. Further work will be done, including investigation of the effect of coal properties on the surfactant function and evaluation of surfactant function through some dynamic tests.

## **Acknowledgments**

The authors would like to thank the technicians Mujesira Vukancic and Anusha Kumara at Curtin University for their invaluable help with the experiments.

## 4 Chapter 4

### **Characterization of coal particles wettability in surfactant solution by using four laboratory static tests**

The work in this chapter has been published on the journal of Colloids and Surface A: Physicochemical and Engineering Aspects.

It was entirely written by Yinping Chen and reviewed by Dr. Guang Xu.

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Based on the preliminary result from Chapter 3, this Chapter systematically analyses the reliability of four static tests includes the contact angle measurement, the sink test, the capillary rise test and the drop penetration test to characterize the coal wettability.

## **Abstract**

Coal dust is a health threat to underground coal miners, and the current common practice is to use water spray to control it. The coal wettability plays a significant role in studying the effectiveness of coal dust suppression, and the addition of surfactant to water can greatly improve the coal wettability. The aim of this paper is to evaluate the reliability of four methods that are commonly used to quantify the coal wettability in present of surfactant, they are the sessile drop measurement, the sink test, the capillary rise test and the drop penetration test. The advantages and disadvantages of these four methods were detailed. The results indicated that the sink test is the most reliable method in terms of its simplest operation and the highest accuracy. It also has the ability to quantitatively compare the wettability of different coal particle sizes, which the other three methods failed to identify. The addition of surfactant resulted smaller contact angles, less sink time, less drop penetration time and more capillary rise weight gains compared with using water only, which indicated that the coal wettability is improved by surfactants. Of the two studied surfactants, SDBS performed better than SDS. This study provided a guideline on both evaluation method and surfactant selection when studying the wettability of coal particles.

Keywords: Coal wettability; Surfactant; Contact angle; Coal dust suppression; Sink test

## **4.1 Introduction**

In the presence of some immiscible fluids, the tendency of one liquid to adhere to, or spread on a solid surface is referred to the wettability (Crain, 2000). The wettability involves the interaction between liquid phase, solid phase and gas phase. Coal wettability is one of the most important surface properties of coal. It reflects the ability of coal surface to contact with liquid resulting in an interaction of intermolecular (Cybulski, Malich, & Wieczorek, 2015). Understanding the coal wettability is crucial for the development of efficient coal dust suppression system by using water spray.

The coal wettability is generally pictured in terms of the contact angle of a liquid on the coal surface. The contact angle, shown as in Figure 25, is formed by the interaction of the liquid-solid interface and liquid-air interface. Geometrically, it is obtained by applying a tangent line

from the contact point along the liquid-air interface in the liquid profile (Yuan & Lee, 2013b). A small contact angle generally implies high wettability. For coal briquettes, the contact angle can be acquired by using the captive bubble or the sessile drop techniques (Jaroslaw Drelich, 2013; Gosiewska et al., 2002). These two techniques both require a flat and smooth coal surface. For fine coal particles, it is quite challenging to measure the contact angle on a single particle surface, and the measured result of one particle cannot sufficiently represent the majority particles' wettability (Chau, 2009). Although it is possible to create a compacted coal surface by pressing the coal particles into pellets or discs (R. Crawford & D. Mainwaring, 2001; Guy et al., 1992), the surface smoothness and flatness of the compacted pellets is not comparable to that of the polished coal briquette, thus resulting large measurement deviations (Galet et al., 2010). To overcome these difficulties, other indirect methods are commonly used including the sink test, the capillary rise test and the drop penetration test. These indirect methods characterize coal wettability by parameters such as the sink time, the capillary rise weight gain and the drop penetration time.

Coal is a hydrophobic mineral and its wettability is poor in water, which is one of the major reasons leading to low coal dust suppression efficiency. Numerous previous studies indicated that the coal dust suppression efficiency can be greatly improved by adding surfactants to water used for the spraying system (W. Ren, Wang, Guo, & Zuo, 2014; Tessum et al., 2014; K. Wang et al., 2014). This is believed to mainly attribute to the improvement of the coal wettability. But some inconsistent conclusions about the coal wettability in surfactant solutions were found in previous studies as different test methods employed. For example, Zhou (G. Zhou et al., 2016) found worse coal wettability by non-ionic surfactant based on contact angle measurement. However, Tien and Kim (Tien & Kim, 1997) found non-ionic surfactant performs better in terms of the sink test results. This paper aims to systematically analysis the reliability of these four test methods including the direct method (the contact angle measurement) and the indirect methods (the sink test, the capillary rise test and the drop penetration test) to characterize the coal wettability. Advantages as well as disadvantages of each method were presented through experimental comparison and analysis. Also, the impact of surfactant and coal particle size on coal wettability was investigated. The findings and recommendations in this paper provide practical guidance for researchers to use the proper method for developing effective coal dust suppressant agent.

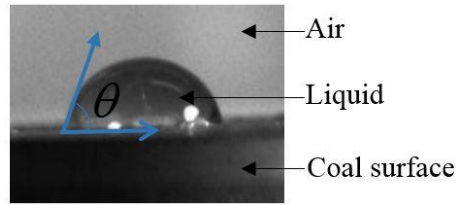


Figure 25. One example of the contact angle measurement

## 4.2 Experimental

### 4.2.1 Materials

The coal sample, a medium volatile subbituminous coal, was obtained as lumps from the Premier Coal Company in Collie, Western Australia. The proximate and ultimate analysis of the coal sample are shown in Table 5. Two anionic surfactants, Sodium dodecylbenzenesulfonate (SDBS) and Sodium dodecyl sulfate (SDS), were used in this study. They are supplied by Sigma Aldrich Pty. Ltd.

The preparation of coal samples followed the procedures of American Society for Testing and Materials (ASTM) (ASTM, 2012a, 2012b, 2015). A Jaw Crusher was first used, and then the crushed coal was pulverised for 2 minutes by a pulveriser. Pulverised coal sample was dried in an oven at 35° C until the weight loss is no more than 0.1% per hour. The dried coal particles were sieved into three ranges: 0~38, 38~53 and 75~90  $\mu\text{m}$ . A 0.001 g accuracy balance was used to monitor the quality of sieving. For each sieve, the sieving process was considered thorough when only less than 0.5% of the coal particle passes in a 5 minutes sieving period. The prepared samples were stored in glass containers sealed with rubber gaskets.

Surfactant solutions were prepared in deionized water by using a magnetic stirrer, and the surface tension of the prepared solution was measured by the Analite surface tension meter, model 12141.

Table 5. Proximate analysis of Collie Coal

Proximate analysis (%)					Ultimate analysis (%)			
$M_{\text{ad}}$	$V_{\text{ad}}$	$A_{\text{ad}}$	$FC_{\text{ad}}$	$C_{\text{ad}}$	$H_{\text{ad}}$	$O_{\text{ad}}$	$N_{\text{ad}}$	$S_{\text{ad}}$
19.5	35.0	4.3	41.2	56.9	3.44	14.5	0.91	0.44

( $M_{\text{ad}}$ : moisture,  $V_{\text{ad}}$ : volatile matter,  $A_{\text{ad}}$ : ash,  $FC_{\text{ad}}$ : fixed carbon,  $C_{\text{ad}}$ : calorific value,  $H_{\text{ad}}$ : hydrogen,  $O_{\text{ad}}$ : oxygen,  $N_{\text{ad}}$ : nitrogen,  $S_{\text{ad}}$ : sulfur)

## 4.2.2 Laboratory tests

The test methods used in this study are shown in Figure 26. The direct parameter, the contact angle, was obtained by using the sessile drop technique, and the other three indirect parameters were measured by employing the sink test, the capillary rise test and the drop penetration test. In this study, each test was repeated three times to obtain more accurate data.

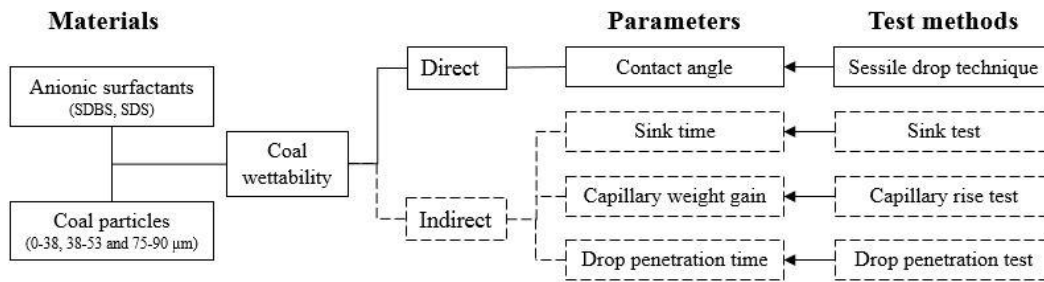


Figure 26. Test methods to evaluate the coal wettability

## 4.2.3 Sessile drop technique

The sessile drop technique is a direct method to measure the contact angle that is formed by the liquid on a solid surface, and it is considered as one of the most common methods to determine the particles' wettability (Alghunaim, Kirdponpattara, & Newby, 2016). In this study, 1 g coal particle was placed in a mould with a 25 mm inner diameter, and a pressure of 30 MPa was loaded on the coal particle for 2 minutes to compress the coal powder into a pellet. A drop of 0.5 mL surfactant solution was gently placed on the pellet by using a dropper. The profile of the sessile drop was photographed by a camera (Fujinon, HF8XA-1), and the contact angles were obtained by using a computer program. One measured contact angle profile example is shown in Figure 25.

### 4.2.3.1 Sink test

The sink test is regarded as a conventional measurement method to analyse the effect of surfactant on the coal wettability (Cybulski et al., 2015; Tien & Kim, 1997). It was firstly proposed by Draves and Clarkson and later was modified by Walker which can be applied specifically to coal powders (Draves & Clarkson, 1931; Walker, JR, et al., 1952). In this test, 0.5 g coal particle was gently placed on the surface of the surfactant solution, and the sink time is defined as the time that required for all coal particles to disappear from the surfactant solution surface. Complete wetting is defined as the time required for all coal particles break through the solution surface. The measurement was stopped when the sink time is greater than the 1500 s, which assumes that the complete wetting fails to occur.



#### 4.2.3.2 Capillary rise test

To characterize the coal wettability by using the capillary rise test, 2 g coal particle was packed into a glass tube (100 mm high, 8 mm inner diameter). A glass microfiber filter (Whatman, supplied by Sigma Aldrich Pty. Ltd) was attached to the bottom of the tube by the water-resistant glue. In order to obtain a uniform coal particle column in the tube, a modified method by Galet was employed (Galet et al., 2010). In that method, coal particles were filled into the glass tube manually, and the tube was placed in a laboratory centrifuge to achieve uniform compaction. The rotate speed was fixed as 2000 rpm for 10 minutes. This method allows to control the porosity of the coal column to a certain degree and to produce uniformly packed coal particle columns. After the sample was prepared, the glass tube hung on the hook below an electronic balance. As shown in Figure 27, 60 ml surfactant solution was placed in a glass beaker that was slowly lifted to allow the bottom of the glass tube immerses into the solution for 2 mm. The weight gain was continuously recorded by the electronic balance.

Equation 16 holds in the capillary rise test (Alghunaim et al., 2016; Galet et al., 2010; Lazghab, Saleh, Pezron, Guigon, & Komunjer, 2005; Susana et al., 2012), where  $k$  is a constant which is determined by the properties of the packed powder,  $\rho$  is the liquid density,  $r_{LV}$  is the liquid-air interfacial tension,  $\theta$  is the contact angle and  $\mu$  is the liquid viscosity. It can be noticed that the squared weight gain ( $w^2$ ) has a linear relationship to the measured time ( $t$ ), and the coefficient (slope) of the equation is inversely proportional to the contact angle. Large slope means small contact angle with better wettability and vice versa.

$$\frac{w^2}{t} = k \frac{\rho^2 r_{LV}}{\mu} \cos \theta \quad \text{Equation 16}$$

Figure 28 shows a typical curve of the squared weight gain versus time in the capillary rise test. There are three stages: In stage I ( $t_0$  to  $t_1$ ), weight gain is negative. One potential reason is that the liquid exerts an upward buoyant force on the bottom of the glass tube, and the force equals to the weight of liquid being displaced by the part of the glass tube that immersed in the liquid. The value of the weight gain increases to 0 when the weight of absorbed liquid equals to the buoyant force. The surfactant solution continue to rise in the coal powder column in stage II, which is the most important index because the rate of weight gain directly reflect contact angle and wettability. The final stage III ( $t_2$  onward) is when the capillary action reaches to maximum, and the weight gain keeps almost no change. Only the first and second stage were observed in this study due to time limitation.

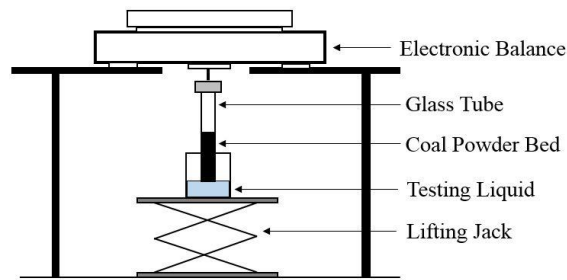


Figure 27. Experimental setup for the capillary rise test

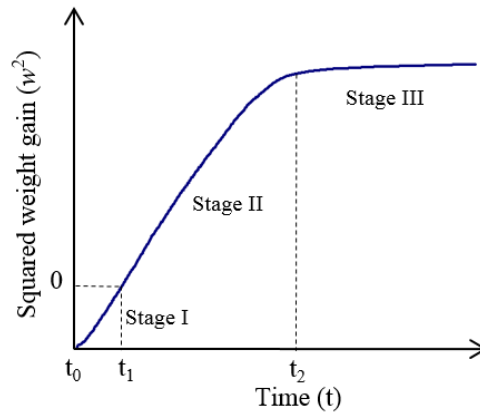


Figure 28. A typical curve of capillary rise rate for coal particles

#### 4.2.3.3 Drop penetration test

In the drop penetration test, the glass tube used and the sample preparation procedures are the same with that in the capillary rise test. The glass tube was fixed vertically next to a paper ruler. 1 ml surfactant solution was filled gently on the top surface of the packed coal particles using a dropper. The time needed for the surfactant solution to penetrate to the pre-measured height was recorded. The apparatus used for the drop penetration test is shown in Figure 29.

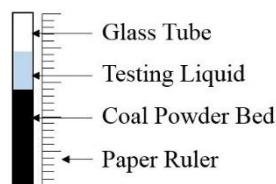


Figure 29. Apparatus for the drop penetration test

## 4.3 Results and discussion

### 4.3.1 Coal wettability characterized by different methods

#### 4.3.1.1 Contact angle

Figure 30 indicates several images taken from the sessile drop experiments when using pure deionised water, 0.10% SDS and 0.10% SDBS surfactant solution. It can be noticed that for all cases, the shape of the water drop continuously became flatter. This phenomena was also reported in other studies (Q. Li et al., 2013). This is because that the solution drop is adsorbed into the coal pellet due to the thermodynamic and kinetic contributions (Lazghab et al., 2005; Susana et al., 2012). However, the actual contact angle, the Young's contact angle, can only be formed when the liquid drawing kinetics into the particle is low enough then the force balance that support the droplet shape and reach a final equilibrium. This equilibrium was not achieved when using coal particles pellets, and the solution drop was totally absorbed by the coal pellet eventually. Thus, the contact angle of coal particle pellets measured by the sessile drop technique can only be considered as the apparent contact angle instead of the actual angle, thus can only be used as a qualitative method (Alghunaim et al., 2016).

Figure 30 also quantitatively shows that the coal wettability of surfactant solutions is better than the deionised water. The solution drop shapes are flatter at any time for the deionised water compared to the other two surfactant solutions. The solution drops are fully penetrated to the coal pellet at around 6 s for the surfactant solutions, but still, a large water drop exists at 6 s for the deionised water. These indicate that the contact angles of coal pellets in presence of surfactant liquid drop are smaller than that of the deionised water, which means the coal wettability is improved by the addition of surfactants.

Figure 38 illustrates the experimental mean contact angle and its standard error for each concentration of the two surfactants liquid drop at the initial contact of 0 s. The mean contact angle is an indicator for coal wettability, larger contact angles imply the wetting of the coal surface is unfavourable and smaller ones mean better wetting performance (Yuan & Lee, 2013b). It can be observed that contact angles are reduced when the surfactants concentration increases.



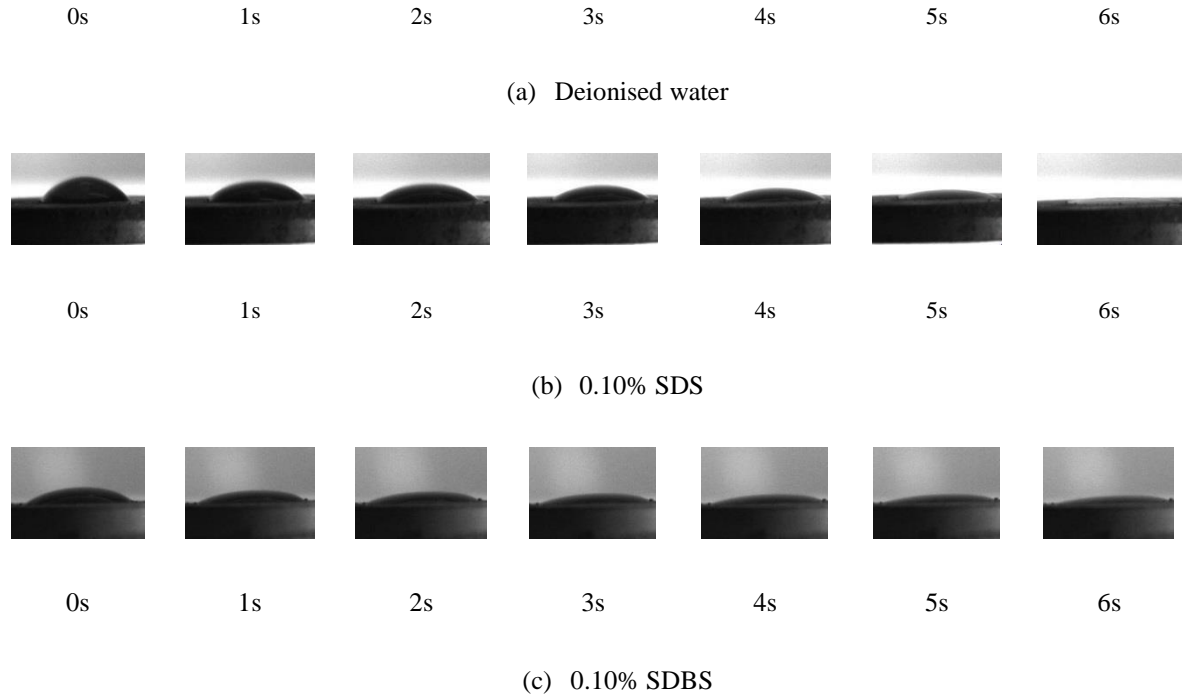


Figure 30. Images of sessile drops on compressed coal particle (0~38  $\mu\text{m}$ ) pellets

#### 4.3.1.2 Sink time and surface tension

The addition of surfactants reduces the water's surface tension as well as the sink time of coal particles. The change of the surface tension and the sink time for 0~38  $\mu\text{m}$  particle sizes are plotted in Figure 31 against the concentration. A steep reduction in both the surface tension and the sink time occurred at the when the SDBS concentration increased from 0.00% to 0.20%, and then the values kept almost unchanged as surfactant concentration further increases. The surface tension is reduced to 39.40 mN/m from 72.80 mN/m by adding as little as 0.05% SDBS surfactant, and it is further reduced to 33.80 mN/m as the concentration increased to 0.10%. After that, it remains almost constant even with the concentration up to 0.20%.

The surface tension is the primary factor that influences the sink time. Table 6 shows that the Spearman's rank correlation coefficients between the surface tension and the sink time are 0.90, 0.90 and 0.93 for coal particle sizes of 0~38  $\mu\text{m}$ , 38~53  $\mu\text{m}$  and 75~90  $\mu\text{m}$ , respectively. This indicates that the sink time is well correlated with the surface tension.

The surface tension is the primary factor that dominates the coal wetting. Surface tension of less than 45 mN/m (the critical surface tension) is needed for a spontaneous spreading of the liquid over the coal surface (Parekh & Aplan, 1978). This means in the sink test, a complete wetting is not likely to occur when the surface tension of the test solution is greater than 45 mN/m. For example, in this study, coal particles remain on the deionised water surface after 24

hours. This is because the deionised water has a high surface tension of 72.8 mN/m, which is difficult for coal particles to penetrate (Beattie et al., 2014). A non-complete wetting is one of the reasons that lead to low dust suppression efficiency. Figure 31 also indicates that all the complete wettings occur with the surface tension lower than 45 mN/m for 0 ~38  $\mu\text{m}$  coal particles.

After the surface tension reaching the critical value, it is not the dominating factor and other factors, such as coal properties, start to affect the coal wetting performance (Parekh & Aplan, 1978) (Howard W Kilau, 1990). In other words, due to other factors, some complete wetting may fail to happen although the surface tension is lower than 45 mN/m. For example, as shown in Figure 31, non-complete wetting occurred for 0 ~38  $\mu\text{m}$  coal particles in the 0.05% SDBS solution although its surface tension is 39.40 mN/m which is below 45 mN/m. This maybe because other factors affect the wetting process.

Table 6. The surface tension and the sink time in presence of SDBS with different concentrations

SDBS concentration (wt%)	Surface tension (mN/m)	Sink time (s)		
		0~ 38 $\mu\text{m}$	38~53 $\mu\text{m}$	75~ 90 $\mu\text{m}$
0.00	72.80 $\pm$ 1.67	1500 $\pm$ 0	1500 $\pm$ 0	1500 $\pm$ 0
0.05	39.40 $\pm$ 1.04	1500 $\pm$ 0	1500 $\pm$ 0	1500 $\pm$ 0
0.10	33.80 $\pm$ 0.15	519 $\pm$ 4.50	80 $\pm$ 2.08	1500 $\pm$ 0
0.20	34.60 $\pm$ 0.37	108 $\pm$ 6.60	27 $\pm$ 1.52	12 $\pm$ 1.15
0.30	34.30 $\pm$ 1.05	71 $\pm$ 4.04	18 $\pm$ 0	7 $\pm$ 0.57
0.40	33.70 $\pm$ 0.15	66 $\pm$ 3.78	12 $\pm$ 0.58	7 $\pm$ 1
0.50	33.60 $\pm$ 0.26	65 $\pm$ 4.04	10 $\pm$ 0	5 $\pm$ 0.57
0.60	33.70 $\pm$ 0.50	45 $\pm$ 5.03	9 $\pm$ 0.58	6 $\pm$ 0.57
The correlation coefficient of surface tension and the sink time		0.90	0.90	0.93

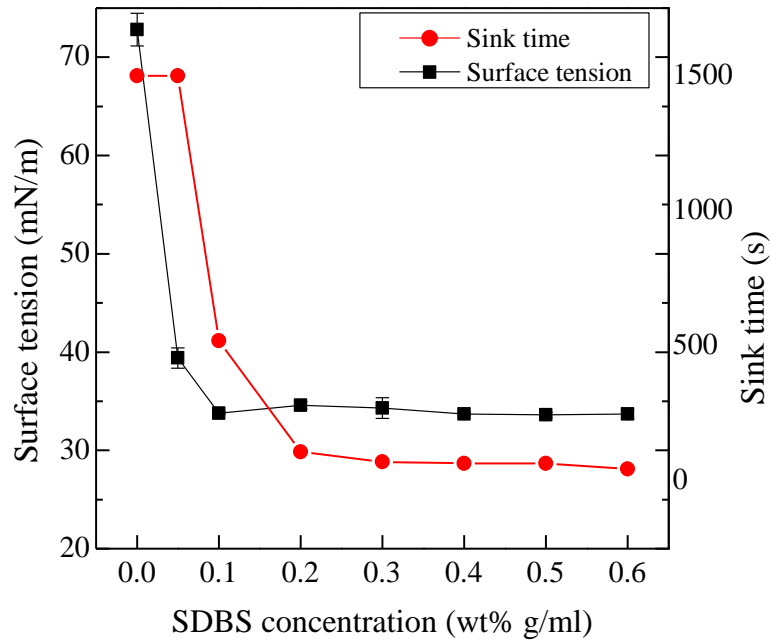
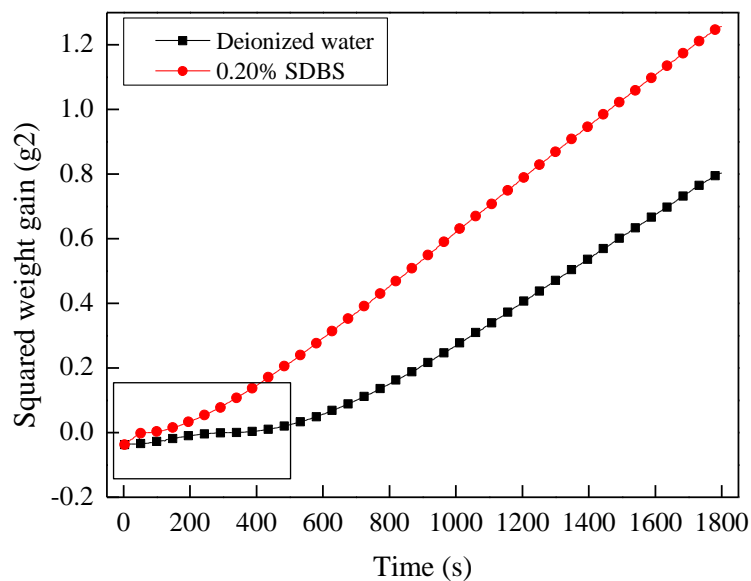


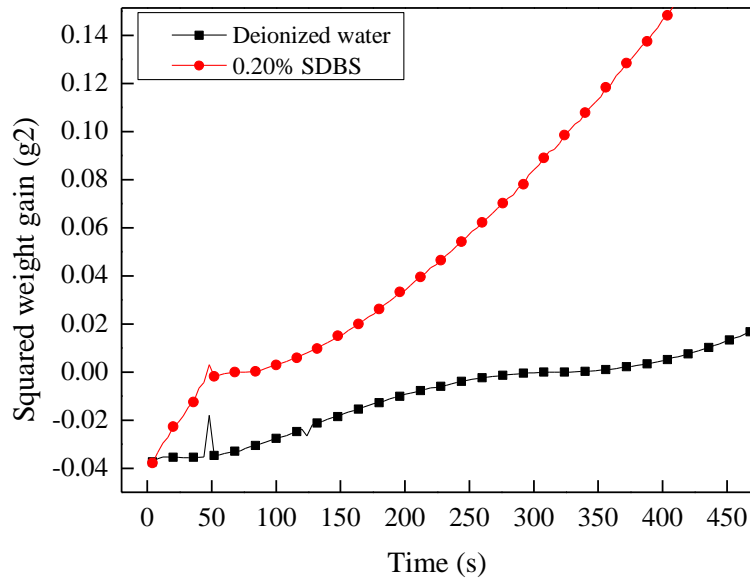
Figure 31. Surface tension and sink time versus SDBS with various concentration for 0~38  $\mu\text{m}$  coal particles

#### 4.3.1.3 Capillary weight gain

For the tested liquid of water and SDBS, the mass of liquid penetrated the coal column followed the first two stages of the typical curve. Figure 32 (a) gives the results of the squared mass of deionised water and 0.20% SDBS taken by capillarity as a function of time for the whole test duration. The experiments reveal that SDBS enhances the coal wettability. It can be seen in Figure 32 (b) that in the first 450 s, for SDBS only 100 s were used to pass stage I while more than 300 s was used for water. In addition, the slope of the second stage of SDBS ( $8 \times 10^{-4}$ ) is steeper than that of water ( $5 \times 10^{-4}$ ).



