

**Curtin Engineering Faculty  
Department of Civil Engineering**

**The Properties of Geopolymer Concrete Incorporating Red Sand  
as Fine Aggregate**

**Kaveh Soltaninaveh**

**This thesis is presented for the Degree of  
Master of Engineering (Civil Engineering)  
of  
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## **DECLARATION**

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

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.

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*to Mahta...*

## **ABSTRACT**

Concrete is the most common building material in the world and its use has been increasing during the last century as the need for construction projects has escalated. Traditionally, concrete uses Ordinary Portland Cement (OPC) as binder, water as the activator of cement and aggregate.

Finding an appropriate replacement for traditional concrete is a desirable solution to obviate the environmental problems caused by cement production. The use of fly ash as a partial replacement for Portland cement is a method to maintain the properties of concrete and reduce the need for cement. Fly ash is a by-product from coal-fired power plants and is abundantly available. The percentage of cement replacement can be varied according to application and mix design.

One of the potential materials to substitute for conventional concrete is geopolymer concrete (introduced by Davidovits in 1979). Geopolymer concrete is an inorganic alumino-silicate polymer synthesized from predominantly silicon, aluminum and by-product materials such as fly ash. Geopolymer properties have been investigated for several years and it is still a major area of interest among researchers and industry partners as it does not contain cement and uses fly ash and alkali liquids as binders to produce a paste to consolidate aggregates.

Furthermore, the aggregate comprises a substantial portion of concrete. Including coarse and fine aggregates it is normally obtained from natural sources. Fine aggregate in Australia is usually mined from sand quarries. As the demand for concrete production increases, more natural sand is needed. The need for fine aggregate should be addressed in an environmentally friendly manner, considering the diminishing sources of natural sand.

Red sand is a by-product generated from the manufacture of alumina from bauxite by the Bayer process. Previous studies on properties of red sand have shown that it has the potential to be used in concrete as a fine aggregate. While the use of red sand in traditional concrete has been investigated by some researchers, no research has been reported regarding the use of this by-product in manufacturing geopolymer concrete.

This research looks into the replacement of natural sand fine aggregates with red sand in geopolymer concrete. Initially, an extensive series of mixtures was prepared and tested. The objective of the research was to identify the salient parameters affecting the properties of geopolymer concrete when natural sand is replaced by red sand. At the next stage, attempts were made to enhance the mechanical and durability features of red sand geopolymer concrete. The final stage consisted of testing red sand geopolymer concrete to find out the various properties of this novel construction material.

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# **1. INTRODUCTION**

## **1.1 Cement and Concrete**

Concrete is the most commonly used construction material, and the demand for it will increase as the demand for infrastructure development increases. Unfortunately, Ordinary Portland Cement (OPC) production depletes significant amounts of natural resources as it is a high energy-intensive construction material to produce, third only after the production of steel and aluminium. Furthermore, natural aggregate constitutes a substantial portion of traditional concrete. The natural source of coarse aggregate is crushed rock; and fine aggregate is naturally extracted from sand quarries.

The production of one tonne of OPC also releases one tonne of carbon dioxide into the atmosphere. The worldwide cement industry is responsible for about 7% (and rising) of the world's total carbon dioxide generation (Mehta 2001).

Apart from environmental issues associated with the concrete industry, traditional concrete is not very durable in harsh environments, such as exposure to freezing weather, sea water or sulphuric soils. Thus, it is essential to find methods to increase the durability of traditional concrete by using appropriate replacements for concrete constituents; e.g. Ordinary Portland Cement (OPC) and aggregate. It is now believed that using more durable and less energy intensive construction materials is inevitable for the construction industry.

## **1.2 Geopolymers**

Geopolymer concrete is emerging as a new environmentally friendly construction material for sustainable development, using fly ash and alkali in place of OPC as the binding agent. The process uses fly ash, itself a by-product from coal-fired power stations, and activates it with highly alkaline liquors to produce a polymeric gel which binds and sets the non-reactive components (coarse and fine aggregates).

The calculated carbon dioxide emission for the production of one tonne of geopolymeric cement is 0.184 tonnes, about 80% lower than the production of OPC. Cost of fly ash-based geopolymer concrete may also be about 10–30% cheaper than that of OPC concrete (Hardjito & Rangan 2005a).

## **1.3 Red Sand**

Red sand is the coarse fraction of bauxite residue when alumina is extracted. Conventionally, this highly alkaline residue is disposed of in large areas.