(a) The capillary rise results for 0-38  $\mu\text{m}$  coal particles in the duration of 1800 s



(b) The magnified version of the capillary rise results of the first 450 s in Figure 8 (a)

Figure 32. The capillary rise results for 0-38  $\mu\text{m}$  coal particles

#### 4.3.1.4 Drop penetration time

The behaviour of a liquid penetrates through a compressed coal column in a glass tube is primarily due to the gravity of the liquid and the wettability of coal. In general, a quick penetration rate suggests better coal wettability and vice versa.

The results of water and SDBS to penetrate to different depths are presented in Figure 33. It can be noticed that the liquid penetrated much faster at the beginning than the latter period of the experiment both for water and surfactant solutions. For example, 124 s was used for SDBS to penetrate the first 10 mm, while 226 s, 363 s and 560 s were used for the second 10 mm, third 10 mm and fourth 10 mm, respectively. This means the penetration ability was weakened during the penetration process. One possible reason leads to this maybe the decrease of the saturated liquid height. The gravity of the liquid provides the major penetration force, but the effective saturated liquid height decreases with time due to the coal adsorption which lead to slower penetration rates.

The penetration ability of surfactant solution is better than that of water. Figure 33 shows that compared to deionised water, the time needed for surfactant solution to penetrate down to 5 mm, 10 mm, 20 mm, 30 mm and 40 mm are all less than that of water, which indicates that surfactants enhance the coal wettability. Figure 33 indicates that the penetration time becomes shorter when the surfactant concentration increases from 0.00% to 0.30%, and reaches the

minimum penetration time when the concentration is 0.30%. Instead of further shortness on penetration time, an increase was observed as the concentration is over 0.30%. This indicates that increasing the concentration of more than 0.30% does not help to further improve the coal wettability measured by the drop penetration test.

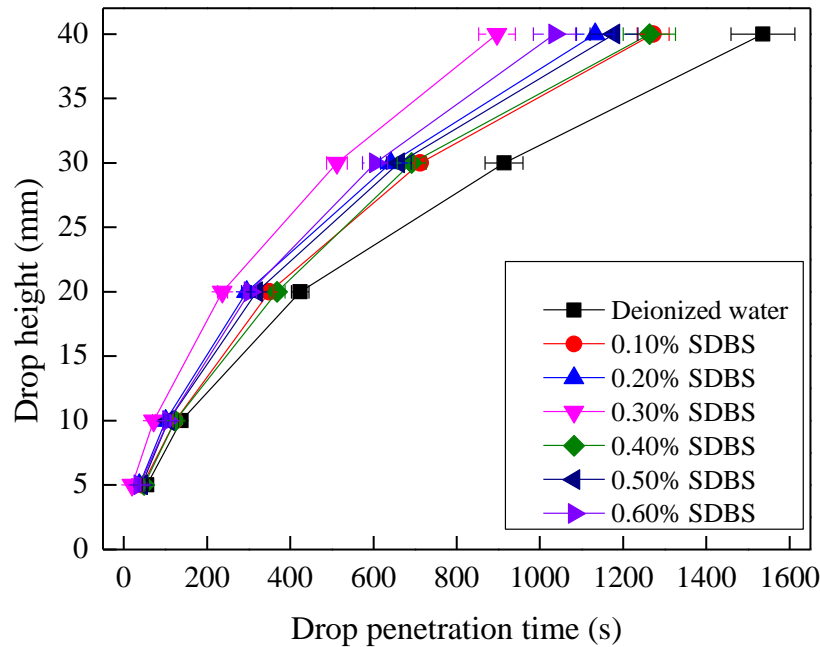


Figure 33. Drop penetration time for SDBS with various concentration in 0~38  $\mu\text{m}$  coal particles

### 4.3.2 Evaluation methods Comparison

Table 7 compares the four methods utilized in this study to determine the coal wettability. Although the sessile drop technique is the only direct contact angle measurement method, there are challenges when applying it to the porous and rough compressed coal particle pellets. It is hard to capture the contact angle for higher concentration surfactant solutions because the solution drop is absorbed by the coal pellet quickly. This challenge was also reported in several previous studies (A. Roman-Gutierrez, J. Sabathier, S. Guilbert, L. Galet, & B. Cuq, 2003; J. Shang, M. Flury, J.B. Harsh, & R.L. Zollars, 2008; V. Karde & C.Ghoroi, 2014). In addition, during the sample preparation process, the high compression pressure applied may alter the coal surface properties and structure because of molecular orientation and polymorphic changes (A. Chawla, G. Buckton, K.M.G. Taylor, J.M. Newton, & M.C.R. Johnson, 1994; X. Pepin, S. Blanchon, & G. Couarraze, 1997), thus, the wettability of the compressed coal pellets may not reflect that of the original coal particles. Therefore, the apparent contact angle obtained by this method can only be used qualitatively for coal wettability comparison. In this study, the sessile drop test did not apply to larger size ranges of 38~53 and 75~90  $\mu\text{m}$  because of two



reasons. The first one is it is difficult to compact the larger coal particles into a pellet under the compress conditions used in this study. The second one is it is challenging to capture the contact angle on the larger coal particle pellet because it disappears from the pellet surface rapidly. Regarding these two points, the sessile drop measurement is not suggested to use for larger coal particles.

The sink test is widely used for wettability evaluation for its simplicity and reproducibility. The required equipment is easily accessible in a laboratory, and the coal particles are not compressed thus maintains their original structure and reflects the true wettability. However, it is difficult to quantitatively compare the coal wettability of two samples if both of them failed to occur the complete wetting or occurred in a very short time beyond the eye observation.

There are commonalities between the capillary rise test and the drop penetration test as the wettability of coal particles determined by them both depends on the packing porosity of the coal column (Alghunaim et al., 2016; Galet et al., 2010). The centrifuge technique is used to prepare coal samples, and it allows to produce the uniform coal columns with the same porosity for the same range sizes. However, it is inapplicable to compare the wettability of different coal particle sizes by these two methods as the packing porosity varies. For example, Figure 34 illustrates in the capillary rise test, the squared weight gain versus time for three size ranges in 0.20% SDBS surfactant solution. The largest coal particles gained much less weight compared to the other two finer coal particles, and the slope of the formed curve is close to 0. This may be related to the larger porosity of the larger coal column thus the capillary rise action is not as obvious as those of finer coal particles. Also, the capillary rise test requires longer measuring time, and the coal column could collapse because the filter paper could fall off from the glass tube bottom after long a period of soaking. For example, in this study, among the 90 test coal column samples, 6 samples ( around 7% of the samples) ended up with the filter papers falling off, and most of them fell off during the latter stage of the experiment, resulted in a duplicated test was performed. For the drop penetration test, in addition to the finer coal particles have worse wettability that leads to longer penetrate time, the potential structures of packed coal column may also contribute to this result. Under the same compressing conditions, the compressed structure of 0~38  $\mu\text{m}$  coal column is expected to be more uniform and the void size is smallest, thus it takes more time to penetrate through. The larger particles tend to create larger voids in the coal column, which leads to the solution front penetrate down faster. Also, because of the void size distribution, it is changeling to determine a reliable penetration depth for the larger coal particle size. As shown in Figure 35 and Figure 36, for the larger coal particle

size, rang 75~90  $\mu\text{m}$ , the penetration front was uneven and non-uniform due to the wall effect that makes it hard to accurately quantify. This experimental phenomenon was also observed by Kirdponpattara (Kirdponpattara, Phisalaphong, & Newby, 2013). Therefore, these two methods are not recommended to comparing wettability of different sizes of coal particles.

The experiments results show that the drop penetration test fails to distinguish the performance of SDBS and SDS due to large error. Figure 37 illustrates that the penetration time is shorter for SDS when the concentration is 0.10%, 0.50% and 0.60%, and it became longer when the concentration is 0.20%, 0.30% and 0.40%. These inconsistent experimental data indicate that the drop penetration test is not suitable to evaluate the surfactants' effectiveness on the coal wettability.

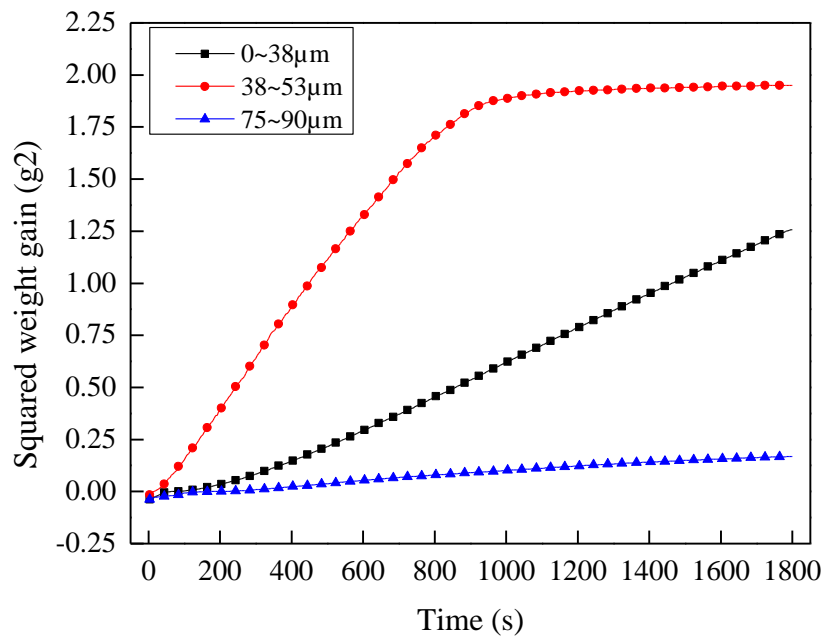


Figure 34. Squared of weight gained by coal particle with three size ranges in 0.20% SDBS concentration

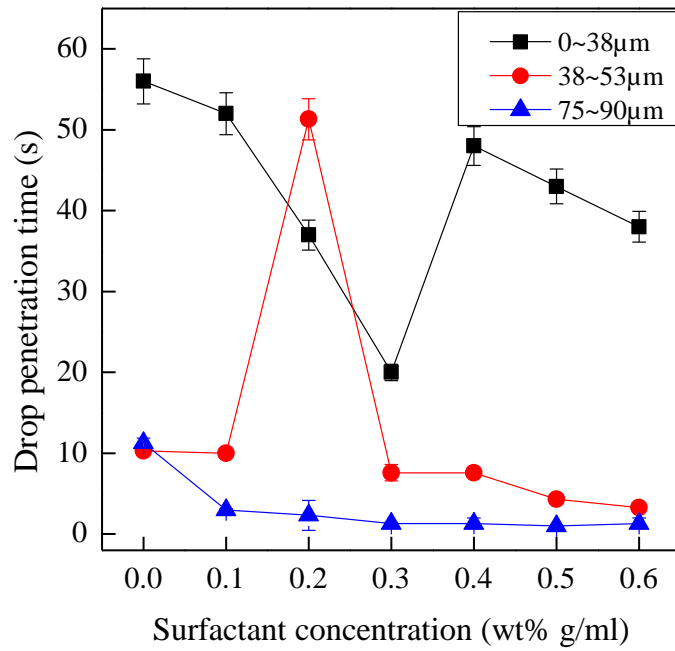


Figure 35. Drop penetration time to 5mm for different size coal particles using SDBS surfactant

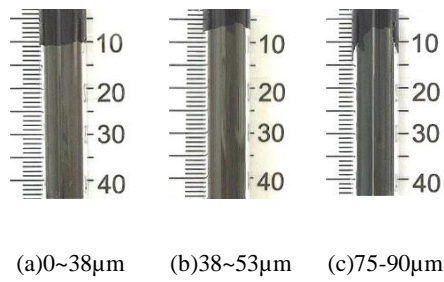


Figure 36. Images of surfactant penetration for three particle sizes

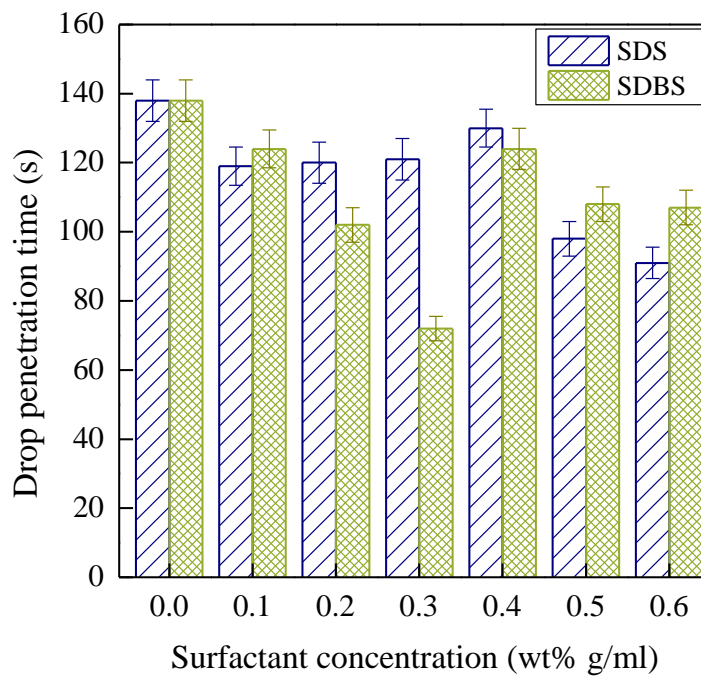


Figure 37. Drop penetration time to 10mm of coal particle (0~38  $\mu\text{m}$ ) for SDBS and SDS

Table 7. Four methods used for evaluating coal wettability

Method	Sample	Advantages	Disadvantages/applicability limits	
Direct method	Sessile drop measurement	Compressed coal particles pellets	Need small amount of test solution	<ul style="list-style-type: none"> <li>• The unknown porosity and roughness of the compressed surface leads to a contact angle hysteresis</li> <li>• Liquid could penetrate to the pellet very quickly, leading to measurement challenge</li> <li>• The measurement accuracy is determined by the camera and software</li> <li>• The applied pellet compress pressure dramatically influence the measuring results</li> </ul>
Indirect methods	Sink test	No special preparation method required	Operationally simple,	<ul style="list-style-type: none"> <li>• Difficult to compare coal wettability of two samples if complete wetting does not occur at all, or wetting occurs in very short time beyond the eye observation</li> </ul>
	Capillary rise test	Coal particles packed in a glass tube with a filter paper at the bottom	N/A	<ul style="list-style-type: none"> <li>• Measuring time could be long</li> <li>• Filter paper may fall off from the bottom due to longer soaking time</li> <li>• Not suitable for larger size coal particles as the capillary rise action are not apparent due to the larger packing porosity</li> </ul>
	Drop penetration test	Coal particles packed in a glass tube with a filter paper support at the bottom	N/A	<ul style="list-style-type: none"> <li>• The wall-effect leads to difficult in determining the penetrate depth</li> <li>• Operationally difficult to place the liquid on the surface of the compressed coal column with minimum disturbing</li> </ul>

### 4.3.3 Factors influencing coal wettability

#### 4.3.3.1 Surfactant

Two typical surfactants SDBS and SDS were selected to evaluate the four methods for determining coal wettability. The results of the four methods consistently indicate that both surfactants improve the coal wettability compared with use water only. For example, Figure 38 to Figure 41 show the improved wettability in terms of the decrease on the contact angle, the drop on the sink time and the surface tension, and the increase on the capillary rise weight gain.

Experimental results reveal that SDBS enhance the coal wettability better than SDS. Firstly, in the sessile drop test, Figure 38 shows that the contact angle changing trend was similar for SDS and SDBS when the concentration was less than 0.30%, while smaller contact angles were

observed for SDBS when the concentration is over 0.40%. Secondly, in the sink test, Figure 39 indicates that the complete wetting starts to occur when SDBS concentration is 0.05%, but a much higher concentration of 0.40% is needed for SDS. The sink time for SDBS is also shorter than that of SDS (the blue line is under the red line). Thirdly, in the capillary rise test, Figure 41 shows that, compared to SDS, a larger weight gain was obtained by SDBS at the concentration of 0.20% and 0.40%. Also, Figure 40 clearly shows that SDBS can reduce the surface tension more effectively than that of SDS. Overall, the SDBS is recommended as a better choice to wet coal particles.

Although surfactant concentration plays a significant role in the improvement of the coal wettability, the experiment results indicate an inconsistent result on the effect of surfactant concentration on the coal wettability. The sessile drop test indicates that the best coal wettability was observed at the highest concentration for both two surfactants. As shown in Figure 38, the smaller contact angles were observed with the increasing surfactant concentration over the whole test concentration range. The same result was also observed in the sink test for the SDS surfactant. However, the sink test for the SDBS suggested that increasing the concentration does not help to improve the coal wettability greatly when the SDBS concentration is over 0.20%. The capillary rise test results show that the coal wettability was weakened when the concentration increased from 0.20% to 0.40%. Figure 41 shows that less weight gain was noticed at the higher concentration compared with 0.20% for both SDBS and SDS.

Different critical micelle concentrations (CMCs) for SDBS and SDS is a significant reason leading to SDBS performs better than SDS on improving coal wettability at the same test concentrations. For SDS with the molecular weight of 288.38 g/mol, the CMC is 8.2 mM (0.236 wt % g/ml) at 25 °C in pure water, and SDBS (348.48 g/mol) with a much lower CMC with 2.0 mM ( 0.07 wt % g/ml) at the same conditions (Mohamed et al., 2015). It can be observed in Figure 15 that both SDS and SDBS failed to occur the complete wetting at a concentration of 0.05% where neither of these two surfactants reached their CMCs. SDBS helped the coal particles to sink completely when the concentration is above 0.10% where its CMC was reached, while SDS remained incompletely wetting until the concentration was over 0.30% (greater than its CMC) where for the SDBS started to decrease the wettability improvement.

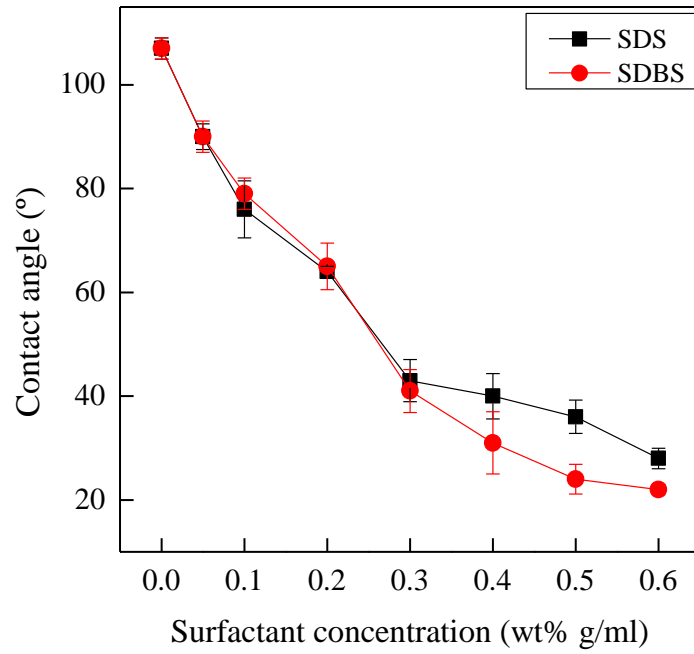


Figure 38 . Contact angles of compressed coal particles (0~38  $\mu$ m) pellets in surfactant solutions with different concentrations

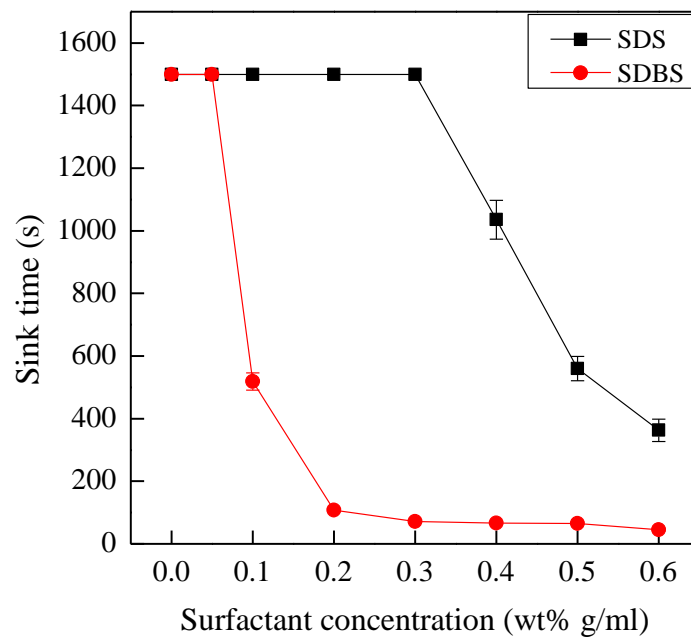


Figure 39. Sink time of coal particle within 0~38  $\mu$ m in presence of SDBS and SDS surfactant with different concentrations

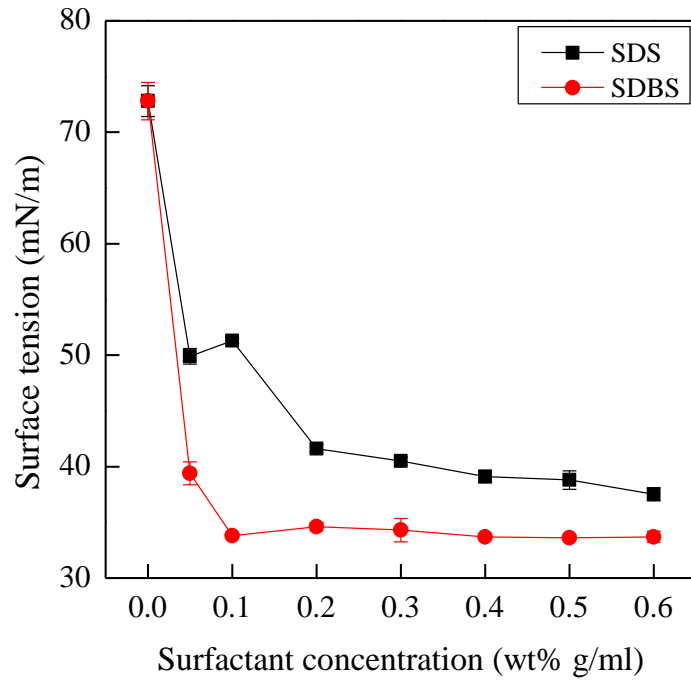


Figure 40. Surface tension of SDBS and SDS with different concentrations

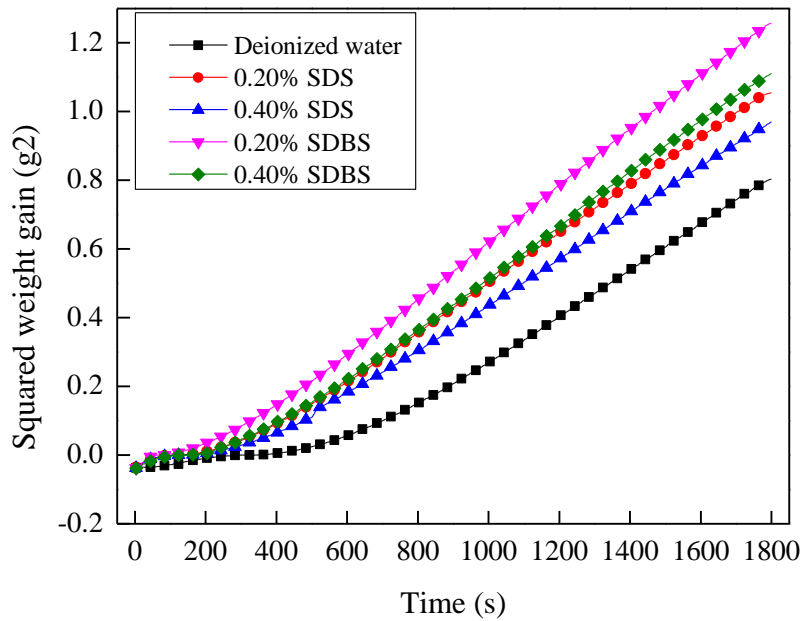


Figure 41. Squared weight gain versus time for SDBS and SDS using 0-38 $\mu$ m coal particle

#### 4.3.3.2 Particle size

Previous studies have shown that the finer coal particles have poor wettability compared with coarser ones (Glanville & Haley, 1982; Q. Li et al., 2013; Tessum & Raynor, 2017b; Wu et al., 2007; Yang et al., 2010). In this study, three size ranges of coal particles (0~38, 38~53 and 75~90  $\mu$ m) were tested using the methods described in section 2.2. According to the discussion in section 3.2, in this study, the sessile drop technique, the capillary rise and the drop

penetration test are not recommended to compare the wettability of coal particles with different sizes.

The sink test shows the most consistent results with the previous findings. Figure 42 illustrates that the longest sink time was observed by the finest coal particles of 0~38  $\mu\text{m}$  at each test concentration, and the largest ones of 75~90  $\mu\text{m}$  require the shortest time to sink and penetrate. The time difference between the longest time and the shortest time is huge, for example, the sink time and the penetration time of 0.60% SDBS for 0~38  $\mu\text{m}$  is more than 10 times of that for 75~90  $\mu\text{m}$  coal samples.

An interesting observation was found at the 0.10% SDBS, for the largest coal size 75 ~ 90  $\mu\text{m}$ , the complete wetting was considered as not occur because a thin oily film remained on the solution surface after the 1500 s. This may be because of the existing of some insoluble substance in coal samples and its size was in the 75 ~ 90  $\mu\text{m}$  range, but it disappeared after increasing the SDBS concentration. For other two smaller ranges, their sink time at 0.10% SDBS was consistent with the results obtained from 0.20% ~ 0.60% SDBS, which can be seen in Table 6.

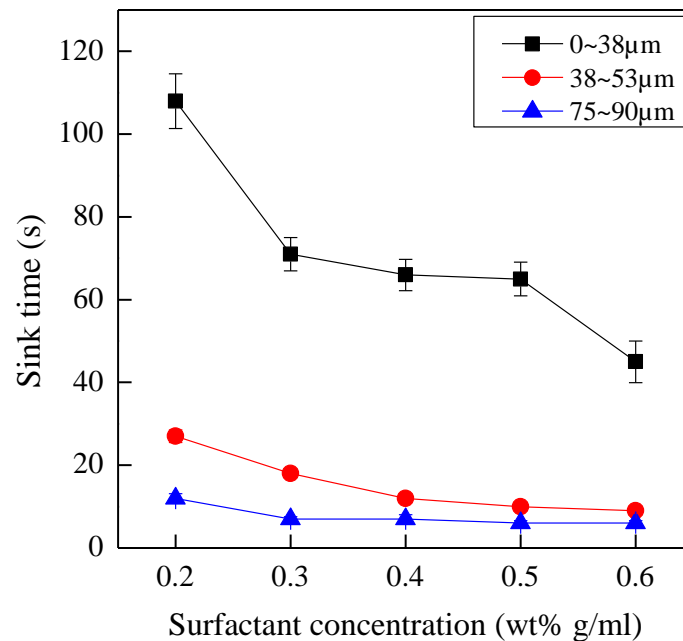


Figure 42. Sink time for different particle sizes with SDBS surfactant

#### 4.4 Conclusions

The coal wettability in presence of surfactant SDBS and SDS was characterized by four methods. Both the advantages and disadvantage of these four methods were illustrated. In terms of operation, the sink test is considered as the simplest method because no special treatment is needed. While the sessile drop measurement requires compressing the coal particles into



pellets, and both the capillary rise test and the drop penetration test require compacting coal particles into coal columns in glass tubes, which complicate the sampling process. Among these three methods, the longer test duration and the possibility of filter paper fall off make the capillary rise test as the most time consuming and complicated one.