Reducing the landfill requirement for bauxite residue is a longstanding challenge for the entire alumina industry. Demonstrating an environmentally, economically and socially acceptable use for this by-product in concrete manufacture would diminish its environmental impact by reducing the size of the footprint for bauxite residue impoundment facilities, stabilizing the alkaline red sand by-product in a benign form, and reduce the need of the construction industry to mine the limited resources of natural sand.

Alumina is a white granular material, a little less coarse than table salt, and is scientifically known as aluminum oxide. Aluminum does not occur as a metal, but must first be refined from bauxite in its oxide form. The Bayer refining process used by alumina refineries worldwide involves four steps of digestion, clarification, precipitation and calcination.

Alcoa's alumina refineries in Western Australia process bauxite ore mined in the nearby Darling Range. The refineries extract alumina from the ore and the remaining material, consisting primarily of iron oxides and quartz sand is stored in residue storage areas. The residue has some degree of alkalinity hence it is currently stored to settle down slowly. In order to reduce seepage to ground water, storage areas are lined. Conventionally, the “wet disposal” method is used and water present in the residue is recycled to the refinery.

In 1987 “dry stacking” was developed and adopted. The process produces a thickened residue in layers. The water is then removed as a result of drainage and evaporative drying. Dry stacking is now fully operational at all three of Alcoa's Western Australian refineries, and is now being adopted by other minerals industries as best practice. The advantages include reduced environmental risk, a more stable landform for immediate rehabilitation and a wider range of potential land uses for storage areas in the future (Alcoa World Alumina Australia).

Bauxite residue can be later separated into two fractions, according to size. The coarse fraction or “red sand” has a particle size in excess of 90 $\mu$ m. The other portion, “red mud” constitutes approximately half of the residue.

The bauxite residue fractions can be used in number of applications. Bauxite residue can be used in agriculture, the ceramic industry, urban sewage systems and the plastic industry. Red sand has also been used in concrete, pavements and in construction of dyke walls for red mud impoundments.

However, as huge amounts of bauxite residue need large areas of land for storage and considering the costs associated with monitoring this residue, finding a sustainable use for bauxite residue can be a promising solution to residue disposal management.

Recent studies in Western Australia have shown that Red sand can be used in concrete mix design, as fine aggregate. A study by Sri Wahyuni (2006) showed that concrete containing red sand has similar properties to concrete using natural sand. Jitsangiam, Nikraz & Jamieson (2007) investigated the use of red sand as base course for roads and another recent study showed that red sand can be used in marine grade concrete (Ghiafeh Davoodi, Nikraz & Jamieson 2007a).

The present thesis studies the properties of geopolymer concrete made with red sand. While the previous researchers have studied its influence on the properties of traditional concrete, the use of red sand in geopolymer concrete has not been investigated to date. Geopolymer concrete uses fly ash – itself a by-product of coal-fired power stations – and alkali instead of Ordinary Portland Cement. Red sand geopolymer concrete is a new construction material made from two by-products. It is a “green concrete” as it does not use cement; and by using red sand as a fine aggregate, it becomes a more environmentally friendly material as it decreases the need for extracting natural sand mines.

## **1.4 Aim of the Research**

The aim of this thesis is to evaluate the properties of geopolymer concrete mixtures made with red sand as a fine aggregate. Making workable, high strength and durable geopolymer concrete containing red sand can lead to the usage of bauxite residue in the future and paves the way to gradually replacing conventional concrete with less energy-intensive construction materials. Therefore, identifying, testing and enhancing the mechanical and durability features of red sand geopolymer concrete has been the main objective of this research.

## **1.5 Significance**

This project aims to reduce the impact of bauxite tailings on the environment, through investigating potential industrial applications for this by-product as a construction material. Successfully demonstrating that red sand geopolymer concrete meets the relevant Australian Standards will effectively convert a by-product into a valuable construction material. This would significantly reduce the amount of bauxite residue that requires disposal, storing and monitoring.

Furthermore, with the ever increasing demand for quality construction materials, materials such as natural sand are becoming scarce. If red sand can be shown to be an acceptable substitute for natural sand, it will provide valuable new material for the construction industry; and will improve the sustainability of construction operations by reducing the need to mine a natural resource. Using geopolymer concrete will also decrease the need for making traditional concrete that uses cement, which plays a key role in releasing carbon dioxide emissions to the atmosphere.

## **1.6 Organization of the Thesis**

This thesis is divided into eight chapters. Chapter 1 contains the introduction. Chapter 2 reviews previous studies and the literature related to geopolymers, geopolymer concrete and bauxite residue. Chapter 3 outlines the experimental program that has been set up for this project. Chapter 4 discusses the experimental methodology adopted to evaluate various properties of geopolymer concrete mixtures. Chapter 5 discusses the “rapid hardening” phenomenon experienced throughout the project. The results and discussion are presented in Chapter 6. Chapter 7 and Chapter 8 contain the conclusions and recommendations for future studies respectively.

## 2. LITERATURE REVIEW

This chapter presents the background to the fly ash usage in concrete as a replacement for Portland cement and also the development of geopolymers and geopolymer concrete. The production, usage and properties of bauxite residue present in available literature are also reviewed.

### 2.1 Fly Ash

#### 2.1.1 Definition

Fly ash is a by-product from the combustion of pulverized coal which is widely used as pozzolanic ingredient in hydraulic cement concrete, High Volume Fly Ash concrete (HVFA) and geopolymers.

American Concrete Institute, ACI 116R, defines fly ash as” *the finely divided residue that results from the combustion of ground or powdered coal and that is transported by flue gases from the combustion zone to the particle removal system.*” (ACI Committee 232 2004) The source material affects the characteristics of fly ash such as chemical composition and strength enhancement. Other variables which have an effect on chemical composition can be the grinding mill efficiency, the combustion environment, the boiler configuration and the rate of cooling.

The fly ash particles are spherical and have high fineness. The majority of particles are smaller than 100  $\mu\text{m}$ . According to ASTM C 618, fly ash is categorized by chemical composition and source material. Class F fly ash is produced from anthracite or bituminous coal which contains less than 15% calcium oxide. Compared to Class F, Class C fly ash normally comprises more than 20% calcium oxide and is generated from lignite or Sub-bituminous. Another aspect is pozzolanic effect. A pozzolan is a siliceous or siliceous and aluminous material that has little or no cementitious effect but, when finely

divided and in the presence of moisture, will react with calcium hydroxide to form compounds with cementitious properties. Class F fly ash possesses pozzolanic effect whereas Class C fly ash shows both pozzolanic and cementitious features.

The fineness of fly ash is an essential factor for pozzolanic reaction to take place. The amorphous silica, which is present in a glassy form, reacts with calcium hydroxide to form additional C-S-H bonds. The inclusion of fly ash can improve the workability of fresh concrete and as a consequence, decrease the required amount of water. This could be attributed to the spherical shape of fly ash particles. Also, small particles of fly ash can fill voids and compensate deficiency in aggregate grading. Researchers recognize other beneficial properties of fly ash such as enhancing the concrete's resistance to deterioration from sulfates, improving the ability of concrete in preventing the alkali-aggregate reactions and the reduction in the temperature of hydration. The latter is particularly useful in mass concreting in the construction of large concrete dams (ACI Committee 232 2004)

### **2.1.2 Fly Ash Composition**

The principal constituents of fly ash are SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and CaO. According to ASTM C 618, if the sum of the first three constituents exceeds 70%, the fly ash is classified as Class F whereas this limit is only 50% in the case of Class C. Other constituents of fly ash, present in lower percentages, are MgO, SO<sub>3</sub> and carbon. Fewer amounts of other elements may be present such as titanium, phosphorous and lead. The loss on ignition or (LOI) is an indicator of carbon content which is a result of incomplete combustion of the coal. Class C fly ashes usually have LOI less than 1% but class F fly ashes range from this low level to values as high as 20%; however, ASTM C 618 allows class F fly ash to be used with up to 12% LOI if acceptable

performance records or laboratory test results are made available (ACI Committee 232 2004)

### **2.1.3 The Use of Fly Ash in Concrete**

Initially fly ash was used as a partial replacement of hydraulic cement in the construction of large dams, in US, for example, the Hungry Horse Dam was one of the first applications. Using more than 120,000 tons of fly ash, the project was completed in 1953. From that time until 1970 more than 100 major locks and dams were constructed in the US (ACI Committee 232 2004).