From the point of the accuracy, due to no special sample preparation is needed, the sink test is believed to reflect the coal wettability more accurately. In the other three methods, the compression or compaction may modify the natural structure of the tested coal particles and lead to measurement inaccuracy. In addition, for the sessile drop measurement, the fast liquid penetration into the coal pellets make it is challenging to accurately capture the contact angle. The porosity of the coal column leads to inaccuracy for the capillary rise test and the drop penetration test.

Speaking of the applicability, the sessile drop measurement, the capillary rise test, and the drop penetration test are not recommended for evaluating the impact of the particle size on the coal wettability as they are not suitable to larger coal particles. Also, the drop penetration fails to indicate the influence of the surfactant type and concentration on the coal wettability as the inconsistent experimental results observed.

Experiment results shows that compared to the use of water, the coal wettability was improved by surfactant solution, which is indicated by the achieved smaller contact angles, less sink time, less drop penetration time, and steeper slopes for the capillary rise test. For the surfactant, the sessile drop measurement, the sink test, and the capillary rise test all indicated SDBS enhanced the coal wettability better than SDS, However, the drop penetration fails to distinguish the difference of these two surfactants. For the surfactant concentration, the sessile drop test shows the best coal wettability occurs at the highest tested concentration for both two surfactants, but the sink test and the capillary rise test suggests an inconsistent result that increasing the concentration does not help to improve the wettability. The results of the sink test confirmed that the finer coal particles have worse wettability than larger ones.

Overall, the sink test was recommended as the most reliable method in terms of its ability to compare the coal wettability in different surfactants with various concentrations. Also, it was suitable to investigate the effect of particle size on wettability. The disadvantages of other three methods were detailed, and these three methods are not suggested for comparing the coal wettability of different sizes. Although the sessile drop is the direct method to reflect the coal wettability, the obtained apparent contact angle can only be used for evaluating wettability

qualitatively. The drop penetration test is not suitable to evaluate different surfactant. The results of these four tests indicated SDBS with concentration of around 0.20% is preferable to improve the coal wettability, which can be used as a starting point when applying SDBS in coal dust suppression.

## **Acknowledgments**

The authors gratefully acknowledge Mrs Mujesira Vukancic and Mrs Anusha Kumara from the Department of Mining and Metallurgy at Western Australian School of Mines, Curtin University for providing laboratory technical supports.

## **5 Chapter 5**

### **Characterized of coal wettability in presence of surfactants and electrolytes additions**

The work in this chapter will be submitted to a peer reviewed Journal.

It was entirely written by Yinping Chen and reviewed by Dr. Guang Xu

This chapter uses the most reliable measurement, which is based on the result of Chapter 4, to study the impacts of surfactant type, surfactant concentration, coal dust particle properties and the electrolytes addition to surfactants on improving the coal wettability.

## **Abstract**

The sink test is a useful and simple method to evaluate the ability of surfactant to wet coal particles. Two measuring methods was compared, and it was shown the visual sink test produced more reliable results. The parameter sensitivity analysis indicated that the sink test result was closely affected by the coal sample mass used but not influenced by the baker size. Among the selected surfactants, the non-ionic surfactant TX100 improved the coal wettability best, then followed by anionic surfactant SDBS. The CTAB failed to improve the coal wettability at all the test concentrations. Poor wettability was observed on the coal particles with finer particle size and higher rank in all the surfactants. The addition of all the salts into the SDBS reach a synergistic effect in coal wettability enhancement, but no improvement was achieved for non-ionic surfactant TX100. The  $\text{KNO}_3$  additive was the best additive on improving the cationic surfactant CATB's performance.

Key words: coal dust, surfactant, sink test, wettability, synergistic effect

## **5.1 Introduction**

Coal Workers' Pneumoconiosis (CWP) has received much attention in the last decades due to its increasing prevalence, which resulted in severe health issues for thousands of coal miners (Ayoglu et al., 2014b; DE Pollock et al., 2010). For example, its prevalence increased from 2.1% in the 1990s to 3.2% in the 2000s, and during 2005 to 2015, still a great number of coal miners suffered from CWP (Joy et al., 2012; Laney & Attfield, 2010; Mo et al., 2014; Reynolds et al., 2017). In Australia, it was predicted that more than 1,000 coal miners could have CWP in Queensland (Zosky et al., 2016). CWP is one of the lung diseases that caused by long exposure to excessive concentrations of finer coal parties. As it is an irreversible and potentially fatal lung disease, it contributes to a higher mortality rate (Han et al., 2017). Thus, it is vital to maintain the coal dust concentration in underground mines below the safety limits. However, it has been found traditional suppression methods include using ventilation dilution and water spray failed to meet the recommended exposure limit of  $1 \text{ mg/m}^3$  that was adopted by the National Institute for Occupational Safety and Health (NIOSH) in September 1995 (NIOSH, 2003a).

Past research has shown that adding surfactant to the water spray system can substantially increase the dust suppression efficiency, the detailed description were illustrated by Xu (G. Xu, Chen, et al., 2018). Xu (G. Xu, Chen, et al., 2018) also described that the addition into surfactant solution is a method to improve the surfactant efficiency. However, it was found that not too much research has been conducted on this research topic. In terms of the way to evaluate the surfactants' effectiveness, Xu (G. Xu, Chen, et al., 2018) presented the description for all the methods as well as the advantages and disadvantages of each method. Chen (Chen et al., 2019) concluded that the sink test was the most simple and reliable static method by comparing other methods. Thus, in this study, the sink test was employed. However, there are two ways to perform the sink test, to determine which measurement is one of the objectives of this study. Overall, the study is organised as follows: the first purpose is to describe and examine the two measurements for the sink test and determine the more reliable measurement that will be used in this study. The second purpose is to investigate the effect such as temperature, apparatus diameter and coal sample mass on the coal wettability. The third objective is to evaluate the effectiveness of electrolytes addition to surfactants on improving the coal wettability.

## 5.2 Experiment

### 5.2.1 Materials

Three coal samples with different ranks were collected from mine sites. The proximate and ultimate analysis of these coal sample are shown in Table 8. All the procedures for preparing coal samples followed the standards of the American Society of the International Association for Testing and Materials (ASTM). To prepare samples for tests, the coal was crushed, pulverised, dried and sieved. Four sizes (0~38, 38~53, 75~90 and 0~106  $\mu\text{m}$ ) of coal particles were obtained.

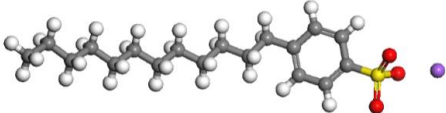
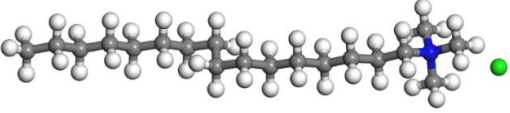
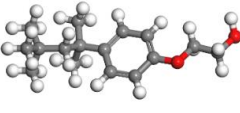
Table 8. Proximate and ultimate analysis of three coal samples

Coal sample No.	Coal rank	Proximate analysis (%)			Ultimate analysis (%)				
		$M_{ad}$	$V_{ad}$	$A_{ad}$	$C_{ad}$	$H_{ad}$	$O_{ad}$	$N_{ad}$	$S_{ad}$
Coal 1	Lignite	19.5	29.2	16.3	47.4	3.02	12.7	0.74	0.34
Coal 2	Subbituminous	19.5	35.0	4.3	56.9	3.44	14.5	0.91	0.44
Coal 3	Anthracite	2.1	6.6	7.5	82.5	2.77	3.0	1.06	1.07

(where  $M_{ad}$  means moisture,  $V_{ad}$  means volatile matter,  $A_{ad}$  means ash,  $FC_{ad}$  means fixed carbon,  $C_{ad}$  means calorific value,  $H_{ad}$  means hydrogen,  $O_{ad}$  means oxygen,  $N_{ad}$  means nitrogen,  $S_{ad}$  means sulfur)

Three surfactants, purchased from Sigma Aldrich Pty. Ltd, include sodium dodecylbenzenesulfonate (SDBS), Triton X-100 (TX100), and cetyl trimethylammonium bromide (CTAB) were employed in this study. They are anionic, non-ionic, and cationic surfactant, respectively, and their molecular structure presented in Table 9. Four salts named sodium chloride (NaCl), sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>), potassium chloride (KCl) and potassium nitrate (KNO<sub>3</sub>) were used as additions to enhance surfactants' effectiveness on coal wettability. The purities of all chemicals used in this study are over 99.0%.

Table 9. Molecular structure of SDBS, CTAB and TX100

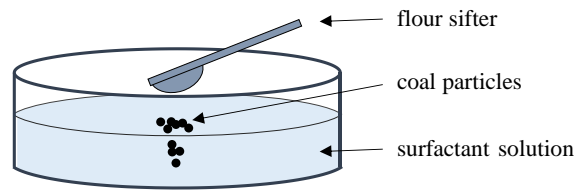
Surfactant	Linear formula	Molecular Weight (g/mol)	CMC	Molecular structure
SDBS	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>11</sub> C <sub>6</sub> H <sub>4</sub> SO <sub>3</sub> Na	348.48	1.5 mM <sup>a</sup> (0.052 wt% g/ml)	
CTAB	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>15</sub> N(Br)(CH <sub>3</sub> ) <sub>3</sub>	364.45	1 mM <sup>b</sup> (0.036 wt% g/ml)	
TX100	<i>t</i> -Oct-C <sub>6</sub> H <sub>4</sub> - (OCH <sub>2</sub> CH <sub>2</sub> ) <sub>x</sub> OH, x=9-10	625	0.22 mM <sup>c</sup> (0.014 wt% g/ml)	

(<sup>a</sup> Data collected by (Mohamed et al., 2015), <sup>b</sup> Data collected by (Roman Marsalek et al., 2011), <sup>c</sup> Data collected by (Dipankar Koley & Bard, 2010). Coloured balls represent C in grey, H in white, O in red, S in yellow, N in blue and Br in green.)

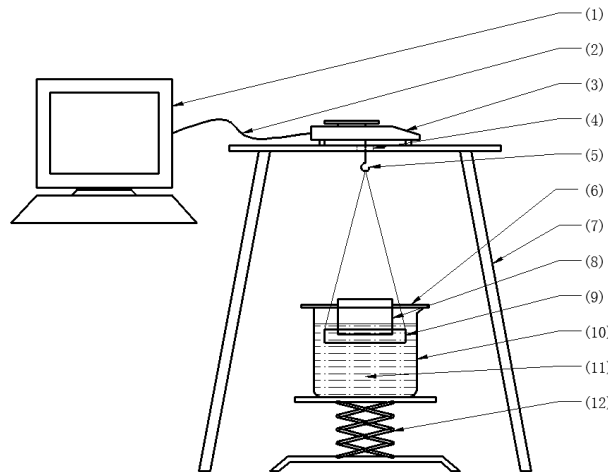
## 5.2.2 Experiment methods

The sink tests can be performed in two ways. One way is the visual sink test method (Figure 43(a)) that records the sink time when all coal particles disappear from the surfactant solution surface by visual observation. The other way is the weight sink test method (Figure 43(b)) that observes the weight change with time, and the sink time is determined when the weight gain do not change anymore. To determine which measurement can produce more reliable results, a simple comparative experimental design is applied. For each measurement, 20 replicate sink tests were conducted with the same experimental conditions. In this study, the sink test is stopped measuring when the sink time is over 1000 s. In this case, we assume coal wettability

will not be improved. It is generally accepted that the less sink time means the better coal wettability. In this study, each test was repeated three times to obtain more accurate data.



(a) The visual sink test



(1) Computer (2) Data cable (3) Electronic Balance (4) Drilled Hole (5) Hook (6) Barrier Support  
(7) Wood Stand (8) Barrier (9) Balance Pan (10) Glass Beaker (11) Solution (12) Lab Jack

(b) The weight sink test

Figure 43 .Schematic diagram of the two sink tests

## 5.3 Results and discussion

### 5.3.1 Two measurements comparison

Figure 44 shows the results of sink time obtained using the two methods mentioned in section 0. The stand deviation of visual sink test and weight sink test is 1.89 and 3.89, respectively. F hypothesis test was adopted for the comparing these two measurements. In this test, the hull hypothesis  $H_0$  is the visual sink test and weight sink test have the same variance, and it is against the alternative hypothesis  $H_1$  that the weight sink test has a larger variance than the visual sink test. The Analysis of Variance (ANOVA) is summarised in Table 10. A fixed significance level with  $\alpha=0.05$  is used. It is found the  $F_{0.05, 1, 38}=4.1 < F_0=40.29$  (Montgomery, 2013), and the p-value (0.000) is less than 5%, which means sufficient statistical evidences conclude that there is difference in the sink time variance of the two measurements. Thus, the

null hypothesis can be rejected. The variance of the weight sink test is larger than that of the visual sink test. Therefore, the visual sink test is employed to characterize the coal wettability.

Table 10. ANOVA for the sink time measured by the two methods

Source of Variance	SS	DF	MS	F <sub>0</sub>	P-Value
Sink time	378.2	1	378.225	40.29	0.000
Error	36.8	38	9.388		
Total	735	39			

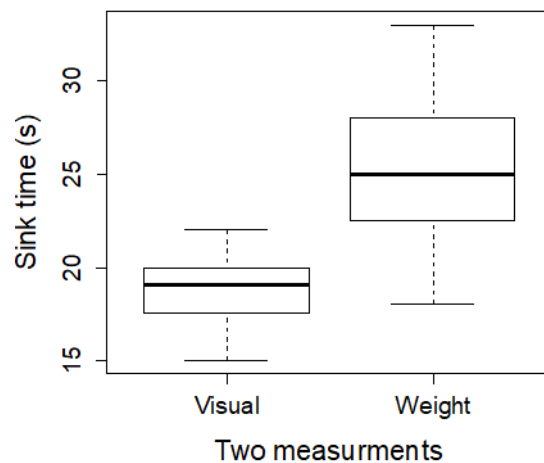


Figure 44. Boxplot of two measurements of sink time with using surfactant 0.40% SDBS on collie coals.

## 5.3.2 Sink test parameter sensitivity analysis

### 5.3.2.1 Temperature

The sink time of coal particles 0~106  $\mu\text{m}$  in three surfactant solutions were measured under three temperature of 20°C, 30°C and 40°C. The effect of temperature on the sink time varies in different type surfactants with different concentrations. For SDBS as shown in Figure 45 (a), at the concentration of 0.10%, the sink time was decreased greatly from 152 s to 129 s and 103 s when the temperature increased from 20°C to 30°C and 40°C, respectively. But at 0.20% SDBS, no obvious reduction was observed on the sink time when temperature increased. A slight increase on the sink time was even found as the temperature went up at 0.40% SDBS. For TX100 as shown in Figure 45 (b), the sink time went up slightly when the temperature increased from 20°C to 40°C at all the tested concentrations. This indicated that the coal wettability was weaken when the temperature increased for the non-ionic surfactant. For CTAB, the sink time remains over 1000 s when the temperature increased from 20°C to 40°C



at the 0.10% and 0.20% concentration. But for 0.40% CTAB, the sink time was not reduced to around 960 s until the temperature reached to 40°C.

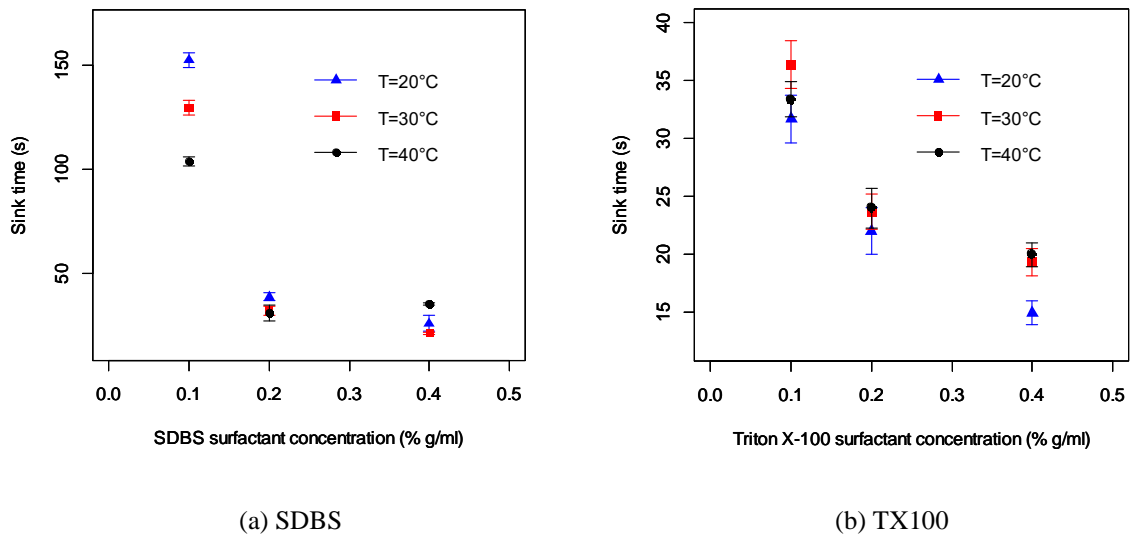


Figure 45. Sink time of coal particles (0~106  $\mu\text{m}$ ) with different temperature

### 5.3.2.2 Coal sample mass

Usually, 0.50 g coal particle sample is used in the sink test (G. Xu, Chen, et al., 2018), but different sample masses were utilized in different studies, for example, 0.1 g and 0.5 g coal particle samples were used by Glanville (Glanville & Wightman, 1979), 0.5 g was used by Chen (Chen et al., 2017), 0.7 g was used by Tien (Tien & Kim, 1997) and Chander (S. Chander et al., 1987) used 0.04 g. In this study, the coal particle samples in the mass range of 0.05~1.5 g were tested by 0.20% TX100. Figure 46 shows the sink time of different coal sample masses within the range of 0.05~1.5 g. The correlation coefficient of 0.86 indicated that the sink time has a close correlation with the sample mass. The correlation was much closer in the range of 0.05 to 0.5 g with a larger correlation coefficient of 0.99.

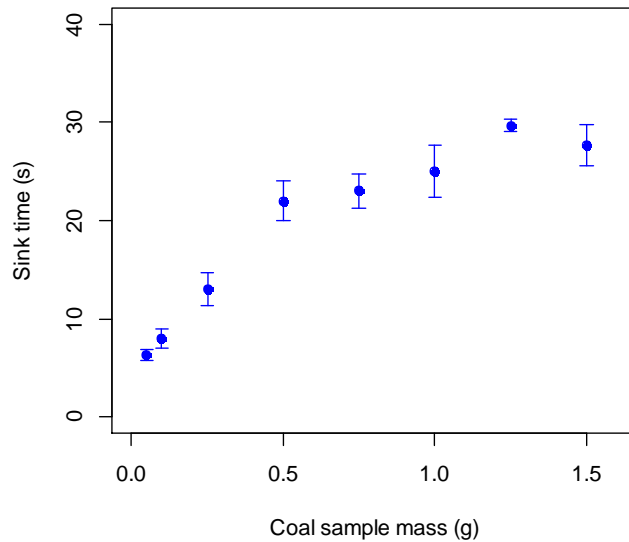


Figure 46. Sink time of coal particles with different coal sample mass

### 5.3.2.3 Apparatus size

A beaker is the common apparatus used for the sink test, different size beakers were used by different studies. For example, 250 ml and 1 l were used by Walker and Glanville, respectively (Glanville & Wightman, 1979; Walker, JR, et al., 1952). In this study, five different sizes (60, 90, 125, 150 and 180 mm) of beakers were used, and Figure 47 illustrates the sink time of 0.5 g subbituminous coal particles with 0~106  $\mu\text{m}$  in 0.40% SDBS solution. A correlation coefficient of 0.0153 was calculated between the sink time and beaker size, which presents that the sink time is not affected by using different size beakers. Also, the p-value of ANOVA is 0.493 that larger than 0.05 which means the data fails to provide sufficient evidence that baker size contributes to the difference in the mean sink time, the sampling variability may attribute to this.

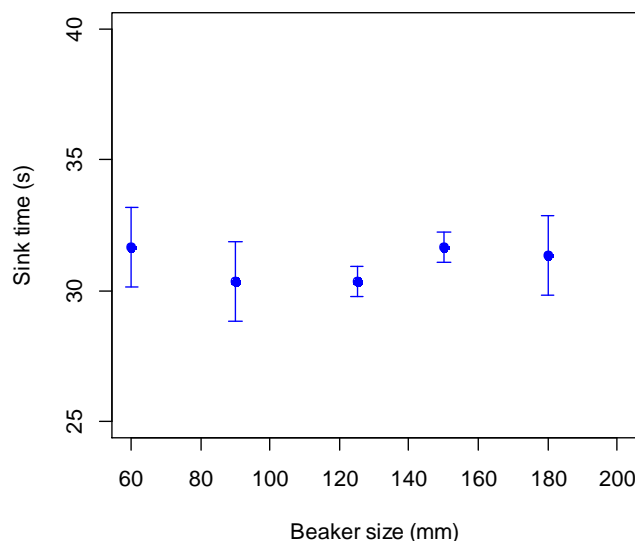


Figure 47. Sink time of coal particles with using different beaker sizes

Based on the results of the sink test parameter sensitivity analysis, 20°C, 0.5 g and 90 mm baker were applied for the sink test in the following study.

### 5.3.3 The impact of particle size and coal rank

Three size ranges (0~38, 38~53 and 75~90  $\mu\text{m}$ ), and three rank coal samples were test under three type surfactants. For CTAB with all the test concentrations, all the size range particles and all the rank coal particles remain on the CTAB solution surface over 1000 s, which means that CTAB with all the test concentrations failed to improve the coal wettability with the tested three range sizes. Thus, the following analysis focus on SDBS and TX100 solution.

Figure 48 indicated that for both SDBS and TX100 surfactant solution, the larger coal particles were more quickly to disappear from the solution surface for all tested concentrations. For example, for the 0.10% TX100 solution, the coal particles of 0~38  $\mu\text{m}$  took more than 100 s to sink while it only used less than 30 s for particles of 75~90  $\mu\text{m}$ . There maybe two reasons that finer particles have poor wettability. The first one is finer particles have larger specific area and stronger of air adsorption ability. It requires longer time to wet the coal surface for finer particles, and air films are more easily generated due to air adsorption which prevents contact between water and coal. The second reason is that the finer coal particles have rougher surface due to smaller average pore radius, more developed pore structure and larger specific pore volume. These properties facilitate air adsorption and thus prolong the sink time (Glanville & Haley, 1982; Q. Li et al., 2013; Yang et al., 2010; Q. Zhou et al., 2018b).

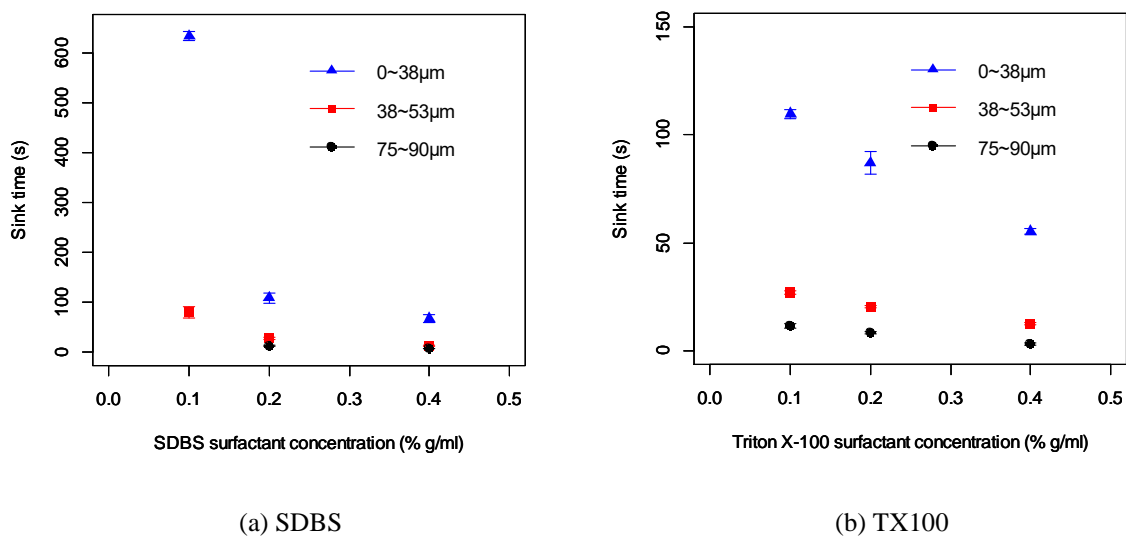


Figure 48. Sink time of coal particles with different sizes

Figure 49 clearly shows that higher rank coal particles have poor wettability in both SDBS and TX100 solution. For example, for the 0.10% SDBS solution, the sink time was 22 s, 158 s and 369 s for lignite, subbituminous and anthracite coal particles, respectively, and for the 0.20% TX100 solution, it took 13 s, 23 s, and 31 s for coal particles of lower rank to higher rank to disappear. It can be seen that the sink time of higher rank coal particles were dramatically reduced by the increasing surfactant concentration for both SDBS and TX100. For example, the highest rank anthracite particles' sink time was shorten three times (44 s to 15 s) when the TX100 concentration increased from 0.10% to 0.40%. However, the lowest rank lignite particles' sink time almost remained unchanged. Similar trend was also observed by using SDBS. Other laboratory studies also indicated surfactant solution is more effective on enhancing wettability of higher rank coals (R. J. Crawford & D. E. Mainwaring, 2001; Q. Li et al., 2013). Compare with low rank coal particles, higher rank coal particles tend to be more hydrophobic due to its less moisture, higher carbon content and less volatile matter content (Speight, 2015; J. Yu et al., 2013).

It can be observed from Figure 49 that compare with anionic surfactant SDBS, non-ionic surfactant TX100 can improve coal wettability to a larger extent for each rank coal sample that used in this study. For example, at the both 0.10% concentration, it took 22 s, 158 s and 369 s for lignite, subbituminous and anthracite coal particles, respectively, while it only took 15 s, 30 s and 40 s for these three rank samples to disappear on TX100 solution surface. This result is consistent with the conclusions in Xu's review (G. Xu, Chen, et al., 2018).

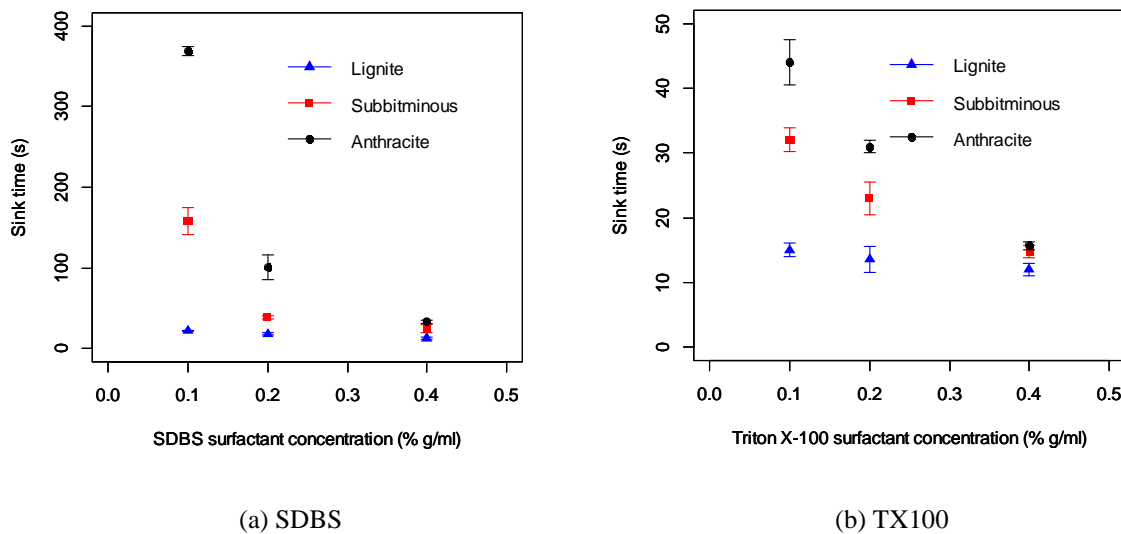


Figure 49. Sink time of coal particles with different ranks

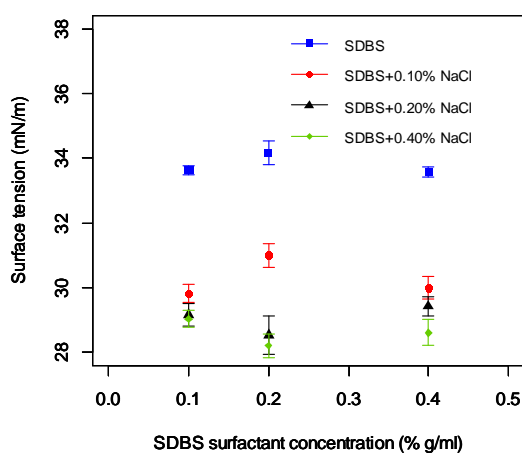
### 5.3.4 Synergistic effect analysis

#### 5.3.4.1 Effect of salts addition on surface tension

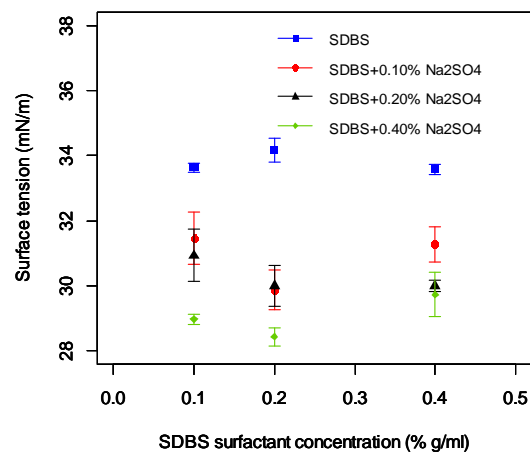
It is difficult for coal particles to penetrate water surface due to its high surface tension (72.8 mN/m at 20 °C) (Cheng et al., 2012a). In this study, 0.5 g coal particles with size of 0~106  $\mu\text{m}$  remains on water surface after 24 hours which confirms that coal particle has a rather poor wettability in water. It was believed that for coal particles to be wetted effectively, the critical surface tension of 45 mN/m is needed (Parekh & Aplan, 1978; Zisman, 1964).

Surfactants can decrease water surface tension greatly with a small amount. Figure 50 to Figure 52 show that the three surfactants can lower water's surface tension from 72.8 to around 32~35 mN/m with the concentration of 0.10%. But the surface tension does not decrease too much with further increase of surfactant concentration.

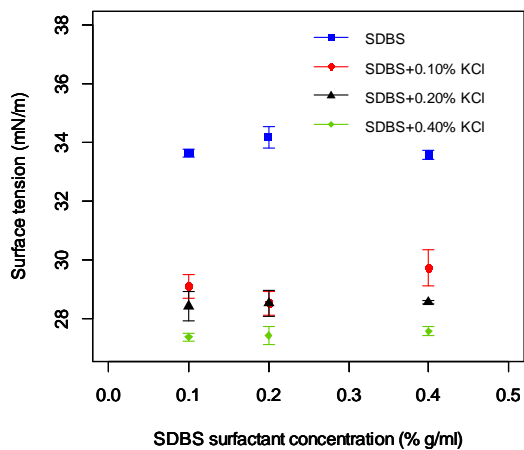
For SDBS, Figure 50 show that the addition of these four salts into SDBS can further reduce the solution's surface tension at all the test SDBS concentrations. It also can be observed that for all the salts, the surface tension was further reduced when the surfactant concentration increased from 0.01% to 0.40%. But for TX100 and CTAB as shown in Figure 51 and Figure 52, respectively, the addition of salts results in no apparent effects except for adding  $\text{KNO}_3$  to CTAB. The surface tension of 0.10% CTAB was reduced with the addition of  $\text{KNO}_3$ , and it was further decreased when the  $\text{KNO}_3$  concentration increased.



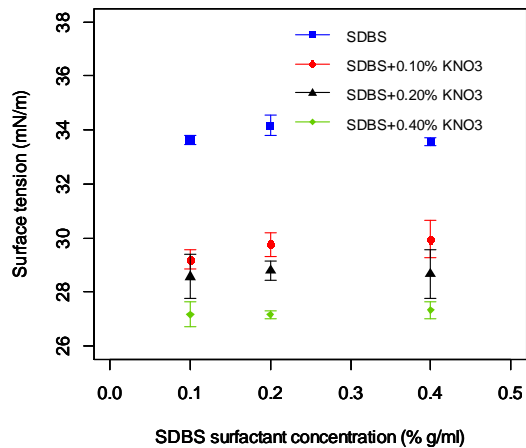
(a) NaCl



(b) Na<sub>2</sub>SO<sub>4</sub>

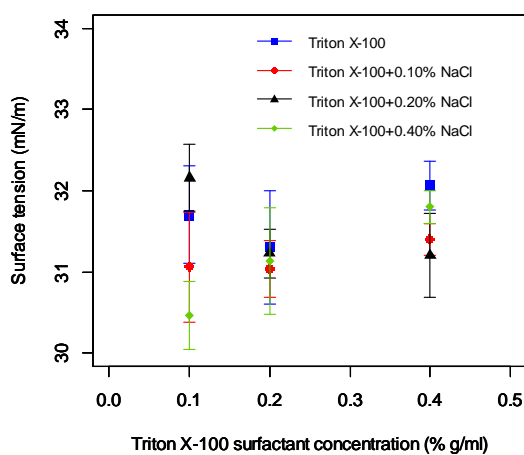


(c) KCl

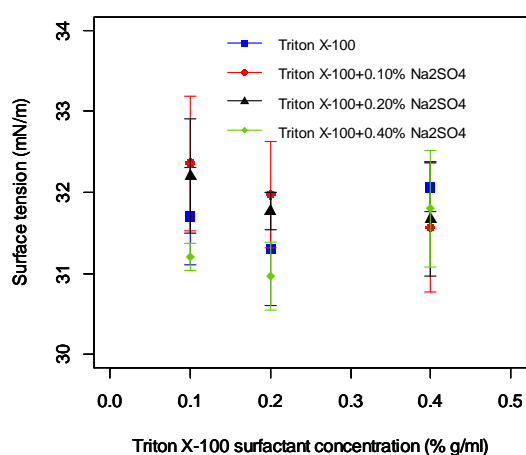


(d) KNO<sub>3</sub>

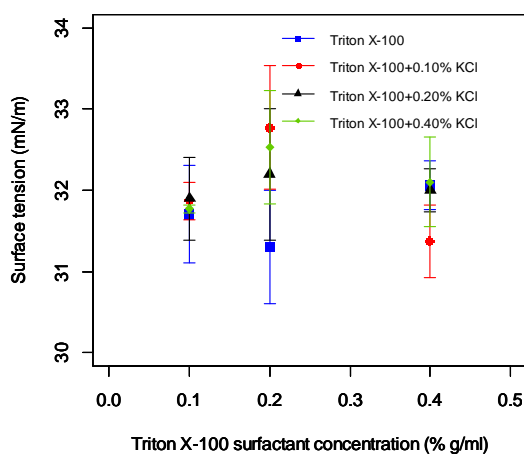
Figure 50. Surface tension of SDBS solution with salt additions



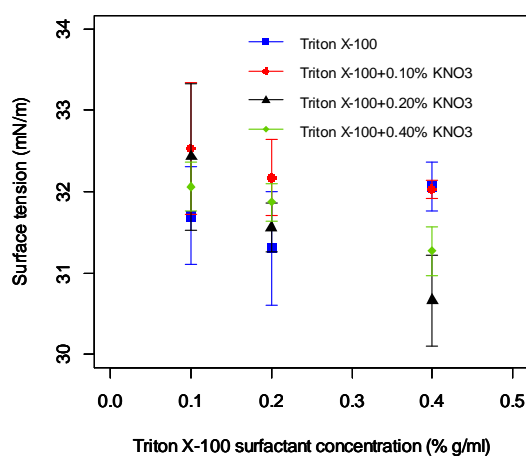
(a) NaCl



(b) Na<sub>2</sub>SO<sub>4</sub>



(c) KCl



(d) KNO<sub>3</sub>

Figure 51. Surface tension of TX100 solution with salt additions

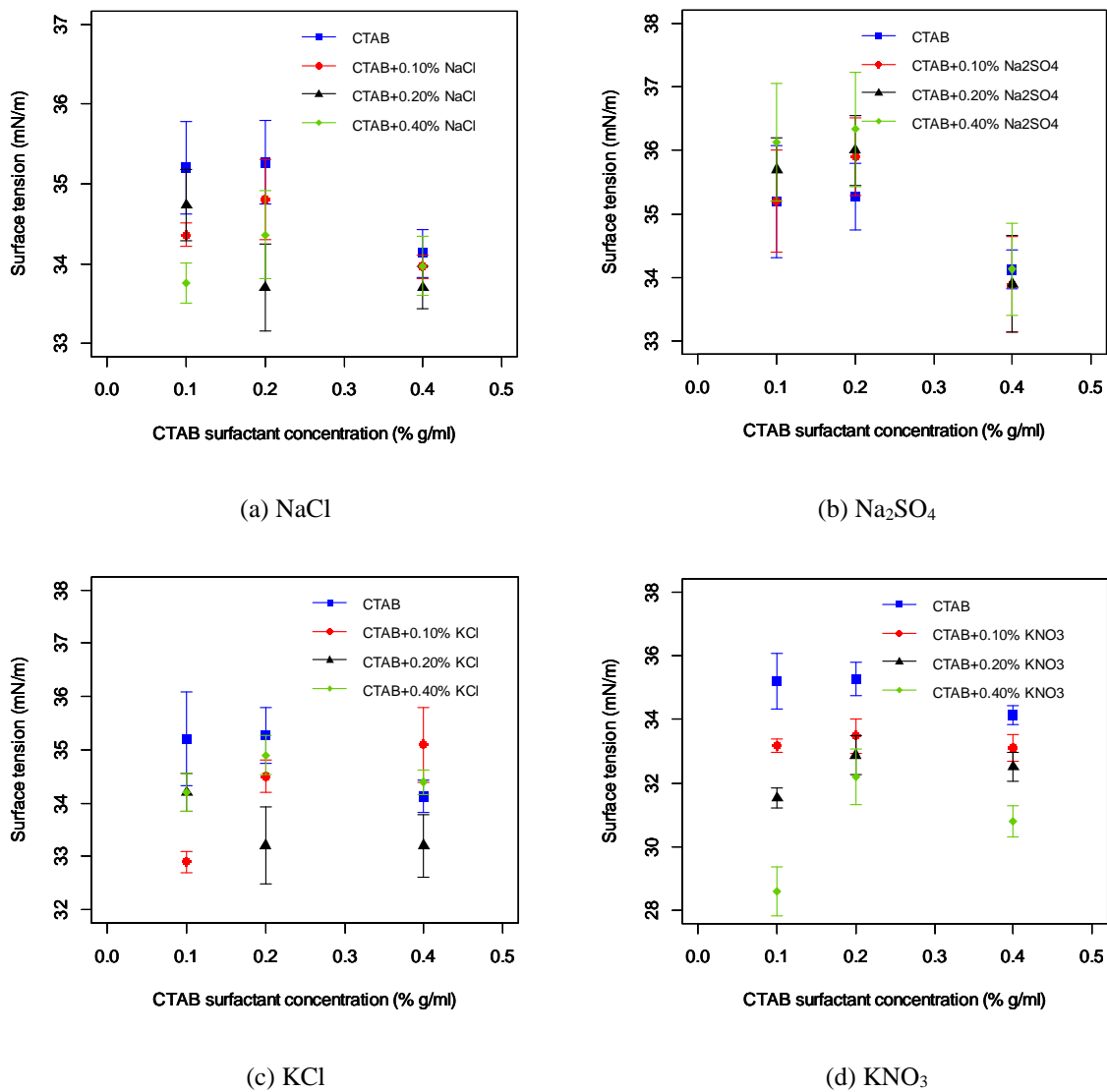


Figure 52. Surface tension of CTAB solution with salt additions

### 5.3.4.2 Coal wettability in surfactants with salt addition

#### 5.3.4.2.1 Coal wettability in surfactants only

Among these three type surfactants, the cationic surfactant CTAB failed to improve the coal wettability as most of coal particles remain on the surfactant solution surface after 1000 s even when the surfactant concentration is 0.40%. Although CTAB can reduce the surface tension to the same extent as the other two surfactants, the coal wettability is not enhanced. This finding is consistent with one previous study (Howard W Kilau, 1990).

Figure 53 show that The 0.10% SDBS and 0.10% TX100 both effectively reduced the sink time from more than 24 hours to around 170 s and 35 s, respectively. Both the minimum sink

time occurred when the concentration was 0.40%. The out performance of TX100 has been reported elsewhere (Tang et al., 2016) (R. J. Crawford & D. E. Mainwaring, 2001; Tessum & Raynor, 2017b; Tien & Kim, 1997). As it is a non-ionic surfactant, the lower critical micelle concentration (CMC) and higher aggregation number may contribute to its superior performance. CMC is the concentration that surfactant molecules form a unimolecular layer on the coal particle surface. When the surfactant concentration is higher than the CMC, a micelle is formed. The aggregation number is the number of surfactant molecules in a micelle (Porter, 1994). Lower CMC and higher aggregation number indicate micelles can be formed at a lower concentration and more surfactant molecules in a micelle, in which means a higher efficiency (G. Xu, Chen, et al., 2018).

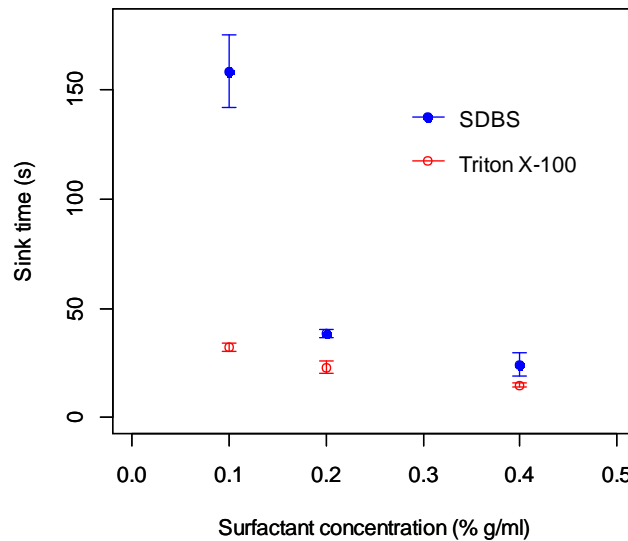


Figure 53. Sink time with three types of surfactants with different concentrations for the coal particle size 0~106  $\mu\text{m}$

### 5.3.4.2.2 The synergistic effect on coal wettability

The synergistic effect of salts addition varies in different surfactants.

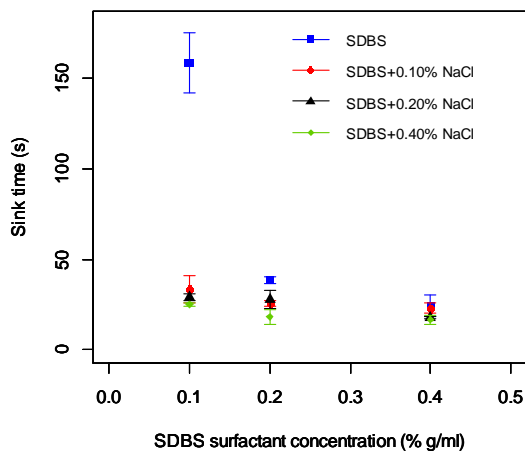
For SDBS, as shown in Figure 54, the sink time in 0.10% SDBS was substantially decreased with 0.10% of these four salts addition, but it remains unchanged when increasing the salts' concentrations. However, the addition of salts fails to improve the higher concentration (0.40%) SDBS's efficiency as no apparent reduce was found in the sink time. This change trend is similar with the SDBS surface tension change.

Figure 55 indicates that the addition of salts makes no improvement on coal wettability in the TX100 solution because no further improvements were observed for the sink time. The results were consistent with TX100's surface tension when adding these four salts.

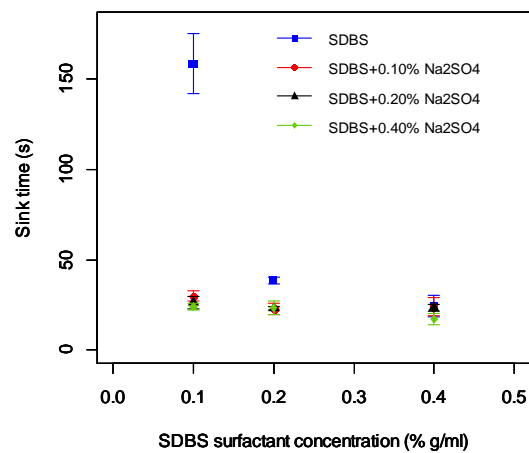


For CTAB, the obvious coal wettability improvements were observed for all the salts addition. The four salts function rank as  $\text{KNO}_3 > \text{NaCl} > \text{KCl} > \text{Na}_2\text{SO}_4$ . Adding  $\text{KNO}_3$  achieved the best synergistic effect. With 0.10%  $\text{KNO}_3$ , the sink time was reduced from over 1000 s to less than 200 s for all test concentrations of CTAB. Further increasing  $\text{KNO}_3$  only resulted in minimum sink time reduction. However, no synergistic effect was shown at all concentrations of other three salts added to 0.10% CTAB.  $\text{NaCl}$  and  $\text{KCl}$  started to work when CTAB's concentration reached 0.20% with their concentration was 0.10% and 0.20%, respectively, and the sink time kept decreasing with the increasing concentrations of these two salts. When adding  $\text{Na}_2\text{SO}_4$ , coal particles not disappeared from the solution surface until CTAB's concentration achieved 0.40%, and increased concentration of  $\text{Na}_2\text{SO}_4$  made the sink time longer instead of further reducing it. Overall, the  $\text{KNO}_3$  is the best additive candidate to improve coal wettability when using CTAB.

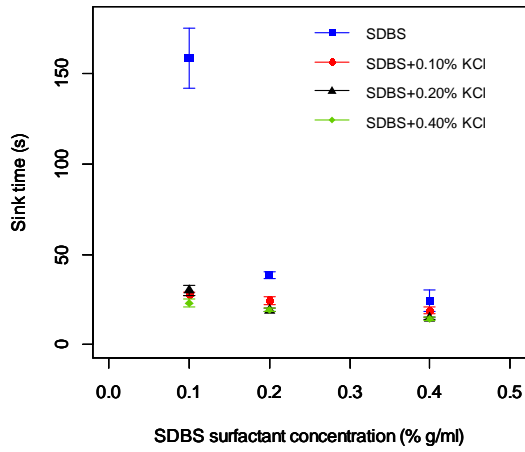
The reason for the above results may because the CMC of anionic and cationic surfactants can be significantly decreased by adding electrolyte, such as salts, while non-ionic surfactants are less affected. The charge in the surfactant solution can be decrease by the electrolyte additives, and the repulsion between ionic hydrophilic groups and force 1 (force 1 exists between the polar groups repelling one ionic to another due to the mutual charge repulsion) can be weakened due to this charge reduction (Paria, Manohar, & Khilar, 2005; Porter, 1994).



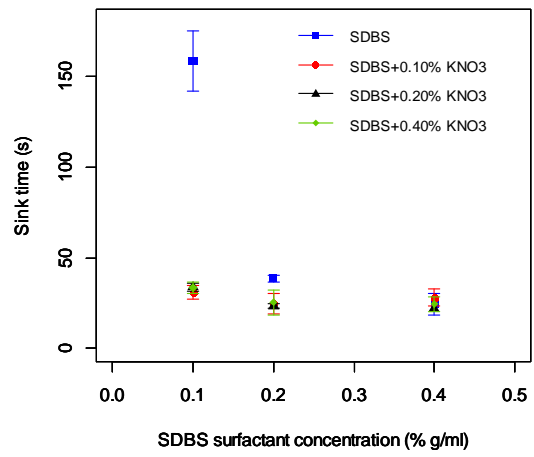
ml.(a) NaCl



(b)  $\text{Na}_2\text{SO}_4$

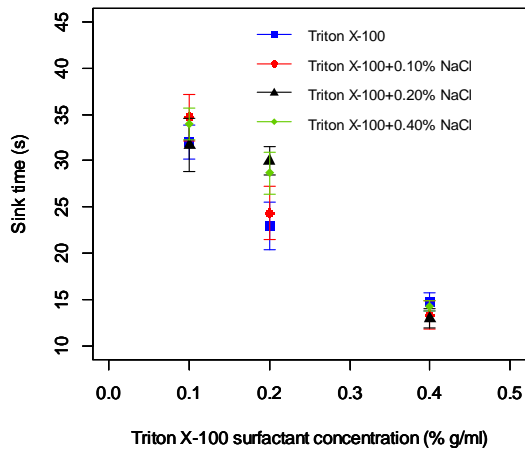


(c) KCl

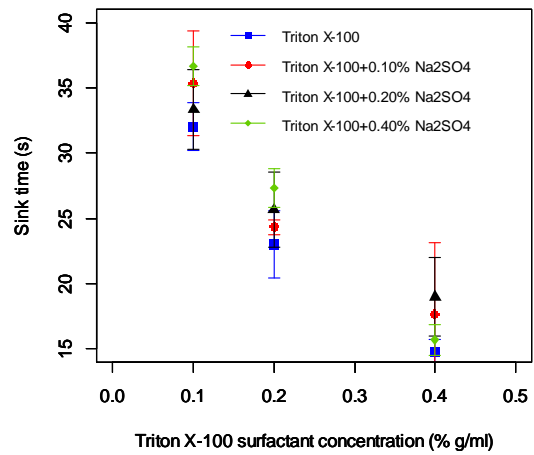


(d) KNO<sub>3</sub>

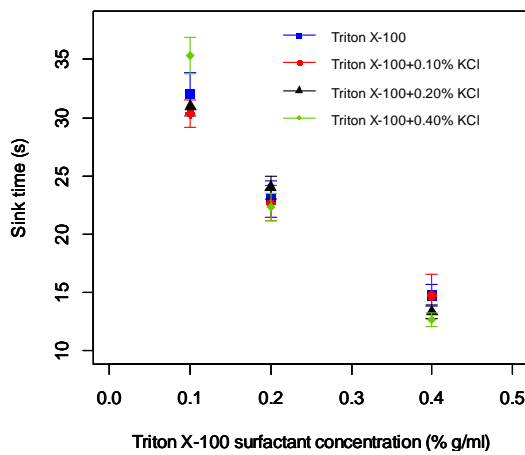
Figure 54. Sink time of coal particles in presence of SDBS solution with salt additions



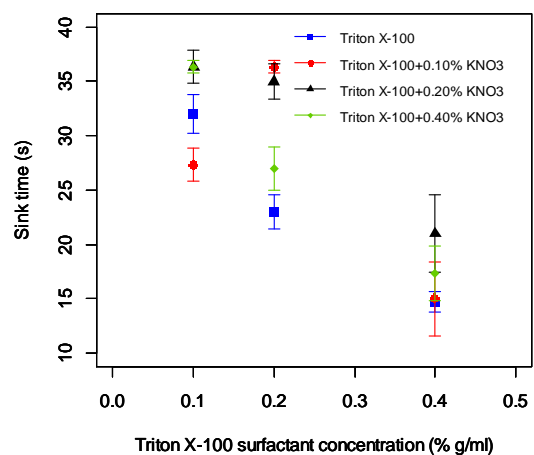
(a) NaCl



(b) Na<sub>2</sub>SO<sub>4</sub>



(c) KCl



(d) KNO<sub>3</sub>

Figure 55. Sink time of coal particles in presence of TX100 solution with salt additions

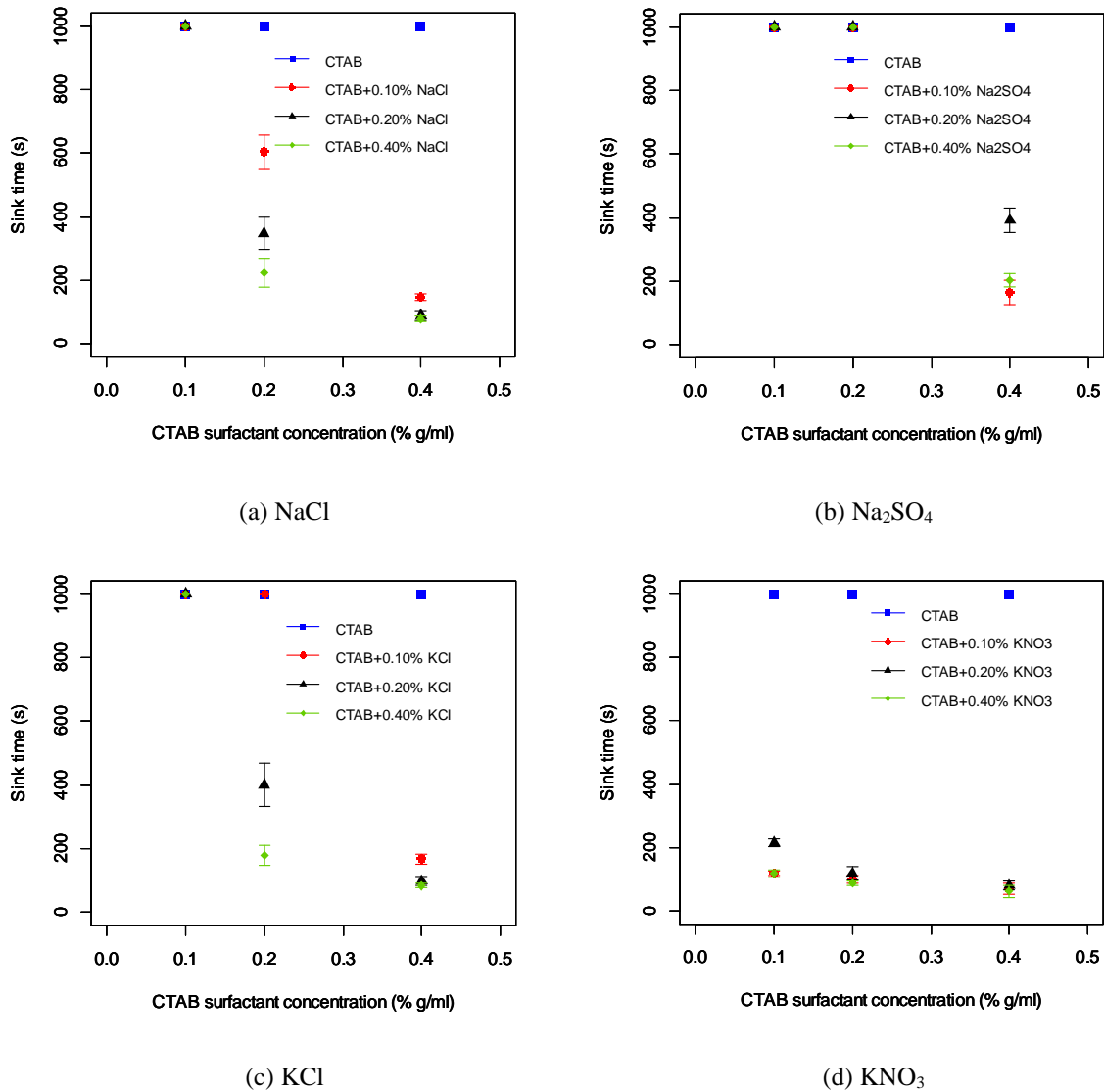


Figure 56. Sink time of coal particles in presence of CTAB solution with salt additions

## 5.4 Conclusions

The comparison of two sink test measurements was conducted and the visual sink test was considered as a simpler performable and more reliable method although the weight sink test can produce more detailed wetting performance.