In Australia, for the first time, fly ash was used to build a monolith crest section of a dam in Sydney in 1954. Initially, the percentage of fly ash used in place of cement was generally less than 20% of total mass of binder. As the more favorable properties of fly ash were recognized, higher percentages were incorporated in conventional concrete. The term ‘High volume fly ash concrete (HVFA)’ refers to concrete containing fly ash more than 40% weight of cementitious material. The development of HVFA started in Australia after 1991 following investigations in other countries like Canada and the US (Sirivivatnanon et al. 1995). In Perth, Western Australia, concrete containing 50% fly ash concrete was reported (Ryan and Potter) to have been used for the construction of the Roe Street Tunnel (cited by (Sirivivatnanon et al. 1995)).

## **2.2 Geopolymers**

### **2.2.1 Structure**

In 1979, Davidovits used the term “Geopolymer” for the first time to describe the inorganic polymer binders resulting by geochemical processes (Davidovits 1999a). Geopolymerisation involves a chemical reaction in which Si-O-Al-O bonds are formed as a result of the reaction between an alkaline and a source

of Alumina-Silicate oxides. Geopolymer compositions are similar to natural Zeolites; however, their structures are amorphous to semi-crystalline. This is due to the faster reaction time of geopolymers compared with Zeolites that yield crystalline structures .

The geopolymers, like other polymers, undergo transformation and polycondensation and take shape at low temperatures; but they are also "geopolymers"; thus they are mineral materials which are hard, weather resistant and can stand high temperature (Davidovits 1988).

The polymerization process can be indicated by the following formula (Davidovits 1999a):



Where, "z" is 1, 2 or 3; M is a cation such as potassium or sodium and "n" is a degree of polycondensation.

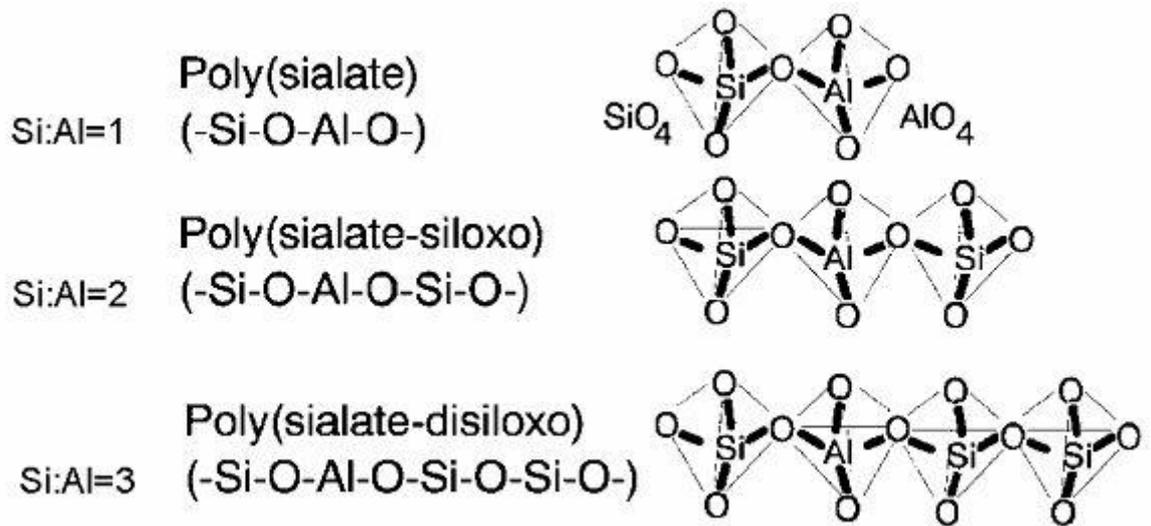
Regarding the Si:Al ratio, geopolymers structures may consist of three of the following forms:

[-Si-O-Al-O-] bonds or Poly (sialate)

[-Si-O-Al-O-Si-O-] bonds or Poly (sialate-siloxo)