The results of the parameter sensitivity analysis of the sink test indicated that the coal wettability was affected by the coal particles sample mass with the correlation coefficient of 0.86 in the test range of 0.05~1.5 g. But the coal wettability is not affected by the beaker size. The effect of temperature on the coal wettability was varies when different type surfactant applied. A stronger dependence of lower concentration SDBS with temperature was observed,

increased temperature greatly enhanced the ability of 0.10% SDBS to wet coal particles but not for 0.40% SDBS. Increase temperature also enhance the coal wettability in 0.40% CTAB. However, increased temperature slightly weak the TX100 effectiveness.

The sink tests show that the finer coal particles have poor wettability in both SDBS and TX100. Compare with lower rank coal particles, higher rank ones have relatively poor wettability both in the low concentration of SDBS and TX100 surfactant solution, but their wettability was sharply improved when surfactants' concentration increased while lower rank coal particles' wettability was not sensitive to the concentration change. Also, TX100 worked better on enhancement of the higher rank coal particles' wettability.

Three types of surfactants all decreased the surface tension of water distinctly with the concentration of 0.10% but increasing concentration do not help to further greatly lower the surface tension. The non-ionic surfactant TX100 showed a superior efficiency to improve coal wettability with the greatest sink time reduce and followed is the efficiency of the anionic surfactant SDBS. But the cationic surfactant CTAB failed to enhance coal wettability resulted in it's the worst surfactant candidate.

A synergistic effect was achieved for SDBS with all the four salts both on the surface tension and the sink time. KNO<sub>3</sub> is the best additive candidate for CTAB to lower the sink time, and other three salts only improved the CTAB's efficiency when they were presented in higher concentrations of CTAB. While less enhancement on coal wettability was noticed for non-ionic surfactant TX100.

## **6 Chapter 6**

### **Research on three type surfactants adsorption performance on different rank coal particles**

The work in this chapter will be submitted to a peer reviewed Journal.

It was entirely written by Yinping Chen and reviewed by Dr. Guang Xu

This chapter demonstrates the surfactant adsorption on the coal dust particles by using zeta potential test and the adsorption measurement and investigates the factors includes surfactants properties and coal dust properties that affect surfactant adsorption on coal dust particles.

## **Abstract**

The zeta potential and adsorption of three type surfactants on coal particles that with different ranks and particle sizes were studied. It was confirmed that the coal surface was negatively charged and the zeta potential of coal particles was changed based on the addition of surfactant type. In SDBS and CTAB, the lower rank coal particles showed lower zeta potential and the finer coal particles possessed the larger zeta potential, while opposite results were found in TX100. The lignite adsorbed more SDBS and less TX100 than subbituminous and anthracite but anthracite adsorbed the most amount CTAB at higher concentration. The finer coal particles adsorbed the more surfactant amount for all the surfactants at each concentration. Increasing temperature help to improve all the surfactant adsorption capacity but failed to lift all the coal particles' zeta potential at the tested surfactant concentrations. Adding salts increased the SDBS adsorption but no obvious improvement was found for other two surfactants.

## **6.1 Introduction**

Coal dust is well known for its hydrophobic property. Due to its hydrophobicity, it is challenging in underground mines to suppress coal dust concentration to regulatory limits, thus, thousands of miners are under threat of the lung diseases caused by the high coal dust concentration. Surfactants aid water spray are believed an effective method to lower coal dust concentration ((G. Xu, Chen, et al., 2018)).

It was believed that surfactant help to modify the surface characteristic of coal particles, and a number of studies have indicated that zeta potential measurement is one of the methods to determine the impact of surfactant on coal particle surface characteristic change (Chen et al., 2017; Gallardo-Moreno et al., 2004; R Marsalek, 2008). The electrical double layer was developed by Stern that consists of two layers. The first layer that fixed to a very short distance from the solid is called the Stern layer which presented the surface potential of the solid particle itself (coal particles in this study). The second layer that penetrates to a certain extent in the liquid (surfactant solution in this study). A theoretical boundary where ions and particles form a stable entity exists within this diffused layer, and the potential at this boundary is defined as zeta potential (Hunter, 2013; Pollastri et al., 2014; Smith, Crist, Clogston, & McNeil, 2017). It suggested the higher absolute value the greater stability to the colloidal system. This because

coagulation and flocculation are favoured in the low absolute value zeta potential environment, and the electrostatic repulsion that prevent the aggregation of dispersed particles are easier originated in a higher absolute value zeta potential colloidal system. The zeta potential ranges in -30 mV to +30 mV, in general, is considered as unstable than that over -30 mV or +30 mV (Pollastri et al., 2014). In the surfactant aided water spray system, the zeta potential value of coal particles – surfactant might be used as a method to measure the stability of the surfactant-coal particle colloidal system.

This study firstly aims to investigate the effects of surfactant on modifying coal particle surface characteristic by measuring the zeta potential of coal particles in presence of surfactants. Secondly, to determine the adsorbed surfactant amount on coal particle surface under different conditions by applying UV/Vis spectroscopy.

## 6.2 Experimental

### 6.2.1 Experimental materials

Three coal samples with different ranks used in this study. The lignite and anthracite were obtained from coal mines in Shanxi, China, and the subbituminous was provided from the Collie coal mine in Western Australia. The raw coal briquettes were crushed, pulverised, oven dried, sieved to the designed size ranges. All the sample preparation procedures followed the America standard practice for preparing coal samples for analysis. The proximate and ultimate analysis of these coal samples were presented in Table 11.

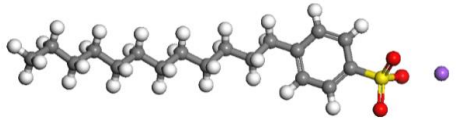
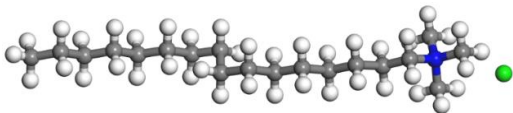
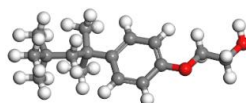
Three type surfactants, Sodium dodecylbenzene sulfonate (SDBS), Cetyl trimethylammonium bromide (CTAB) and Triton X-100 (TX100) were supplied by Sigma Aldrich Pty. Ltd. They are anionic, cationic and non-ionic surfactant, respectively. Their chemical structures are shown in Table 12. The surfactant solution was dissolved in deionized water and stirred by magnetic stirrers.

Table 11. The properties of three coal samples

Coal properties		Content (%)		
		Lignite	Subbituminous	Anthracite
Proximate analysis (%)	Moisture	19.5	19.5	2.1
	Volatile matter	29.2	35.0	6.6

	Ash	16.3	4.3	7.5
	Fixed carbon	47.4	56.9	82.5
	Hydrogen	3.02	3.44	2.77
Ultimate analysis (%)	Oxygen	12.7	14.5	3.0
	Nitrogen	0.74	0.91	1.06
	Sulfur	0.34	0.44	1.07

Table 12. Molecular structure of SDBS, CTAB and TX100

Surfactant	Linear formula	Molecular Weight (g/mol)	CMC	Molecular structure
SDBS	$\text{CH}_3(\text{CH}_2)_{11}\text{C}_6\text{H}_4\text{SO}_3\text{Na}$	348.48	1.5 mM <sup>a</sup> (0.052 wt% g/ml)	
CTAB	$\text{CH}_3(\text{CH}_2)_{15}\text{N}(\text{Br})(\text{CH}_3)_3$	364.45	1 mM <sup>b</sup> (0.036 wt% g/ml)	
TX100	$t\text{-Oct-C}_6\text{H}_4\text{-}(\text{OCH}_2\text{CH}_2)_x\text{OH}$ , $x=9\text{-}10$	625	0.22 mM <sup>c</sup> (0.014 wt% g/ml)	

(<sup>a</sup> Data collected by (Mohamed et al., 2015), <sup>b</sup> Data collected by (Roman Marsalek et al., 2011), <sup>c</sup> Data collected by (Dipankar Koley & Bard, 2010). Coloured balls represent C in grey, H in white, O in red, S in yellow, N in blue and Br in green.)

## 6.2.2 Experimental methods

### 6.2.2.1 Zeta potential test

The measurements of zeta potential for the surfactant solution on the coal particles were performed by employing a Zetasizer 3000. The aqueous solution of surfactants mixed with coal particles with a proportion of 0.001g/ml, and it was stirred at 100 rpm for 30 mins at 25 °C. 1 ml of the solution was used for the zeta potential analysis. The pH was measured before each zeta potential measurement. In this study, each zeta potential test was repeated three times to obtain more accurate data.



### 6.2.2.2 Adsorption measurement

In the adsorption study, 0.025 g coal particles were added to 25 ml surfactant solution, and the solution was stirred at 100 rpm for 30 mins. The supernatant, after the adsorption, was separated by using filter papers. The concentration of the filtered surfactant solution was determined by employing a UV/Vis spectroscopy (Lambda, 35 series, PerkinElmer) operating between the ranges of 200 to 700 nm. The SDBS, CTAB and TX100 concentration was determined by measuring UV absorbance at the wavelength of 260 nm, 200 nm and 275 nm, respectively. All the measurements (each measurement was repeated three times) were conducted at room temperature of 25 °C.

The amount of the adsorbed surfactant was determined by the surfactant solution concentration changed before and after adsorption, according to the following equation:

$$a = \frac{(C_o - C_e)V}{m}$$

Where  $a$  is amount of the adsorbed surfactant (mmol/g),  $C_o$  and  $C_e$  are the initial concentration and the adsorption equilibrium concentration (mmol/l),  $V$  is the surfactant solution volume (l) and  $m$  is the mass of the coal particles (g).

## 6.3 Results and discussion

### 6.3.1 The characteristics of coal particles and surfactants

#### 6.3.1.1 Zeta potential of coals

The zeta potential of coal particles in suspension is determined by the oxygenated functional groups, including the hydroxyl groups (-OH) and carboxyl groups (-COOH) on the coal particle surface (Debadutta Das, Sagarika Panigrahi, Pramila K. Misra, & Nayak, 2008). Previous studies have revealed that the zeta potential of coal particles in a neutral pH condition is negative, indicating that on the coal particle surface, more negative charges than positive charges are presented (R Marsalek, 2008; Y. Yu, Liu, Wang, Zhou, & Cen, 2012). This conclusion was confirmed in this study, as shown in Figure 57, the original zeta potential of coal particle in water remains at around -20 mV. The addition of surfactants influenced the zeta potential. The presence of anionic SDBS enhanced the negative charge from about -20 mV to around -60 mV. A charge reversal of from around -20 mV to approximately +50 mV was observed in CTAB solutions which indicates that the coal surface was completely covered by the positive ions that provided by CTAB. While the addition of non-ionic surfactant TX100

not changed the original zeta potential so much as it was still kept around -20 mV. All these zeta potential was measured at original pH of surfactant solution.

Figure 57 also shows the change of coal particles' zeta potential within the stirring time. The P value is 0.000 that is less than 0.05 which means the zeta potential means is affected by the stirring time. But it can be observed that the coal particles' zeta potential was not changed greatly when prolong the stirring time after half hour. This indicates that the time for coal surfactant suspension to reach the equilibrium stability may be around half hour in this study.

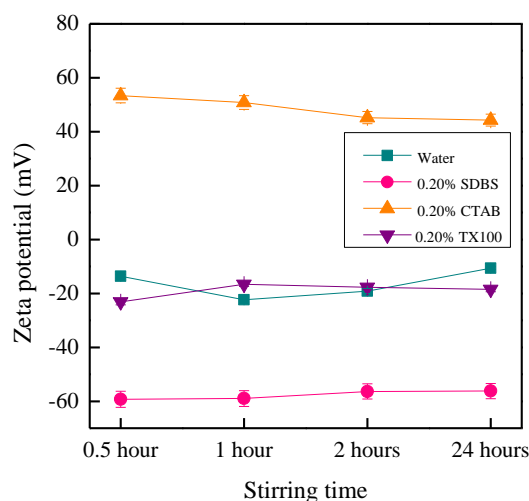


Figure 57. Zeta potential of coal particles in water and surfactant solutions at different stirring time

### 6.3.1.2 Surfactants Adsorption

The UV-VIS adsorption spectra of three surfactants in the deionised water are shown in Figure 58. The Figure 58 (a) reveals for SDBS a strong adsorption band was found at  $\lambda=260$  nm as the absorbance at this wavelength are easy to be distinguished within the tested concentration range, this observation was consistent with the results obtained by Geng (J. Geng, Johnson, Wheatley, & Luo, 2014), although the band of around  $\lambda=223$  nm is popularly applied by other researchers (Mina, Xuepin, & Bi, 2006; Paria et al., 2005; R. & Taffarel, 2010). The adsorption peak bands selected to analysis for CTAB and TX100 are  $\lambda=200$  nm (Figure 58(b)) and  $\lambda=275$  nm (Figure 58(c)), respectively. Figure 58 also indicates that for all surfactants, the absorbance increased with the increasing concentration.

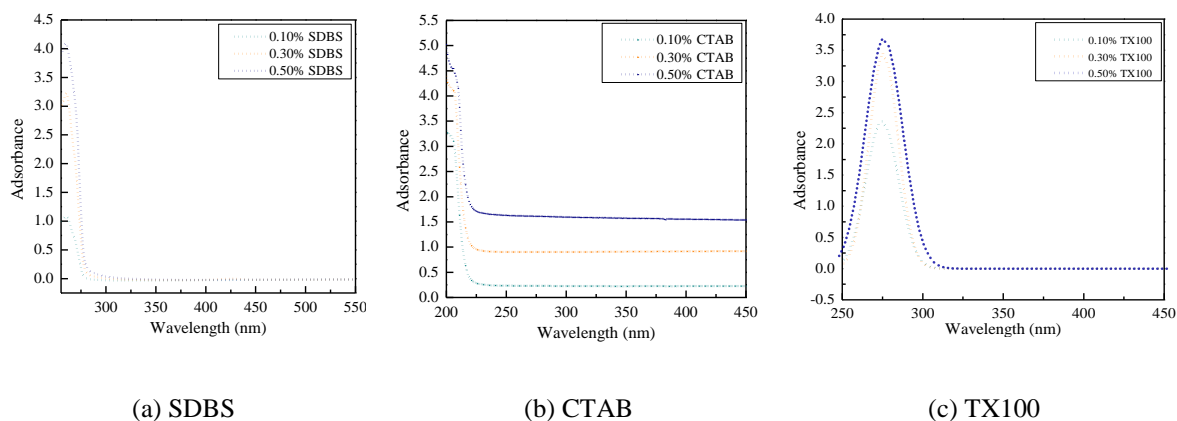


Figure 58. UV-VIS adsorption spectra of three surfactants in deionised water: (a) SDBS, (b) CTAB, (c) TX100.

## 6.3.2 Effects of coal properties on the surfactant adsorption on coals

### 6.3.2.1 Coal rank

#### 6.3.2.1.1 The effect of coal rank on the zeta potential

The zeta potential of lignite, subbituminous and anthracite were determined in the SDBS, CTAB, and TX100 within the concentration of 0.10% to 0.60%. Figure 59 shows that in SDBS and CTAB surfactant solutions, the zeta potential values for all these three rank coals raise as the surfactant concentration increasing, and the order of zeta potential values both were: anthracite > subbituminous > lignite. But an opposite changing trend and order were observed when they were in presence of TX100 surfactant solutions.

The difference on three rank coals zeta potential in SDBS and TX100 solution was larger than that in CTAB solution. The zeta potential difference between the lignite and anthracite was around 25 mV for SDBS and TX100, while for CTAB, it was around 10 mV. For example, at the surfactant concentration of 0.60%, the difference on zeta potential between the lignite and anthracite were around 22 mV, 10 mV and 25 mV in SDBS, CTAB and TX100, respectively.

Table 13 illustrates that the pH of three rank coal particles as a function of surfactant concentration in three surfactants. The subbituminous and anthracite are slightly moderately acidic in the all surfactants within the concentration of 0.10% to 0.60%, while the lignite are moderately alkali in SDBS and TX100 and are almost neutral in cationic surfactants in the test concentration range. Also, it can be noted that increase the surfactant concentration lowered the pH of the solution for all the coals with different ranks.

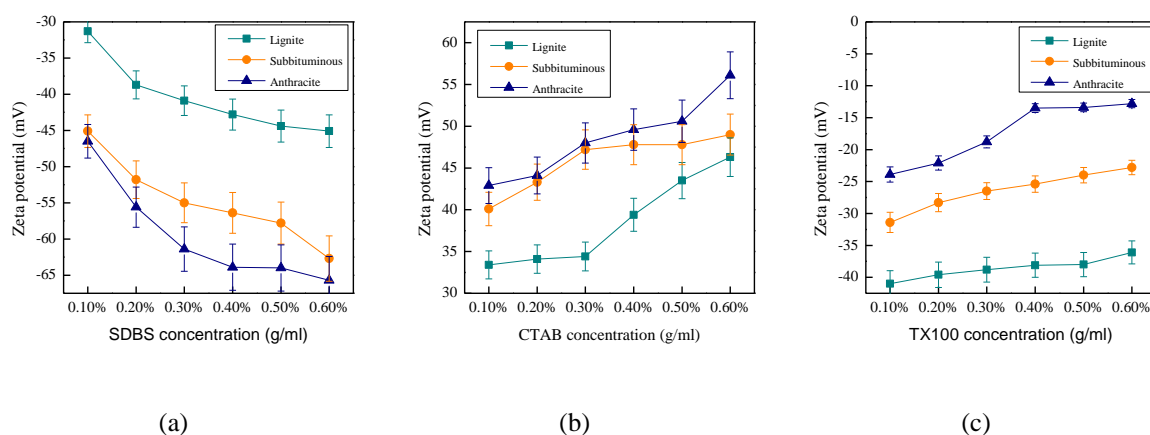


Figure 59. Variation of the zeta potential of three rank coals with different surfactants. (a) SDBS, (b) CTAB, (c) TX100.

Table 13. pH of coal particles surfactant solution at different concentrations

Surfactant concentration g/ml	pH								
	Lignite			Subbituminous			Anthracite		
	anionic	cationic	non-ionic	anionic	cationic	non-ionic	anionic	cationic	non-ionic
0.10%	7.28	5.68	7.64	4.50	3.97	5.79	5.83	5.53	5.87
0.20%	7.11	5.34	7.52	4.70	4.44	5.60	5.05	5.86	5.61
0.30%	7.08	5.07	7.40	4.79	3.97	5.77	4.82	5.23	5.25
0.40%	7.05	5.08	7.38	4.66	4.10	4.92	5.04	5.28	5.48
0.50%	7.04	5.01	7.39	4.51	4.13	4.78	5.14	5.08	5.05
0.60%	7.02	5.00	7.31	4.43	4.11	4.98	4.94	4.87	5.03

### 6.3.2.1.2 The effect of coal rank on the adsorption amount

The adsorbed surfactant amounts on different rank coals were calculated and the results show in Figure 60. For all three rank coal samples, the adsorbed amount for each surfactant went up when the surfactant concentration increased from 0.10% to 0.50%. But the increase varies for different surfactant types. For SDBS, the lignite adsorbed the most surfactant at all the test concentrations, especially at the concentration of 0.50%, the amount it adsorbed is more than twice of that adsorbed by subbituminous and anthracite. The change trends of subbituminous and anthracite are similar, the adsorbed surfactant amount greatly increased when the surfactant concentration went up from 0.10% to 0.30%. But only a slight improvement was observed

when the concentration continued increasing. For CTAB, the trends of adsorbed surfactant amount are similar, subbituminous adsorbed more at the concentrations of 0.10% and 0.20%, but the amount that anthracite adsorbed exceed lignite and subbituminous. For TX100, anthracite adsorbed the most amount at each concentration while no obvious increase was found for lignite.

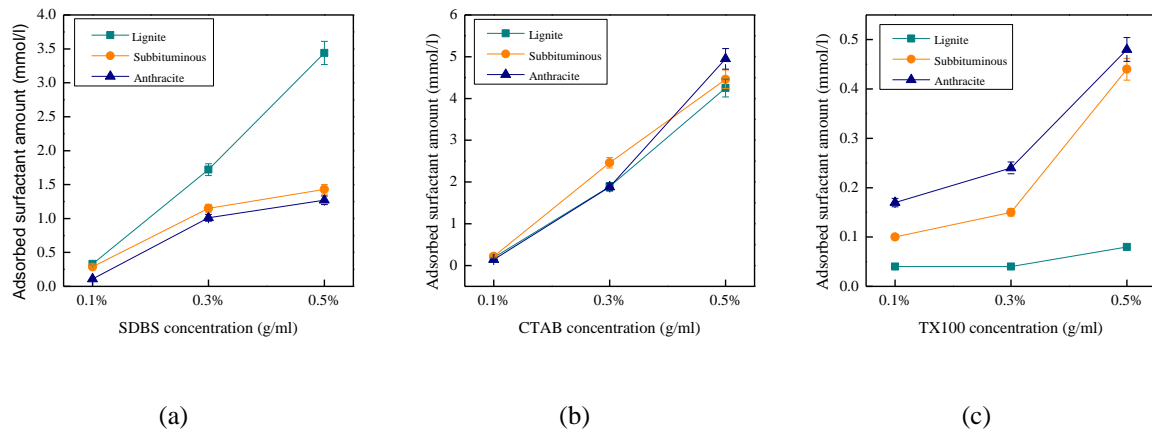


Figure 60. Variation of the adsorbed surfactant amounts on three rank coals. (a) SDBS, (b) CTAB, (c) TX100.

### 6.3.2.2 Coal particle size

#### 6.3.2.2.1 The effect of coal particle size on the zeta potential

Figure 61 indicates the zeta potential of different size coal particles. In surfactants of SDBS and CTAB at all the test concentrations, the finer coal particles, the larger zeta potential, vice versa. However in the TX100, the 75~90  $\mu\text{m}$  shows the largest zeta potential at the test concentration range and the smallest zeta potential was seen on 0~38  $\mu\text{m}$ .

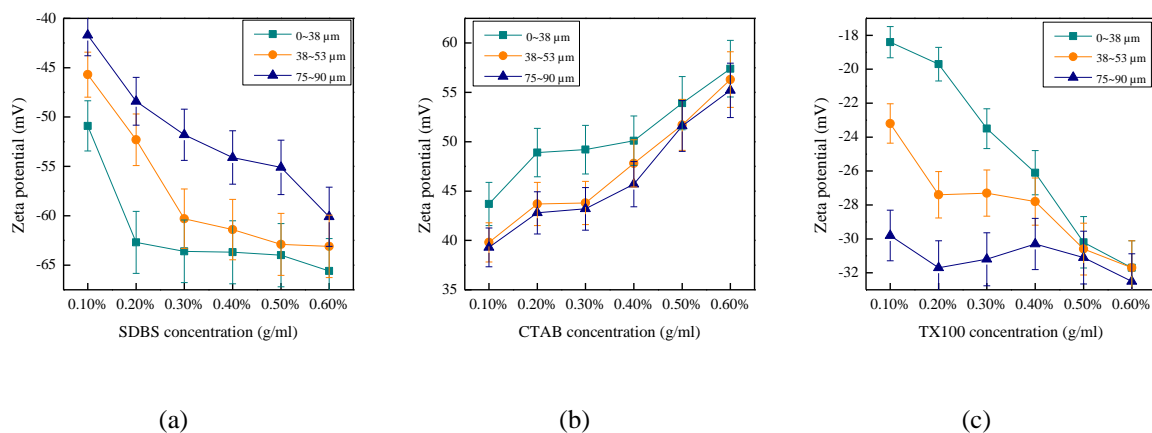


Figure 61. Variation of the zeta potential of three size range coals with different surfactants. (a) SDBS, (b) CTAB, (c) TX100.

### 6.3.2.2 The effect of coal particle size on adsorption amount

Figure 62 shows that overall, the adsorbed surfactant amount increased with the increasing concentration. The finest coal particles adsorbed the most amount for each surfactant at each concentration compare to the other two larger coal particles, especially at the highest concentration of 0.50%. The reason for this result maybe the finer coal particles have the larger surface area, thus, more surfactant amount can be adsorbed on the coal particles surface. Among these three surfactants, less amount of TX100 was adsorbed for each range coal particles, especially for larger sizes of 38~53  $\mu\text{m}$  and 75~90  $\mu\text{m}$ , increased TX100 concentration failed to improve the adsorbed amount.

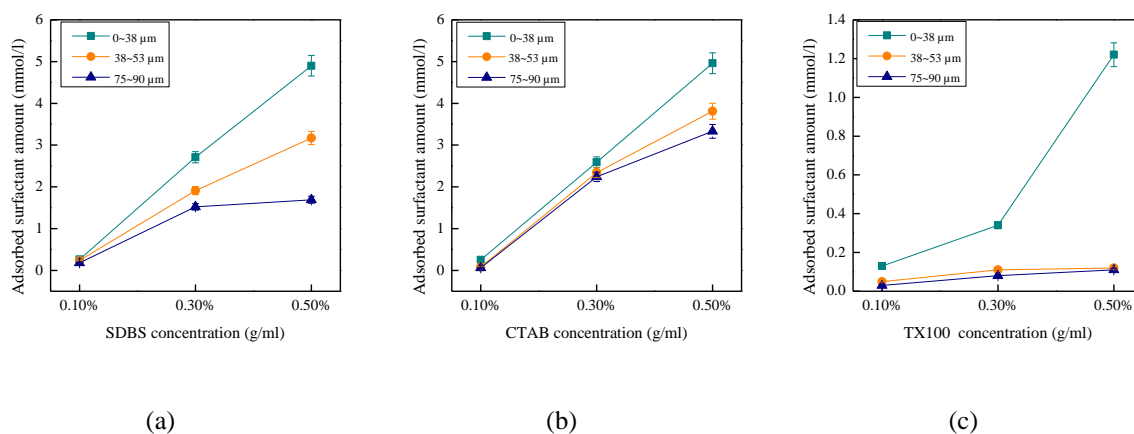


Figure 62. Variation of the adsorbed surfactant amounts on three size coals. (a) SDBS, (b) CTAB, (c) TX100.

## 6.3.3 Effects of temperature on the surfactant adsorption on coals

### 6.3.3.1 The effect of temperature on the zeta potential

The adsorption process of surfactants on the solid surface can be significantly influenced by temperature in a positive as well as negative way. For SDBS, the value of zeta potential increased when the temperature raised from 25  $^{\circ}\text{C}$  to 30  $^{\circ}\text{C}$  but it failed to further improve the zeta potential when the temperature further raised to 40  $^{\circ}\text{C}$ . For CTAB, higher temperature only improved the zeta potential at low concentration of 0.10% CTAB, and big drops were observed on zeta potentials when surfactant concentration over 0.10%. For TX100, the values of zeta potential at 30  $^{\circ}\text{C}$  were greater than those at 25  $^{\circ}\text{C}$  and 40  $^{\circ}\text{C}$ .

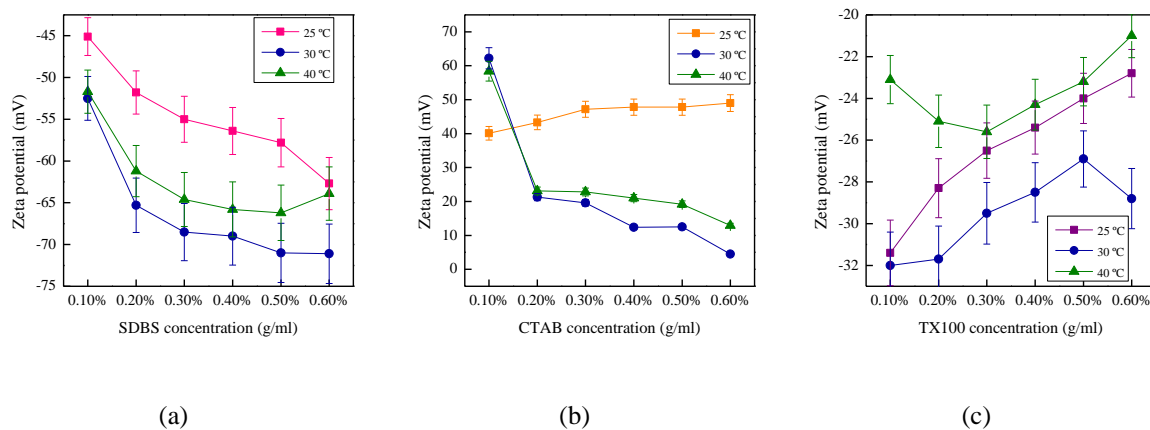


Figure 63. Zeta potential of coal particles in three surfactants at various temperatures

### 6.3.3.2 The effect of temperature on the adsorbed amount

Figure 64 recorded the change of surfactant adsorbed amount within temperature above the CMC level for three surfactants. It can be seen that overall, the surfactant adsorption capacity improved within the rising temperature for all these surfactants with concentrations are over CMC. For example, the adsorbed amount of 0.10% SDBS increased from 1.15 mmol/l at 25° C to 1.36 mmol at 40° C, and for 0.50% TX100, it went up from 0.44 mmol/l at 25° C to 0.60 mmol/l. The largest improvement was noticed for 0.10% CTAB that increased from 0.22 mmol/l to 0.41 mmol/l within the temperature increase from 25° C to 40° C. A consistent result obtained by Roam that higher adsorption capacities occurred under higher temperatures, he believed that higher temperature contributed to the increased amount of surfactant monomer molecules which can be easily adsorb on the coal surface (Roman Marsalek et al., 2011).

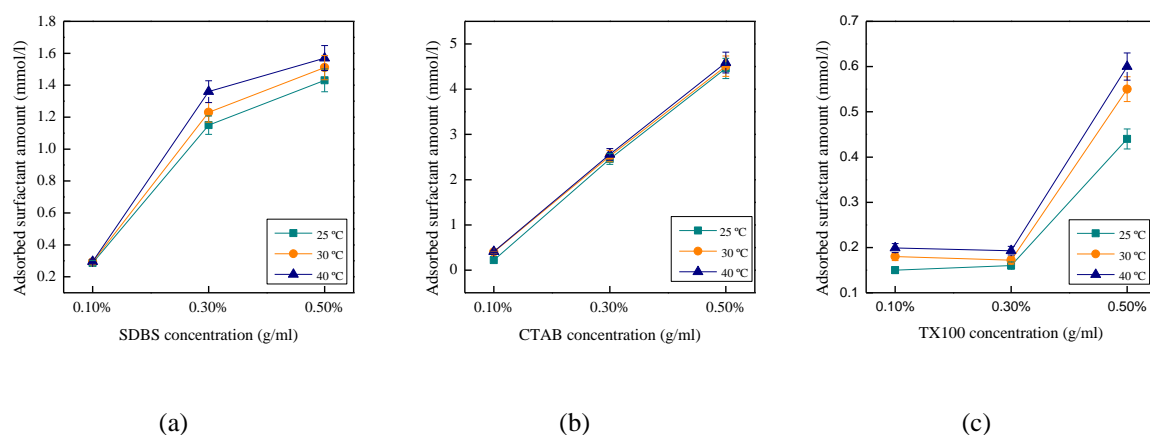


Figure 64. Adsorbed surfactant amounts on coal particles at various temperatures, (a) SDBS, (b) CTAB, (c) TX100

### 6.3.4 Effects of additives on the surfactant adsorption on coals

#### 6.3.4.1 The effect of additives on the zeta potential

It can be observed in Table 14 that, in general, the additives of these four salts lower the pH of surfactant solution at all the test concentrations. It is believed that zeta potential is sensitive to pH (Martí et al., 2003; Pollastri et al., 2014) and that was one reason leading in zeta potential change as shown in Table 15.

The impact of salts addition on coal surfactant solution zeta potential values varied different surfactants. For SDBS, generally, the trend of zeta potential values was increasing with the low concentrations of 0.10% and 0.20% salt additives, but it dropped below the original zeta potential values (without additives) when keep increasing the additives concentration to 0.40%. For example, 0.10% Na<sub>2</sub>SO<sub>4</sub> and 0.20% Na<sub>2</sub>SO<sub>4</sub> lifted the zeta potential of 0.10% from -45 mV to -50 mV and -57 mV approximately, respectively, but 0.40% Na<sub>2</sub>SO<sub>4</sub> dropped the zeta potential to around -43 mV.

The change of CTAB zeta potential by salt additions varied by salts. All the Na<sub>2</sub>SO<sub>4</sub> additives (0.10% to 0.40%) lowered the coal particles' zeta potential in surfactant solution at all the tested concentration. But the zeta potential changed when other three salt additives were presented. For example, lower concentrations (0.10% and 0.20%) of NaCl, KCl and KNO<sub>3</sub> increased the zeta potential while 0.40% these three salts lowered the zeta potential (except for 0.40% KNO<sub>3</sub>, it further helped increasing the zeta potential 0.10% CTAB).

For TX100, compared without salt additives, the decrease on the zeta potential value was found at all the test surfactant concentrations when adding these four additives, and smaller values were observed at higher concentrations of additives for NaCl, KCl and KNO<sub>3</sub>.

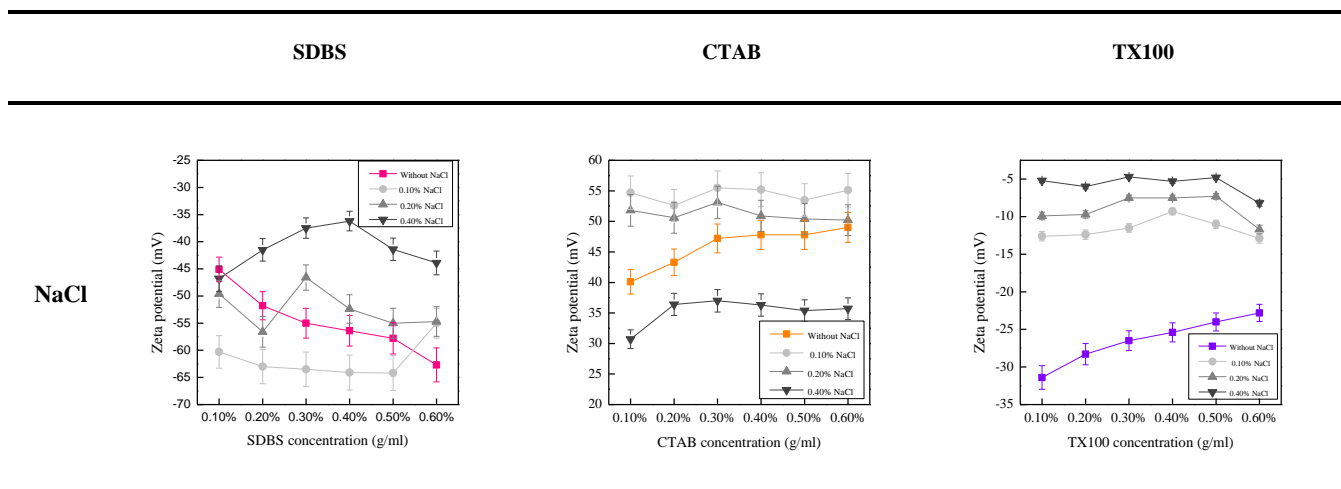
Table 14. pH of coal particles surfactant solution with different additives

Surfactant and its concentration	pH					
	Surfactant only	Surfactant+ 0.10% NaCl	Surfactant+ 0.10% Na <sub>2</sub> SO <sub>4</sub>	Surfactant+ 0.10% KCl	Surfactant+ 0.10% KNO <sub>3</sub>	
SDBS	0.10%	4.50	4.38	4.30	4.00	4.22
	0.20%	4.70	4.35	4.25	4.04	4.20
	0.30%	4.79	4.34	4.35	4.12	4.20
	0.40%	4.66	4.19	4.32	4.19	4.18

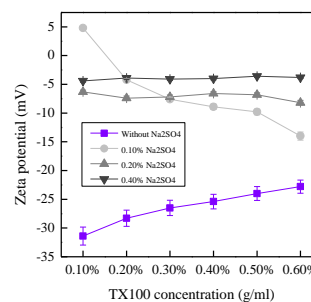
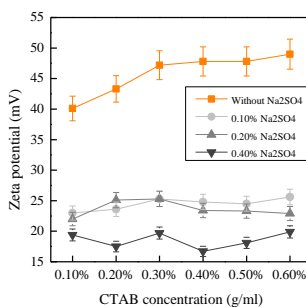
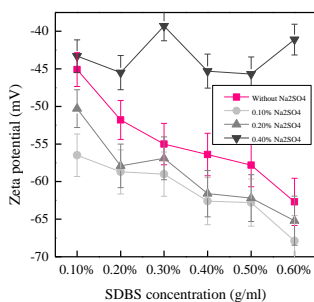


CTAB	0.50%	4.51	4.18	4.44	4.24	4.23
	0.60%	4.43	4.00	4.40	4.26	4.25
	0.10%	3.97	3.86	3.93	3.65	4.02
	0.20%	4.44	4.06	4.03	3.79	3.92
	0.30%	3.97	4.08	4.11	6.83	4.01
	0.40%	4.10	4.14	4.15	3.92	4.03
	0.50%	4.13	4.11	4.15	3.95	4.05
	0.60%	4.11	4.10	4.17	4.02	4.04
TX100	0.10%	5.79	4.08	4.62	4.69	4.74
	0.20%	5.60	4.15	4.55	4.12	4.32
	0.30%	5.77	4.19	4.53	4.21	4.36
	0.40%	4.92	4.18	4.49	4.27	4.38
	0.50%	4.78	4.22	4.45	4.19	4.26
	0.60%	4.98	4.22	4.49	4.21	4.30

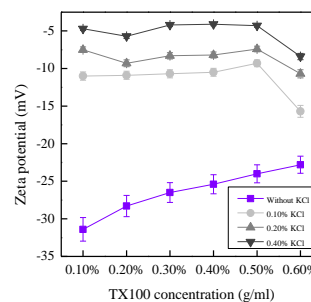
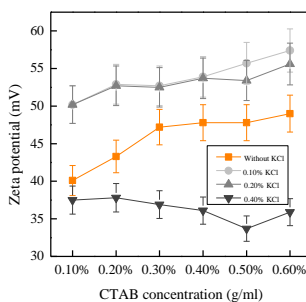
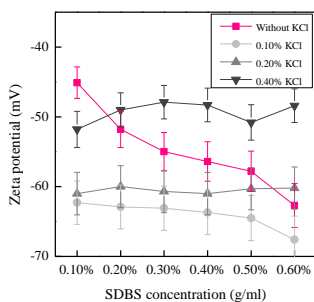
Table 15. Zeta potential of coal particles in presence of surfactants and additives



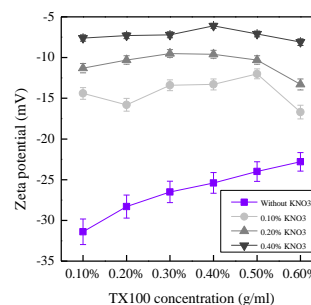
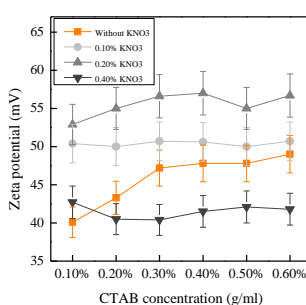
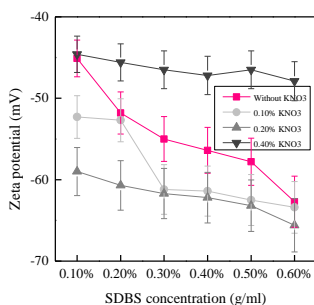
**Na<sub>2</sub>SO<sub>4</sub>**



**KCl**



**KNO<sub>3</sub>**

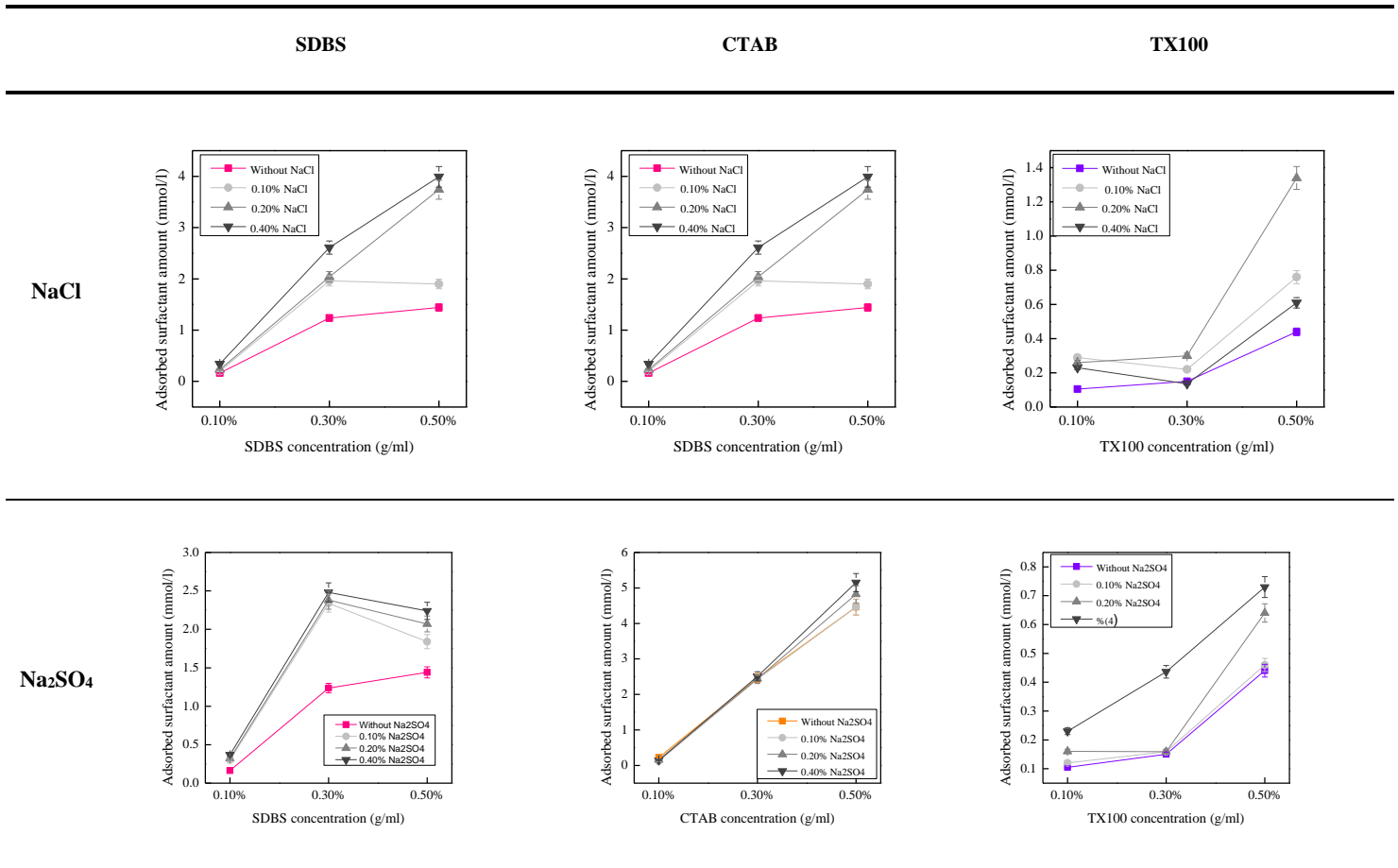


### 6.3.4.2 The effect of the additives on the adsorption amount

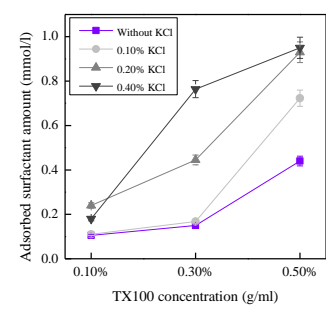
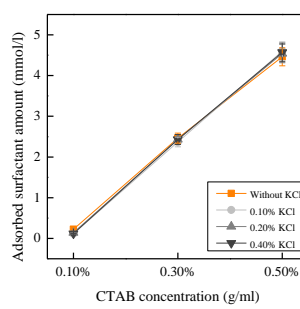
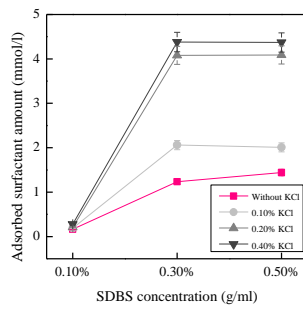
Table 16 demonstrated the impact of salt addition on adsorbed surfactant amounts on coal particles. For SDBS, It can be noticed that these four salts all improved its adsorbed amounts on coal particles with all the SDBS test concentrations. This finding is consistent with the results of the sink test and the surface tension test in Chapter 5 as well as the previous studies. In this study, at the low SDBS concentration of 0.10%, all these four salt additions did not enhance the SDBS adsorption too much, and apparent improvements were found at 0.3% SDBS but the addition not help to further increase the adsorbed amount for 0.50% SDBS except for low concentration NaCl. Among these four salts, KCl and KNO<sub>3</sub> improved more adsorbed SDBS amount than NaCl and Na<sub>2</sub>SO<sub>4</sub>, also increasing the concentration of KCl and KNO<sub>3</sub> from 0.1% to 0.2% resulted in a rise in adsorbed SDBS amount but a higher concentration of

0.4% failed to further increase the improvement. Grigg also found that an abrupt increased in anionic surfactant adsorption can be caused on negatively charged surfaces (coal surface is negatively charged that mentioned in section 2.2.1) due to an improvement in charge sites, and increasing the salts concentration in surfactant solution can lead to that improvement (G. Xu, Chen, et al., 2018). For CTAB, no obvious changes were observed on adsorbed amount except adding  $\text{KNO}_3$  which lower the CTAB adsorbed amount at all the tested concentrations. All salts with different concentrations improved the TX100 adsorbed amount except for when 0.2% and 0.4% of  $\text{KNO}_3$  adding to 0.5% TX100. But the change trends of improvement varied in different salts. For example, for both  $\text{Na}_2\text{SO}_4$  and  $\text{KCl}$ , the overall trend of absorbed TX100 went up with increasing salt concentrations, but for  $\text{NaCl}$ , the performance of a higher concentration of 0.4% was not as good as the lower concentrations of 0.1% and 0.2%.

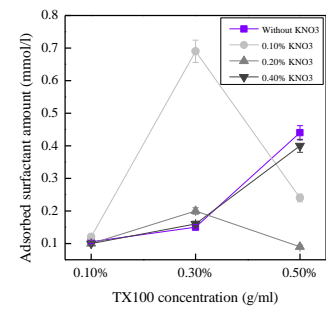
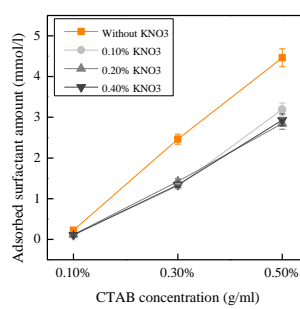
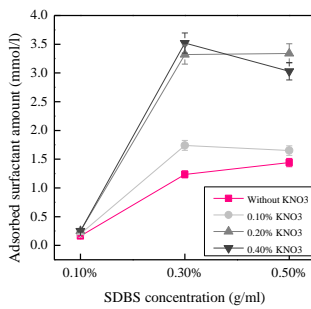
Table 16. Adsorbed surfactant amounts on coal particles with different additives



KCl



KNO<sub>3</sub>



## 6.4 Conclusions

The zeta potential and adsorption of surfactant on coal particles at different conditions were investigated in this study. The following conclusions can be concluded based on the experiment results.

The coal surface was negatively charged and the addition of surfactant changed its zeta potential. The anionic surfactant enlarged the negative value of coal particle zeta potential and the cationic surfactant can converted the zeta potential from negative value to positive value. But no obvious changes were observed for non-ionic surfactant. Prolong the stirring time over the equilibrium time not help to change the zeta potential. The order of zeta potential values for three rank coals in anionic and cationic surfactant was: anthracite > subbituminous > lignite, while an opposite order was observed in non-ionic surfactant. The larger difference zeta potential were found in anionic and non-ionic surfactant with around 20 to 25 mV. In surfactants of SDBS and CTAB at all the test concentrations, the finer coal particles had the lager zeta potential. However, an opposite result was found in the TX100. The impact of temperature on zeta potential was studied and the results indicated continue to increasing the temperature failed to lift all the coal particles' zeta potential at tested concentrations. The addition of salts to surfactants affected the zeta potential varied different surfactants with

various concentrations, and it was believed that the changed pH after salts addition was one reason contributed to that.

For all the surfactants, the adsorbed amount on the coal particles increased with the increasing surfactant concentration. The lignite adsorbed more SDBS but less TX100 than subbituminous and anthracite at all the tested concentrations, and anthracite adsorbed the most amount of CTAB at the higher concentration. The finer coal particles adsorbed the more surfactant amount for all the surfactants at each concentration. Overall, the surfactant adsorption capacity improved within the rising temperature for all these surfactants with concentrations are over CMC. The adsorption of SDBS was greatly increased by adding all of the salts while no obvious improvement was found for CTAB and TX100.

## 7 Chapter 7

### **Numerical study of coal dust behaviours and experimental investigation on coal dust suppression efficiency of surfactant solution by using wind tunnel tests**

The work in this chapter has been published on the journal of Energy Sources, Part A: Recovery, Utilization, and Environmental Effects.

It was entirely written by Yinping Chen and reviewed by Ping Chang and Dr. Guang Xu

Please cite this paper as:

Chang, P., Chen, Y., Xu, G., Huang, J., Ghosh, A. and Liu, W.V., (2019). Numerical study of coal dust behaviours and experimental investigation on coal dust suppression efficiency of surfactant solution by using wind tunnel tests. Energy Sources, Part A: Recovery, Utilization, and Environmental Effects, 1-16.

<https://doi.org/10.1080/15567036.2019.1639855>

The effectiveness of surfactants on coal dust particles by using static methods are conducted in Chapter 5 and Chapter 6. This chapter uses the dynamic test, the wind tunnel test, to evaluate the effectiveness of surfactants on reducing coal dust concentrations. These wind tunnel test are conducted based on the investigation result of coal dust distribution characteristics and air flow behaviours by employing CFD modelling.

## **Abstract**

Coal dust is one of the most serious hazards on mine sites. Water spray with surfactant added is an effective way control the coal dust. In this study, wind tunnel experiments were designed to evaluate the surfactant on coal dust suppression efficiency. A Computational Fluid Dynamics (CFD) modelling was first constructed in to investigate the coal dust particles movement characteristics, and the stimulation results were provided for the wind tunnel design. The CFD results indicated that the location of the nozzle placement and the time for the water spraying start time. The coal dust suppression efficiency was then evaluated in presence of different surfactants. The experiment results suggest that the anionic and non-ionic surfactants are helpful to improve the suppression efficiency while the cationic surfactant failed to increase it. For the SDBS at various concentrations, the highest suppression efficiency is achieved when at 0.20%. Compared with the larger coal dust particles, the finer ones are more difficult to capture for both water and SDBS surfactant solution. The results also demonstrate the potential application of this surfactant on coal mine industries.

Keywords: Computational fluid dynamics; Coal dust suppression; Surfactant; Wind tunnel test

## **7.1 Introduction**

Coal dust is the main hazard for the underground coal mines. Due to the ultrafine characteristics of the coal dust, it not only affects the mining facilities operations, but also has adverse impacts on miners' health. Studies have shown that long-term exposure to higher coal dust concentration working environment can lead to serious lung diseases, such as Coal Workers' Pneumoconiosis (CWP) and lung cancers (Ayoglu, Acikgoz, Tutkun, & Gebedek, 2014a; DE Pollock et al., 2010; G. Xu, Chen, et al., 2018). It was reported that the mortality of former underground coal miners could be about 1,500 due to the CWP each year in the US (James, 2011).

To decrease the health hazard which is caused by coal dust during the mining activity, an effective control method is important. Currently, three approaches are primarily used on the

underground coal, which include ventilation, water spraying and foam technology (X. W. Ren, Wang, Kang, & Lu, 2012; Rider & Colinet, 2007). However, due to the complicated underground working environment, these methods are limited. For example, ventilation requires substantial quantity of airflow and the commonly used fans on mine site are barely meet this requirement (X. W. Ren et al., 2012). In addition, in the development face, the high velocity of airflow may easily blow up the coal dust near the floor. The foam injection equipment for foam technology is usually large and complicated, so it is hard to be placed in the space-limited areas in underground mine. For the water spraying to effectively capture the coal dust, the sizes of water droplets are required to be similar as the coal dust particles' diameter (T. Ren et al., 2014). However, the coal dust particles' diameters are various. Thus, the traditional water spraying device cannot control the coal dust effectively (Goodman, 2000; Douglas Pollock & Organiscak, 2007). For this reason, the water spraying with added surfactant has been suggested by many researchers to control coal dust effectively (Kim & Tien, 1995; Musselman & Chander, 2002).

In the past decades, many researchers studied the application of surfactant on coal dust suppression by using static tests (S. Chander, B. Mohal, & F. Aplan, 1987; Cheng, Urata, Yagihashi, & Hozumi, 2012b; Glanville & Haley, 1982). Few studies by using a wind tunnel test can be found. The advantages and disadvantages were illustrated by Xu et al.(G. Xu, Chen, et al., 2018). The wind tunnel test is a direct method to evaluate the surfactant while it is a challenge to construct a wind tunnel to conduct the experiment because it is time-consumable and non-cost-effective. A detailed description about the wind tunnel test was described in a previous study(G. Xu, Chen, et al., 2018).

Computational fluid dynamics method is widely used in mining engineering field to study the gas-solid two-phase flows characteristics. Especially for the health related field, a number of literatures investigated the coal dust dispersion and distribution characteristics by using simulation modelling technique. Ren et al.(T. Ren et al., 2014; T. Ren, Wang, & Zhang, 2018; Z. Wang & Ren, 2013) conducted a number of studies to investigate the coal dust distribution in different scenarios, related coal dust management method were provided according to the simulation results. Hu.(S. Hu et al., 2016; S. Hu, Wang, & Feng, 2015) studied the distribution characteristics of the coal dust after blasting in an underground development face by CFD modelling and the results were validated by the onsite measurement data. Torano (J Torano, Torno, Menéndez, & Gent, 2011) evaluated the coal dust behaviours under two different ventilation systems in underground mine site by CFD technique and validated the results with



the field measurements, the results demonstrated that the CFD models are reliable to predict the coal dust movement. Geng and Wang (F. Geng et al., 2017; Yingchao Wang, Luo, Geng, Li, & Li, 2015) investigated the coal dust behaviours in an underground roadway under a hybrid ventilation system and the distributions of different sizes of coal dust were presented. The results showed a good agreement with the experiment data. Kurnia (Kurnia, Sasmito, Hassani, & Mujumdar, 2015) studied the dust dispersion patterns under various ventilation conditions by changing the position of wind brattices, the most effective ventilation design for dust control was suggested based on the simulation results. All the research suggests that CFD is a reliable and efficient method to study the coal dust movement behaviours in the airflow.

In this study, the CFD modelling was first built to investigate the air flow behaviours and coal dust distribution characteristics in the wind tunnel. The Eulerian-Lagrangian method was used to calculate the two-phase fluid. Then the simulation data was validated by the experimental measurements. Based on the validated simulation results, a series of wind tunnel experiments were then designed and the effectiveness of the coal dust suppressant in presence of surfactants were evaluated.

## **7.2 Experimental setup**

The designed wind tunnel used in this study is shown in Figure 65. It was built with the iron frame and acrylic plastic. The acrylic plastic was fixed on the iron frame by using bolt and screw, and silicon sealant and tape were applied to seal the gap between the frame and acrylic. The wind tunnel was fixed on the stand that has a distance of 80 cm from the floor.

The wind tunnel has a cross section of 0.5 m × 0.5 m and a 4.5 m length, as shown in Figure 66. The basic structure of the wind tunnel includes three sections: dust generator section, water spray system and coal dust collection with the exhaust fan. The coal dust generator pipe was assembled by several plastic pipes and installed in the wind tunnel, which was used to emit the coal dust particles, as shown in Figure 67. The diameter of the pipe is 0.02 m, and the dust generator pipe is 0.22 m from the wind tunnel inlet. In the experiment, a cyclone dust collector was connected to the outlet of the wind tunnel, which acted as an exhaust fan and provide the designated airflow. To make the airflow more homogeneous and stable in the wind tunnel, the airflow went through a mesh before it entered the wind tunnel. After the exhaust fan working 5 minutes, the dust generator started to work. In this section, the coal dust samples were placed in the cylindrical container with a blender. The coal dust was loaded in the vibrating feeder in

a continuous motion with the loading speed of  $5.46 \times 10^{-5}$  kg/s. Then, it was carried to the wind tunnel by using a blower.

After the coal dust was blown into the wind tunnel and reached a stable concentration. A nozzle (CPB1322 TEEJETBODY and D12-45HSS DISC&CORE, Spraying Systems Co. Pty. Ltd.) installed to spray water droplets to capture the coal dust. The spray water was drawn by using a water pump (Ozito, Constant Pressure Pump 800W) with a 4.97 l/min water flow from a water tank, and the duration of water spray was last around 2 minutes. The coal dust concentration was measured by a real time aerosol monitor (TSI DustTrak™ II Handheld Aerosol Monitor) before and after the water spraying. The monitor was calibrated by the manufacture before it was employed for this study. The remind coal dust that not captured by water droplets was drawn into the dust collector (Grizzly G0637 7-1/2 HP 3-Phase cyclone dust collector. USA) to keep the environment friendly.

The coal dust suppression efficiency can be calculated by the using the following equation, where  $\eta$  (%) is the coal dust suppression efficiency,  $C_{before}$  and  $C_{after}$  ( $\text{mg}/\text{m}^3$ ) present the coal dust mass concentration before and after applying water spray, respectively.

$$\eta = \frac{C_{before} - C_{after}}{C_{before}} \times 100\% \quad \text{Equation 17}$$



(a) Dust generator



(b) Wind tunnel



(c) Spray nozzle



(d) Water pump and water tank



(e) Coal dust monitor



(f) Cyclone dust collector and exhaust fan

Figure 65. Experimental apparatus for the wind tunnel test

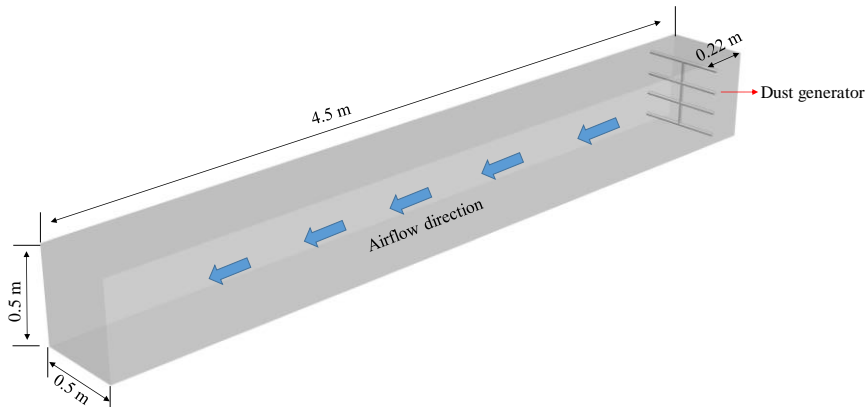


Figure 66. Geometric model of the wind tunnel

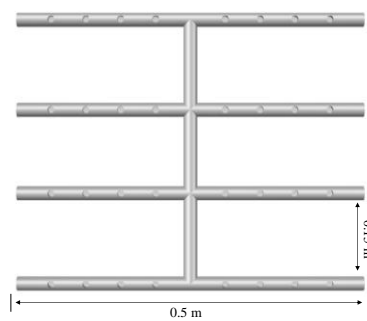


Figure 67. Dust generator pipe

The coal samples used in this study were obtained from the Premier Coal Limited, Western Australia. The coal lamp was crushed, pulverised, oven dried and sieved to 0~38  $\mu\text{m}$  size range. All the preparation procedures were followed the standards of ATSM (ASTM, 2012a, 2012b, 2015).

Three surfactants, SDBS, CTAB and TX 100, were supplied by Sigma Aldrich. They are anionic, cationic and non-ionic surfactant, respectively. The surfactant solution was prepared by dissolving the calculated amount surfactant into water. To obtain more accurate data, each wind test was repeated three times.

## 7.3 Wind tunnel design using CFD

Wind tunnel experiments are time and materials consuming. Before conducting the experiments, it is important to carefully design the experimental parameters, such as coal dust release time and duration, and the location of the nozzle. Such parameters can be obtained by the CFD simulation. Thus, the 3D modelling of the wind tunnel is first built to provide the needed parameters.

### 7.3.1 CFD model setup

In this study, the airflow is assumed as incompressible; no head transfer is considered. The details of simulation parameters are listed in Table 17. The airflow field is first simulated to a steady-state, then the coal dust particles are emitted from the dust generator for 300 s based on the experiment time.

Table 17. Simulation parameters

Parameters	Values
Air density (kg/m <sup>3</sup> )	1.127
Airflow rate of the cyclone dust collector (m <sup>3</sup> /s)	0.111
Airflow rate of the dust generator (m <sup>3</sup> /s)	0.011
Air viscosity (Pa·s)	$1.87 \times 10^{-5}$
Air kinematic viscosity (m <sup>2</sup> /s)	$1.66 \times 10^{-5}$
Coal dust flow rate (kg/s)	$5.46 \times 10^{-5}$
Coal dust size distribution model	Rosin-Rammler
Diameter range (μm)	0~38
Mean coal dust diameter (μm)	15

### 7.3.2 Governing Equations

The Eulerian frame is used to describe the airflow behaviours. The continuity and momentum equations are given as below (Moukalled, Mangani, & Darwish, 2016):

$$\nabla \cdot \mathbf{u}_g = 0 \quad \text{Equation 18}$$

$$\begin{aligned} \frac{\partial \rho_g \mathbf{u}_g}{\partial t} + \nabla \cdot (\rho_g \mathbf{u}_g \mathbf{u}_g) \\ = -\nabla p + \nabla \cdot \left[ \mu (\nabla \mathbf{u}_g + (\nabla \mathbf{u}_g)^T) - \frac{2}{3} \mu (\nabla \cdot \mathbf{u}_g) \mathbf{I} - \rho_g k \right] + \rho_g \mathbf{g} \end{aligned} \quad \text{Equation 19}$$

where  $\mathbf{u}_g$ ,  $\rho_g$ ,  $p$ ,  $\mu$ ,  $k$ ,  $\mathbf{I}$  and  $\mathbf{g}$  is the gas phase (air) velocity, density, pressure, viscosity, turbulent kinetic energy, unit tensor and gravitational acceleration, respectively.

The standard k-epsilon is one of the most commonly used turbulent models in the simulation of coal dust dispersion in airflow due to its accuracy and efficiency. A number of studies have applied this model successfully for the similar computational work, thus this turbulent model is also used in this study (S. Hu et al., 2016; T. Ren et al., 2014; J Torano et al., 2011; Javier Torano, Torno, Menendez, Gent, & Velasco, 2009). The equations are given as below:

$$\frac{\partial(\rho_g k)}{\partial t} + \nabla \cdot (\rho_g k \mathbf{u}_g) = \nabla \cdot \left[ \left( \mu + \frac{\mu_t}{\sigma_k} \right) \nabla k \right] + G_k - \rho_g \varepsilon \quad \text{Equation 20}$$

$$\frac{\partial(\rho_g \varepsilon)}{\partial t} + \nabla \cdot (\rho_g \varepsilon \mathbf{u}_g) = \nabla \cdot \left[ \left( \mu + \frac{\mu_t}{\sigma_\varepsilon} \right) \nabla \varepsilon \right] + C_{1\varepsilon} \frac{\varepsilon}{k} G_k - C_{2\varepsilon} \rho_g \frac{\varepsilon^2}{k} \quad \text{Equation 21}$$

where  $\mu_t$  and  $G_k$  are given by:

$$\mu_t = C_\mu \rho_g \frac{k^2}{\varepsilon} \quad \text{Equation 22}$$

$$G_k = \mu_t (\nabla \mathbf{u}_g + (\nabla \mathbf{u}_g)^T) \nabla \mathbf{u}_g \quad \text{Equation 23}$$

The value of model coefficients  $C_\mu$ ,  $C_{1\varepsilon}$ ,  $C_{2\varepsilon}$ ,  $\sigma_k$ ,  $\sigma_\varepsilon$  are 0.09, 1.44, 1.92, 1, and 1.3, respectively. The governing equations for the airflow is solved by SIMPLE algorithm and the convective derivatives is governed by a second order numerical scheme which calls Gauss LinearUpwind. The motion of coal dust particles is governed by the Lagrangian method, which is described by Newton's second law. There are many forces could influence the particles' movement, such as virtual mass force, Brownian force, thermophoretic force, fluid pressure gradient force, drag force, gravity, Saffman's lift force and turbulent dispersion effects. Virtual

mass force impacts significantly on the large size particles, which could be ignored in this study. Compared to turbulent dispersion effects, Brownian force is too small to influence the particles, thus Brownian force is also neglected. No heat transfer is considered in this study, thus thermophoretic is ignored. In this study, only the forces, like fluid pressure gradient force, drag force, gravity, Saffman's lift force and turbulent dispersion effects, which significant impact on the coal particles' motion are considered (S. Hu et al., 2016; G. Xu, Chang, Mullins, Zhou, & Hu, 2018). The particles' collision is an important force in particle simulation. However, this interaction can be ignored when the particulate volume fraction is less than  $10^{-6}$  (Afkhami, Hassanpour, Fairweather, & Njobuenwu, 2015; Van der Hoef et al., 2006). Thus, one way coupled simulation is applied in this study. In this study, since the coal dust volume fraction less than  $10^{-6}$ , the movement of coal dust has a negligible effect on the airflow (Afkhami et al., 2015; Van der Hoef et al., 2006). Only the airflow forces act on the coal particles is considered, the particle motion does not affect the airflow behaviours. The particle motion equation is given by (Patankar & Joseph, 2001):

$$m_p \frac{d\mathbf{u}_p}{dt} = m_p \mathbf{g} + \mathbf{F}_d + \mathbf{F}_g + \mathbf{F}_p + \mathbf{F}_l \quad \text{Equation 24}$$

where  $m_p$ ,  $\mathbf{u}_p$ ,  $\mathbf{F}_p$ ,  $\mathbf{F}_l$  and  $\mathbf{F}_g$  is the particle mass, particle velocity, fluid pressure gradient force, lift force and the turbulence dispersion effects on the particles, respectively.  $\mathbf{F}_d$  is the drag force, which is given by:

$$\mathbf{F}_d = \frac{3 C_d Re}{4 d_p} |\mathbf{u}_p - \mathbf{u}_g| (\mathbf{u}_p - \mathbf{u}_g) \quad \text{Equation 25}$$

where  $d_p$  stands for particle diameter,  $Re_p$  is the particle Reynolds number,  $C_d$  stands for the drag coefficient, which is expressed by:

$$C_d = \begin{cases} \frac{24(1 + 0.15Re^{0.687})}{Re_p} & Re \leq 1000 \\ 0.44 & Re > 1000 \end{cases} \quad \text{Equation 26}$$

### 7.3.3 Mesh independent study

The accuracy of the simulation results highly depends on the meshes used. Generally, a denser mesh gives a better modelling results. However, the computation is expensive for the large size of mesh. Thus, mesh independent study is a significant part in CFD. To conduct the mesh

independent study, 3 different size of meshes, coarse mesh, medium mesh and fine mesh, were tested in this study. The total number of elements are about 0.14 million, 0.35 million, and 0.76 million for coarse mesh, medium mesh and fine mesh, respectively. Hybrid meshes were used in this study. Medium mesh is presented in Figure 68 as a representative. For the geometry complex part, near the dust generator pipe, the unstructured mesh was used. The structured mesh was used for the rest part of the model.

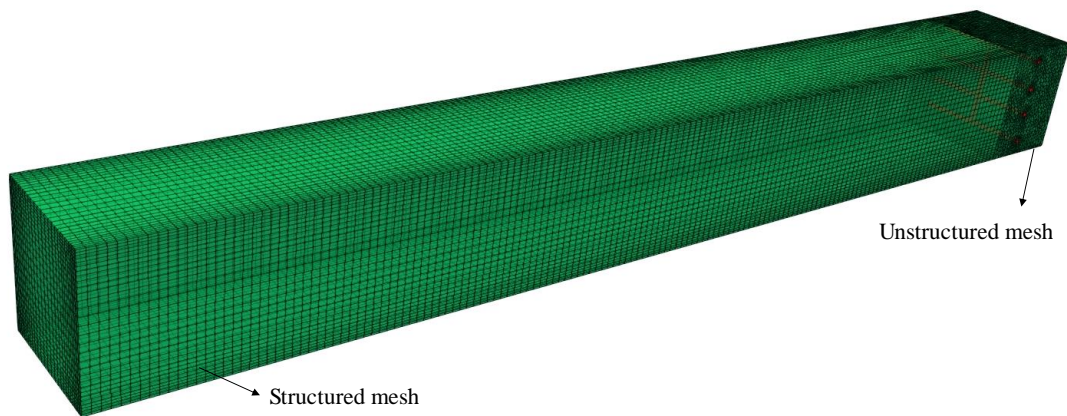


Figure 68. Overview of the medium mesh

Airflow velocity profiles at three monitor lines are presented in Figure 69 to check the mesh independence. Monitor line 1, line 2 and line 3 are at the horizontal centreline 1 m, 3 m and 4 m from the wind tunnel inlet, respectively. As can be seen, the airflow velocity profiles of medium mesh are highly matching that of fine mesh for the three monitor lines, which demonstrates that the mesh independent has been achieved by using the medium mesh. The spacing of the first layer of the medium mesh is 0.008 m according to a given  $Y^+=15$ , the increase ratio for the mesh size is set as 1.1.

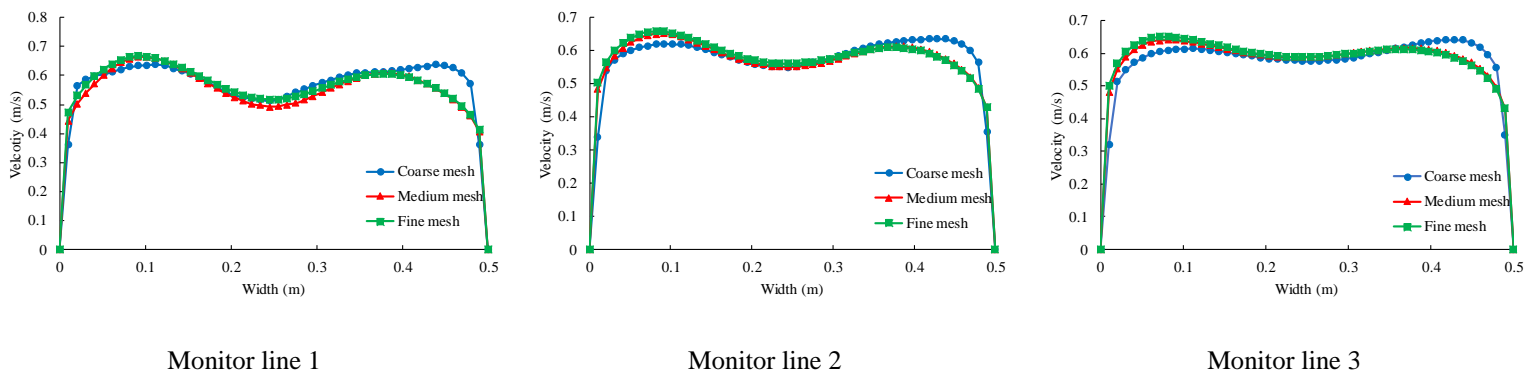


Figure 69. Airflow velocity profiles for different meshes

### 7.3.4 Model validation

Model validation is an important part in the CFD study, which insures the accuracy of simulation results. To validate the model, the air velocity of nine monitor points at a cross-sectional plane (4.25 m from the inlet) were measured. The location of nine monitor points is illustrated in Figure 70. The comparison between experimental data and simulation results at the monitor points is given in Table 18. As can be seen, the maximum error between the measured and simulated data is 4.8 %, and the average error is 2.6%. Such a small deviation indicates that the current simulation setting is appropriate and the simulation results are acceptable.

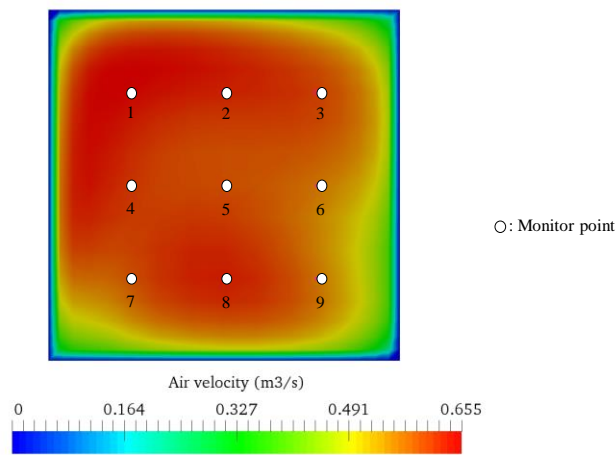


Figure 70. Location of monitor point

Table 18. Comparison between measured data and simulation results

Monitor point number	1	2	3	4	5	6	7	8	9
Experimental data (m/s)	0.63	0.60	0.59	0.59	0.56	0.55	0.58	0.57	0.56
Simulation result (m/s)	0.60	0.58	0.58	0.57	0.56	0.53	0.56	0.56	0.55
Error (%)	4.8	3.3	1.7	3.4	0.0	3.6	3.4	1.8	1.8



## 7.3.5 CFD model

### 7.3.5.1 Airflow distribution

The distribution of coal dust highly depends on the airflow behaviours. Thus, it is important to understand the airflow characteristics, especially for the airflow velocity and recirculation locations. Figure 71 and Figure 72 give the airflow velocity contour and vector at both middle horizontal and vertical cross-sectional planes. It can be seen that a low airflow velocity zone is generated in the middle area of the wind tunnel about 0.3-1 m from the wind tunnel inlet due to the block of the dust generator pipe. As the airflow flowing to the downstream, the air velocity distribute evenly gradually. From the velocity contour in Figure 72 (b), under the combined effect of exhaust fan and airflow from the dust generator pipe, several small turbulent areas occur near the middle area of the dust generator pipe. It is worth noting that the air velocity in these areas are quite lower than other locations. The coal dust may accumulate in these areas because of the low airflow velocity.

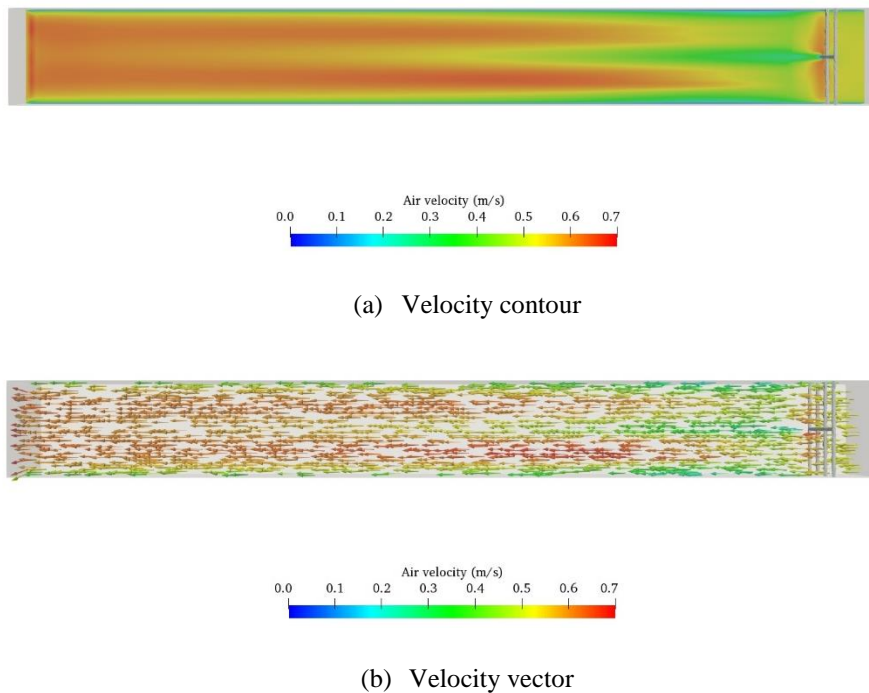


Figure 71. Airflow velocities at horizontal plane 0.25m above the wind tunnel floor

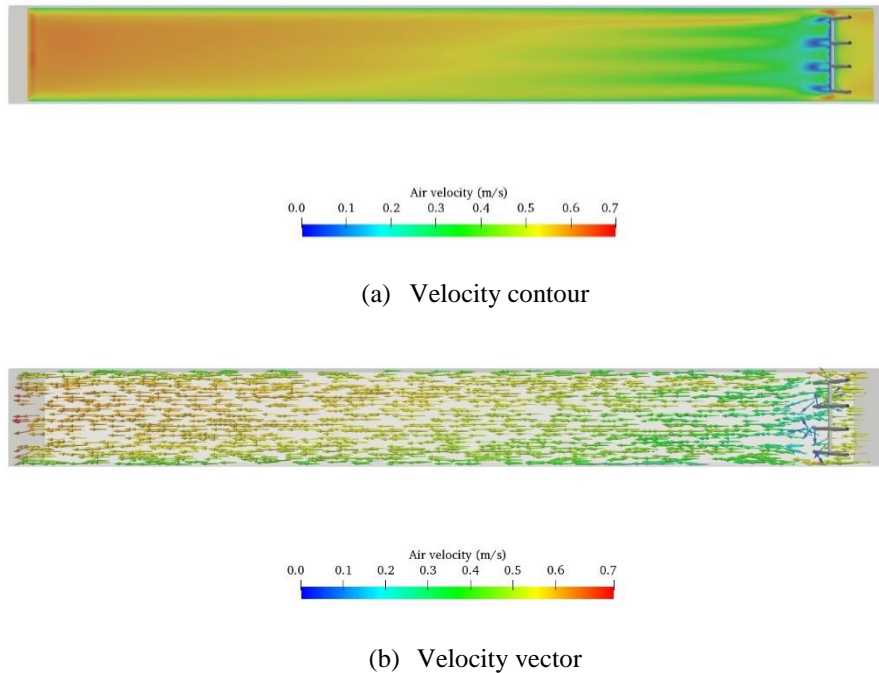
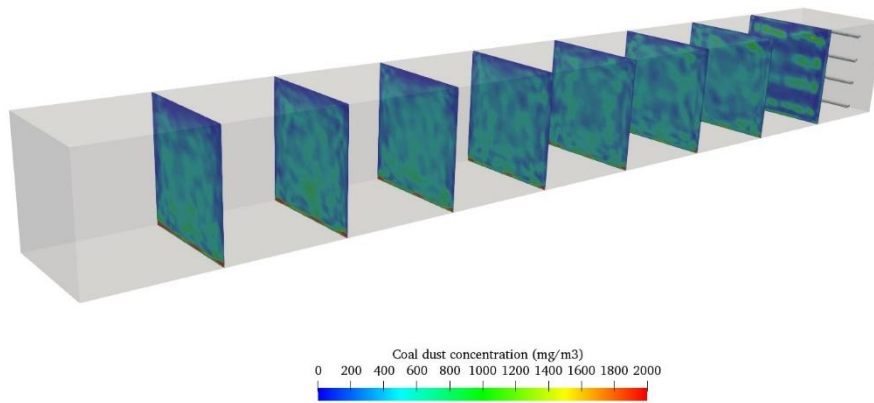


Figure 72. Airflow velocities at vertical plane 0.25m from the wind tunnel wall

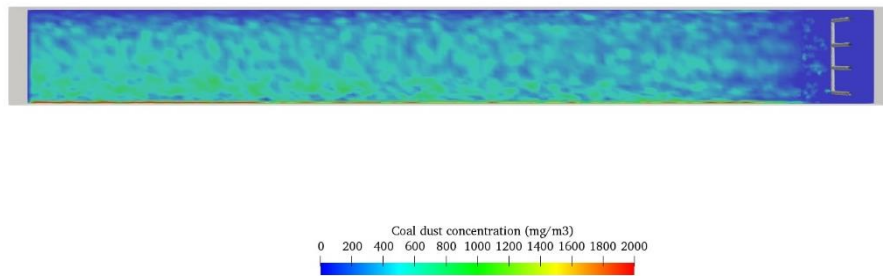
### 7.3.5.2 Coal dust distribution

To investigate the coal dust behaviours, Figure 73 illustrates the coal dust mass concentration distributions at difference cross-sectional planes and middle vertical cross-sectional plane at the end of the simulation ( $t = 300$  s). It can be seen that the coal dust mass concentration distributes near the inlet highly depend on the coal dust emit locations on the dust generator pipe. As the coal dust particles move to the downstream, they diffuse quickly to other areas. As shown in Figure 73 (b), the gravity impacts significantly on the coal dust particles. With the coal dust particles traveling with the airflow, they gradually drop to the bottom of the wind tunnel and result in a high concentration areas. It is noticed that the coal dust distribute relatively evenly at about 1.5 m from the inlet. Figure 74 illustrates the cross-sectional area weighted average mass concentration at different locations. As can be seen, the coal dust diffused quickly once emitted from the dust generator. And the area average concentration changes slightly within  $\pm 10$  mg from 1 m to 4.5 m. For this reason, it is reasonable to place the nozzle at 1.5 m from the inlet. To study the effectiveness of water spray with surfactants, the location of nozzle is important. If the nozzle is placed too far from the dust generator pipe, the water droplet could be carried out by the airflow without completely contact with the coal dust particles. If the nozzle is too close to the dust generator pipe, the water may inject to the dust generator pipe directly and the coal dust particles do not diffuse enough to conduct the

experiment. Thus, it is reasonable to place the nozzle 1.5 m from wind tunnel inlet according to the simulation results and the mentioned reasons.



(a) Cross-sectional planes



(b) Middle vertical cross-sectional planes

Figure 73. Coal dust mass concentration distributions

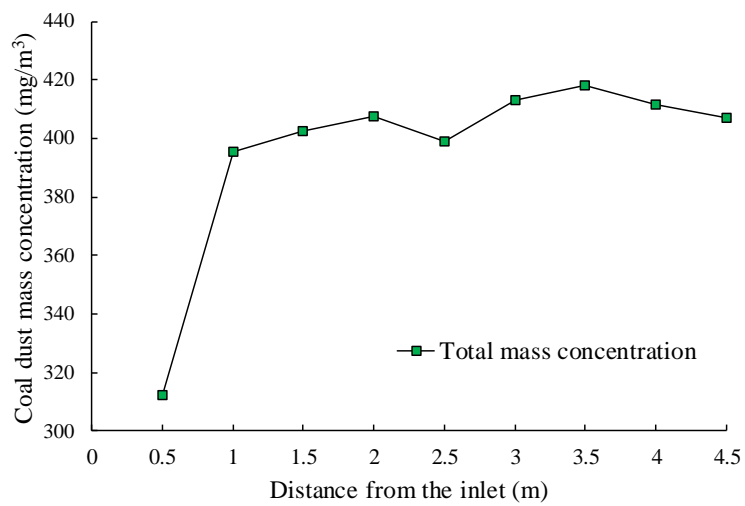


Figure 74. Coal dust mass concentration change at different locations

The vertical cross-sectional plane is 2.25 m from the wind tunnel inlet. It is noted in Figure 75 that the total coal dust concentration increased sharply to about 400 mg/m<sup>3</sup> at around 40 s. After that, although a slight fluctuation of the concentration is observed, the fluctuation is within  $\pm 10$  mg/m<sup>3</sup>. Thus, it is believed that a stable status has been achieved by the simulation. To conduct the wind tunnel experiment, the coal dust distribution should be under a steady state when determining its concentration before the coal particles go through the water spray area. Based on the simulation result, the water spray should start at least 40 s after the injection of the coal dust.

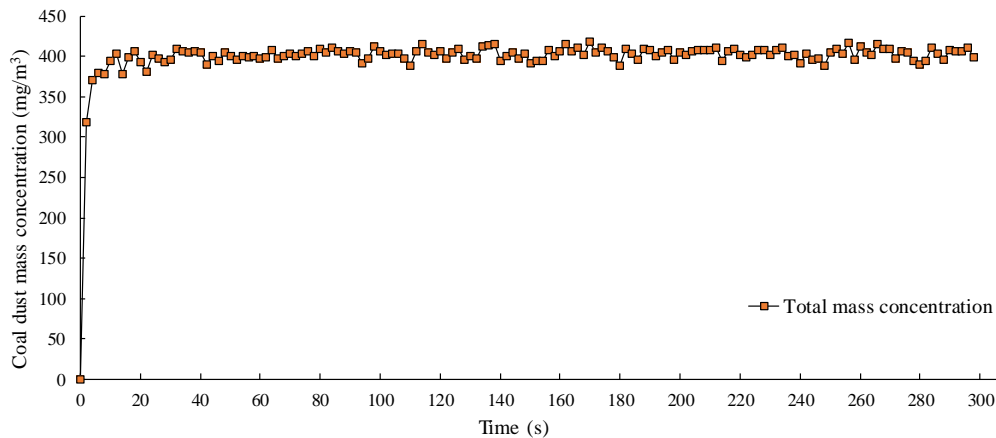


Figure 75. Coal dust mass concentration change during the simulation

## 7.4 Experimental results and discussions

The wind tunnel tests were designed based on the parameters suggested by the simulation results in the previous section. The nozzle location was designed in the middle cross section of the wind tunnel 1.5 m from the wind tunnel inlet. The coal dust was introduced to the wind tunnel for about 2 min, as the CFD results suggest that at least 40 s needed, to reach a stable concentration. After that, the water spray was injected to capture the coal dust. After the design of the wind tunnel experiments. The different types of coal dust suppressants with various concentrations were compared and the effectiveness of these suppressants on the coal dust reduction was also evaluated. Coal dust concentration sample was taken every 20 s and 10 samples were taken at the stable state at each experimental stage. After that, the suppression efficiency for different surfactants were calculated based on equation Equation 17.

### 7.4.1 Effect of surfactant type on suppression efficiency

A boxplot is a figure which presents the experimental data distribution information. It gives the information which includes the minimum value, the maximum value, the median, first

quartile, third quartile and mean value. To evaluate the suppression efficiency of difference surfactants, the boxplot was used in this study. Figure 76 presents the boxplot of suppression efficiency of water and other three surfactants. As can be seen, the solution type used for capturing coal dust particles is a significant factor to suppression efficiency. The suppression efficiency for water, 0.2% SDBS, 0.2% CTAB, and 0.2% TX100 is 47.08% (95% CI: 45.92%–48.24%), 63.09% (95% CI: 62.00%–64.18%), 40.93% (95% CI: 38.65%–43.21%) and 56.67% (95% CI: 53.21%–60.13%), respectively. Compared with water, both 0.2% SDBS and 0.2% TX100 present a higher suppression efficiency. However, the cationic surfactant CTAB failed to improve the suppression efficiency, which is less effective than even water. The efficiency of CTAB is 6.14% (95% CI: 3.13%–9.15%) lower than that of the water. It worth noting that the highest efficiency of the sample in the group of 0.2 % TX100 is similar to that of the 0.2% SDBS. To determine whether the efficiency of 0.2% SDBS different from that of the 0.2% TX100, the t-test of these two groups of data are shown in Table 19. T-test is usually used to investigate the difference of observations (Lalanne, 2006). As can be seen, the p-value is less than 5%, thus the data provide convincing evidence that there is a difference in the suppression efficiency of the two solutions. Thus, 0.2% SDBS provides the highest suppression efficiency, which is 16.01% (95% CI: 14.28%–17.74%) higher than that of water.

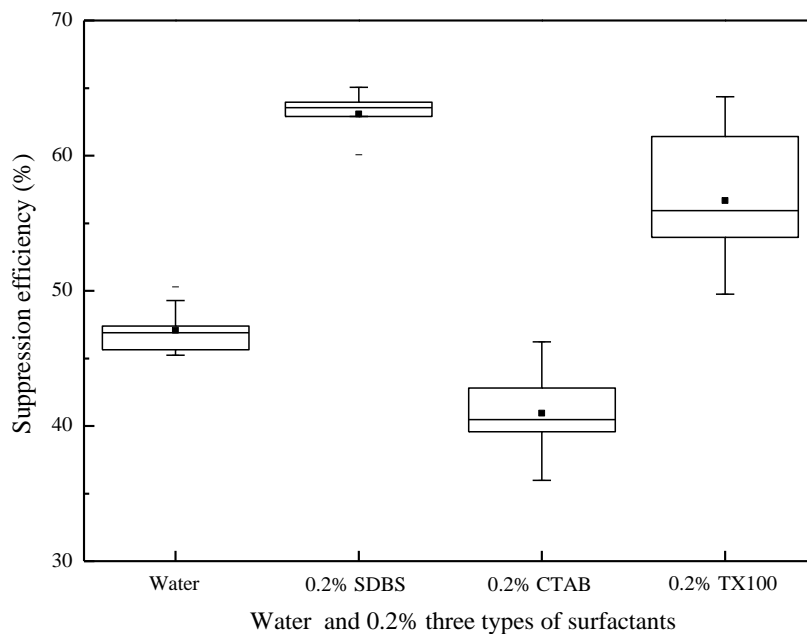


Figure 76. Coal dust suppression efficiency by using water and three surfactants at concentration of 0.20%

Table 19. T-test for the suppression efficiency of 0.2% SDBS and 0.2% TX100

	<i>0.2% SDBS</i>	<i>0.2% TX100</i>
Mean	63.08%	54.94%

Variance	0.0002	0.0003
Observations	10	10
Pearson Correlation	-0.0970	
Hypothesized Mean Difference	0	
df	9	
t Stat	10.96193042	
P(T<=t) one-tail	8.2884E-07	
t Critical one-tail	1.8331	
P(T<=t) two-tail	1.6577E-06	
t Critical two-tail	2.2622	

#### **7.4.2 Effect of surfactant concentration on coal dust suppression efficiency**

Previous section demonstrated that 0.2% SDBS had the highest suppression efficiency than other solution. However, the surfactant concentration also plays a significant role in suppress coal dust particles in the wind tunnel test. To evaluate the impact of SDBS concentration on the coal dust suppression efficiency and find out the optimum SDBS concentration on the coal dust capture, five SDBS concentrations (0%, 0.05%, 0.1%, 0.2%, and 0.3%) were compared. To investigate the difference among 3 or more group means in a sample, the analysis of variance (ANOVA) is commonly used (Lalanne, 2006). The ANOVA data of the suppression efficiency of different concentrations of SDBS is presented in Table 20. As can be seen, the p value is less than 5%, thus the data provide convincing evidence that SDBS concentration significantly impacts on coal dust suppression efficiency. The boxplot for the suppression efficiency of different concentrations of SDBS is illustrated in Figure 77. As shown, all the SDBS solutions (concentration greater than 0%) have a higher suppression efficiency than water (concentration at 0%), with 50.85% (95% CI: 49.04%–52.66%), 50.76% (95% CI: 48.78%–52.74%), 63.09% (95% CI: 62.00%–64.18%) and 54.94% (95% CI: 53.77%–56.11%) at the SDBS concentration of 0.05%, 0.1%, 0.2%, and 0.3%, respectively. It is noted that the 0.05% SDBS has the similar coal dust suppression efficiency as that of 0.1% SDBS. The 0.2% SDBS presents the highest efficiency among the 5 concentration solutions, which is 16.01% (95% CI: 14.28%–17.74%), 12.23% (95% CI: 10.14%–14.32%), 12.34% (95% CI: 9.84%–14.84%) and 8.15% (95% CI: 6.48%–9.83%) higher than that of water, 0.05% SDBS, 0.1% SDBS and 0.3% SDBS, respectively. According to the results, it is believed that 0.20% may be the optimum concentration for SDBS to capture the test coal dust particles.

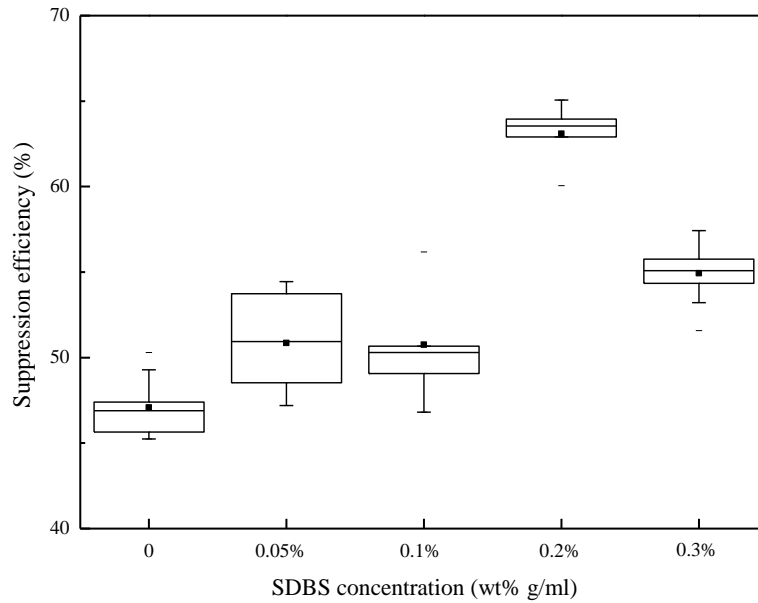


Figure 77. Coal dust suppression efficiency by using SDBS at various concentrations

Table 20. ANOVA for the suppression efficiency of SDBS with different concentrations

<i>Source of Variation</i>	<i>SS</i>	<i>df</i>	<i>MS</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>
Between Groups	0.1496	4	0.0374	86.5210	1.55E-20	2.5787
Within Groups	0.0194	45	0.0004			
Total	0.1690	49				

### 7.4.3 Effect of surfactant on suppression efficiency of different coal particle size

The previous study has shown that the small particles were harder to be captured by the surfactant solutions (Chen et al., 2019), thus it is important to understand the SDBS solution suppression efficiency on different sizes of particles, especially for the finer particles, compared to water. In this study, only the mass concentration of coal dust in the 0~10  $\mu\text{m}$  size range was monitored. It needs to be clarified that the respirable coal dust is referred to the dust that with size less than 10  $\mu\text{m}$  (G. Xu, Chen, et al., 2018), while the TSI DustTrak™ II Handheld Aerosol Monitor used in this study defines the respirable dust as the particles' size less than 4  $\mu\text{m}$ .

The ANOVA for water and 0.20% SDBS is presented in Table 21 and Table 22, respectively. It can be seen that both the p values less than 0.5%, which confirms that the coal dust particles size plays a key role in suppression efficiency for both water and 0.2% SDBS. Figure 78 presents boxplot for the water and 0.20% SDBS surfactant solution suppression efficiency on four sizes of coal dust particles. As expected, the 0.20% SDBS give a higher suppression

efficiency on four sizes of coal particles than that of the water. The mean suppression efficiency on PM1, PM 2.5 and respirable particles are similar for both water and 0.2 SDBS%. Compared to the larger size coal particles, the smaller ones are more difficult to be captured both by using water and 0.20% SDBS. The suppression efficiencies for PM 10 are 47.07% (95% CI: 45.91%–48.23%) and 62.95% (95% CI: 61.93%–63.97%), respectively, by using water and 0.20% SDBS, while only 36.24% (95% CI: 33.10%–39.38%) and 52.98% (95% CI: 51.35%–54.61%) are observed for PM1. There are two reasons that may lead to this result. The first one is that the wettability of smaller coal particles are worse than larger ones, which means the finer coal particles are more difficult to be wetted (Chen et al., 2019; G. Xu, Chen, et al., 2018). The second reason is the collision probability of coal dust particles and water droplets. If the sizes of the water droplets are much greater than the coal particles, the coal particles would have enough time to follow the water streamlines and result in a lower collision probability. The higher collision probability can be achieved when the sizes of water droplets and coal particles are similar (T. Ren et al., 2014). In this study, it is believed that compare to the finer coal particles such as PM1, PM2.5 and respirable dust (PM4), the sizes PM10 may be more close to the water droplets. Thus, a lower suppression efficiency was observed in the finer coal particles. In the further study, a nozzle which is able to spray smaller droplets will be used to better capture the finer particles.

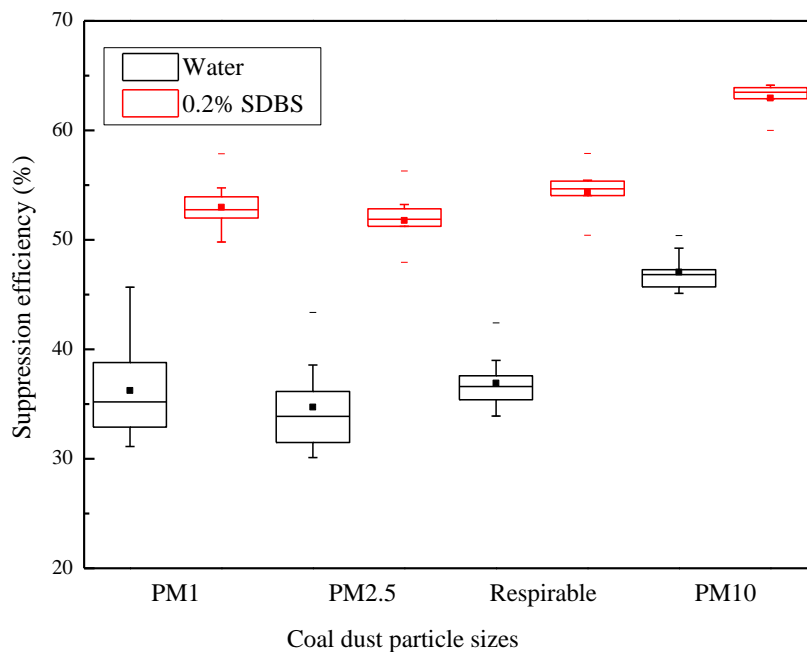


Figure 78. Coal dust suppression efficiency of different coal particle sizes by using water and 0.20% SDBS

Table 21. ANOVA for the 0.20% SDBS suppression efficiency on different coal particle sizes



<i>Source of Variation</i>	<i>SS</i>	<i>df</i>	<i>MS</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>
Between Groups	0.0772	3	0.0257	60.154	4.25E-14	2.8662
Within Groups	0.0154	36	0.0004			
Total	0.0926	39				

Table 22. ANOVA for water suppression efficiency on different coal particle sizes r

<i>Source of Variation</i>	<i>SS</i>	<i>df</i>	<i>MS</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>
Between Groups	0.0950	3	0.0317	28.9492	1.05E-09	2.8663
Within Groups	0.0394	36	0.0011			
Total	0.1344	39				

## 7.5 Conclusions

A CFD modelling was conducted to study the coal dust distribution characteristics and determine the parameters which were needed for the laboratorial experiments. The wind tunnel tests were then designed according to the CFD simulation results to evaluate the effectiveness of surfactants on coal dust suppression.

The CFD simulation results revealed that firstly, the coal dusts distribute relatively evenly at 1.5 m from the inlet, thus, the nozzle was designed to install at this location to obtain a better dust capture performance. Secondly, the stable state of the coal dust mass concentration was achieved after 40 s from the start of feeding the coal dust particles. Thus, the actual waster spray time was set as 2 minutes after the coal particles were introduced.

The wind tunnel tests indicated the water suppression efficiency on coal dust is 47.08% (95% CI: 45.92%–48.24%) with the coal dust particles size less than 10  $\mu\text{m}$ . Among the three types of solution, anionic (SDBS) surfactant performed the highest suppression efficiency, with 63.09% (95% CI: 62.00%–64.18%). Whilst the cationic (CTAB) surfactant failed to enhance the efficiency, with the efficiency of 40.93% (95% CI: 38.65%–43.21%), which is lower than that of water. Also, surfactant concentration plays an important role in coal dust suppression. For SDBS, the concentration of 0.20% is considered as the optimum one for capturing coal dust particles as the highest suppression efficiency is observed at this concentration. For the different sizes of particles, a lower suppression efficiency is found for the finer coal particles according to the experiments.

Overall, the wind tunnel test is the direct method to evaluate the surfactant effectiveness on coal dust suppression. The CFD simulations play a key role in this study, which provides optimized parameters for the experiments and save more time on the designing of the

experiments. The experimental results also demonstrate the potential of the usage of the 0.2% SDBS in the underground coal mines for the coal dust suppression purpose.

## **8 Chapter 8**

### **8.1 Conclusions**

### **8.2 Limitations and future work**

This chapter refers as a conclusion section that highlighted the achievements and summarised the main research findings. Also, the limitations of this research and the recommendations are stated to guide the future work

## **8.1 Conclusions**

A literature review paper and several journal papers that were presented in this thesis to demonstrate the impact of surfactants on suppressing coal dust by using water spray. Both experimental tests and numerical simulation were used to evaluate the effectiveness on coal dust suppression. In summary, the conclusion of this thesis to the current state of knowledge are as follows:

Previous studies have been comprehensively reviewed and the results were concluded in the literature review paper. Both static and dynamic tests can be applied to access the surfactant, and the static test were considered as more cost-effective and easier operational methods by evaluating the coal wettability and surfactant adsorption. While the dynamic test was believed as more reliable as it calculated the coal dust concentration change before and after the surfactant application. Both surfactant itself and coal dust properties contributed to the efficiency of coal dust suppression, and other factors also can affect it in a positive or negative way.

Combined the results in the literature review and the experimental tests results, it can be mainly concluded as below: (1) Comparing the four static test methods, the sink test was determined as the easiest-operational and most cost-effective one in terms of evaluating surfactant by the coal wettability. (2) The non-ionic surfactant TX100 showed a superior efficiency to improve coal wettability and followed is the efficiency of the anionic surfactant SDBS, and the cationic surfactant CTAB might be the worst surfactant candidate. (3) Increasing surfactant concentration helped to improve the coal dust efficiency but keep lifting the concentration over a certain value failed to obtain a further improvement. (4) The synergist effect varies with different surfactant and different salts selected, overall, the greatest synergist effect was achieved for anionic surfactant SDBS. (5) The higher rank coal dust particles indicated poor wettability in both SDBS and TX100 solution, but the lignite adsorbed more SDBS and less TX100 than subbituminous and anthracite at all the tested concentrations. (6) The finer coal dust particles were considered as more difficult to be suppressed as more sink time required, more surfactant adsorption amount observed compared with larger ones. (7) The dynamic

method of wind tunnel test and the optimized static method of sink test obtained some consistent results in terms of selecting the optimum surfactant type and concentration.

The research demonstrated the impact of surfactants on coal dust suppression and the methods that employed to evaluate surfactants. The findings in this research can potentially help to provide a guidance for the mining industry when making strategies on coal dust suppression. Also, the results in this study could be used as references for the further investigation on coal dust suppression.

## **8.2 Limitations and future work**

Although a comprehensive study on factors affect the coal dust suppression by using surfactants was conducted and conclusions described above provided a significant guidance for coal dust control in the mining industry, the limitations still need to be emphasized and the recommendations for the future work are described as follows:

The first limitation is to obtain 0~10  $\mu\text{m}$  coal dust sample due to the dry sieve equipment limitation and the aggregation of fine coal dust particles. The particle size of coal dust plays a major role in determining the surfactant efficiency of suppressing coal dust. In this study, four size ranges of coal dust including 0~38, 38~53, 53~79 and 0~106  $\mu\text{m}$  were selected for the study of coal wettability and adsorption by the static experiments. However, it is believed that respirable coal dust ranges from 0 to 10  $\mu\text{m}$  is the most challenging particles to be captured by spraying surfactant solution. Although the wind tunnel experiment in this thesis demonstrated a lower suppression efficiency was observed for finer coal dust when the particle size is less than 10  $\mu\text{m}$ , a further study on 0~10  $\mu\text{m}$  coal dust wettability and adsorption in presence of surfactants by employing the static methods is still recommended.

The quantity of surfactant used for each surfactant type is considered as the second limitation for this study. Due to the research funding and research time limitation, only four surfactants including two anionic surfactants (SDBS and SDS), one cationic surfactant (CTAB) and one non-ionic surfactant (TX100) were selected in this study. Although these four surfactants are regarded as the most popular ones used in coal dust suppression research area and their chemical properties supposed to be similar with the same type surfactants, it's still not adequate to represent the whole surfactant type by one or two surfactants. To achieve a more persuasive result on the impact of surfactant type on coal dust suppression efficiency, a larger quantity of surfactants for each surfactant type are recommended to be used in the further research.

Thirdly, understanding the interaction between coal dust particles and surfactant solution droplets from microscopic perspective is essential for understanding the underlying mechanisms of using surfactants solution to suppress coal dust. To help quantify the effectiveness of coal dust suppression by surfactants in a microscopic way, a further microscopic study on collision, attachment and stability processes between coal dust particles and surfactant solution droplets is recommended.

Lastly, this research mainly focus on adopting the static methods to evaluate surfactants because they are easier to control variables and more cost-effective, but they not reflect the coal dust suppression efficiency directly as described in Chapter 2. The dynamic methods are more reliable due to the coal dust suppression efficiency can be calculated directly although they are affected by operating errors greatly. However, the disadvantages of high expense and time-consuming results in only the wind tunnel experiments were conducted in this study. To help establishing the relationship between the static methods and the field test method, the application of laboratory results to proper coal mine sites with well controlled onsite conditions are strongly recommended in the further study.

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Appendix

Declaration of Authorship

I, Jinping Chen, contributed:

- 85% of the total work in the paper entitled “*Evaluation of SDBS surfactant on coal wetting performance with static methods: preliminary laboratory tests*”, including experiment design, experiment conduction, authoring and revising the paper. Prof. Guang Xu and Dr. Boris Albijanic provided valuable comments and revised the paper.
- 80% of the total work in the paper entitled “*Surfactant-aided coal dust suppression: A review of evaluation methods and influencing factors*”, including literature review authoring and revising the paper. Prof. Guang Xu and Prof. Jacques Eksteen provided valuable comments and revised the paper.
- 85% of the total work in the paper entitled “*Characterization of coal particles wettability in surfactant solution by using four laboratory static tests*”, including experiment design, experiment conduction, authoring and revising the paper. Prof. Guang Xu and Prof. Jacques Eksteen provided valuable comments, Dr. Jinxing Huang and Mr. Zidong Zhao participated the experiment conduction.
- 75% of the total work in the paper entitled “*Numerical study of coal dust behaviours and experimental investigation on coal dust suppression efficiency of surfactant solution by using wind tunnel tests. Energy Sources*”, including experiment design, experiment conduction, numerical simulation, authoring and revising the paper. Dr. Ping Chang participated the numerical simulation study and provided valuable comments on revised the paper. Prof. Guang Xu supervised this study, Dr. Jinxing Huang participated the experiment conduction, Mr. Ghosh, A and Prof. Wei Liu proved valuable comments on the paper.
- 85% of the total work in the conference paper entitled “*Application of Box-Wilson experimental design method for underground mine’s respiratory coal dust studies*”, including experiment design, authoring and revising the paper. Prof. Guang Xu supervised the study, Dr. Boris Albijanic and Dr. Mahinda Kuruppu provided valuable comments on the paper.
- 85% of the total work in the conference paper entitled “*Study on coal wettability for dust suppression in the presence of surfactants by sink test*”, including experiment design, authoring and revising the paper. Prof. Guang Xu and Dr. Boris Albijanic provided valuable comments and revised the paper, and Ruoxi Wang participated the experiment conduction.
- 75% of the total work in the paper entitled “*Characterized of coal wettability in presence of surfactants and electrolytes additions*”, including experiment design, experiment conduction, numerical simulation, authoring and revising the paper. Prof. Guang Xu supervised the study and provided valuable comments on the paper.
- 75% of the total work in the paper entitled “*Research on three type surfactants adsorption performance on different rank coal particles*”, including experiment design, experiment conduction, numerical simulation, authoring and revising the paper. Prof. Guang Xu supervised the study and provided valuable comments on the paper.