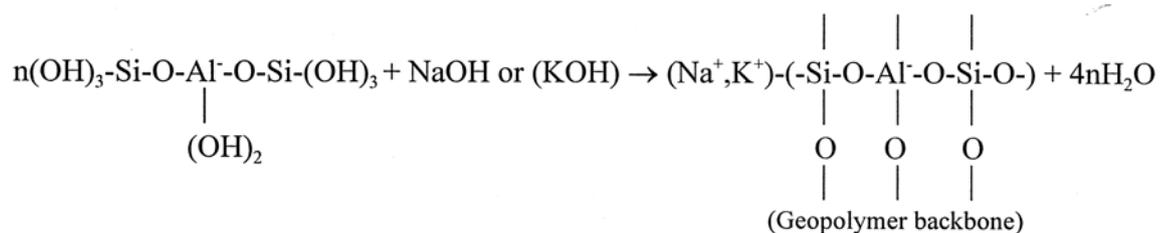
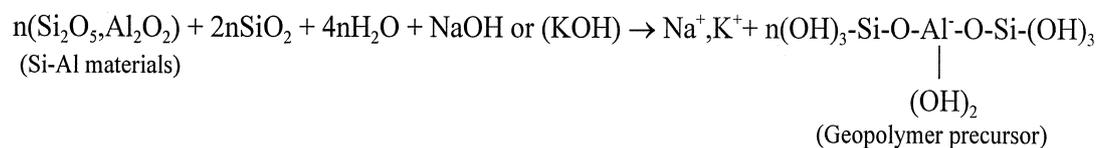
[-Si-O-Al-O-Si-O-Si-O-] bonds or Poly (sialate-disiloxo)

Or schematically,



Sialate is an abbreviation for silicon-oxo-aluminate. The sialate network consists of SiO<sub>4</sub> and AlO<sub>4</sub> linked alternately by sharing oxygen. Positive ions such as Na<sup>+</sup> or K<sup>+</sup> must be present in the framework to balance the negative charge of Al<sup>3+</sup> in IV-fold coordination.

The main steps involved in the formation of geopolymers include the following two equations (Davidovits 1999a; van Jaarsveld, van Deventer & Lorenzen 1997):



Although the exact process of geopolymerisation is not completely understood, it was proposed that the process includes dissolution, transportation or orientation, as well as a re-precipitation (polycondensation)(Davidovits 1988).

An alkali metal or hydroxide is required for the dissolution of silica and alumina. The coordination of alumina in the source material has an effect on the bonding strength of the matrix. While silicon is always 4 coordinated, aluminum ions can be 4 or 6 coordinated. An intermediate gel is formed via the co-polymerization of alumina and silica species; however little is known about the exact nature of this gel.

### **2.2.2 Source Material**

Researchers have used different source materials for developing geopolymer binders. These include natural minerals such as kaolinite, albite, feldspar and stilbite; treated minerals such as metakaolin; and by-products such as blast furnace slag and fly ash. Theoretically, any source of amorphous silica and aluminum could be used to manufacture geopolymer binders (Hardjito & Rangan 2005b). Davidovits (1999a) used Metakaolin as a source of silica and aluminum to produce geopolymer pastes. Metakaolin is obtained by calcining kaolin clay at 750 °C for 6 hours. Other researchers have also used Metakaolin (Barbosa & MacKenzie 2003; P. Duxson et al. 2007a; Fernandez-Jimenez et al. 2008). Hua Xu and van Deventer (2000) examined 15 natural Al-Si minerals to study the effect of mineral properties on the compressive strength of the synthesized geopolymer.

The use of a combination of two source materials was also reported such as ground granulated blast-furnace slag and fly ash (Sofi et al. 2007), kaolonite and stilbite (Xu & van Deventer 2002b), Class C fly ash and granulated blast-furnace slag (Goretta et al. 2004), Metakaolin and class F fly ash (Fernandez-Jimenez et al. 2008) and metakaolin and calcium hydroxide (Alonso & Palomo 2001).

The use of fly ash in geopolymer concrete has been looked at as a promising development in recent years and has been studied by numerous researchers as the sole source of silica-aluminum. Fly ash is a by-product which is generated by the combustion of coal and needs no more process in a laboratory, whereas, for the production of Metakaolin, the kaolin clay must be heated up to 700-800 °C, a highly energy consuming process. According to Duxon et al. (2007b):

*The use of metakaolin in geopolymers would also increase the CO<sub>2</sub> emissions per tonne of product; however, the high cost of metakaolin and the high water demand of metakaolin geopolymers mean that this is not considered a viable possibility for large-scale geopolymer production in construction applications.*

Thus, the utilization of fly ash, as a raw material in the synthesis of geopolymeric materials, has been the subject of numerous studies. Van Jaarsveld, Van Deventer and Lukey (2003) used six types of fly ashes from different sources to investigate the characterization of source materials in fly ash-based geopolymers. Alvarez-Ayuso et al. utilized four types of fly ash, derived from different power plants, in order to investigate the environmental, physical and structural characteristics of geopolymer matrices. Fernandez-Jimenez et al. (2008) used metakaolin and fly ash to examine the variation in mineralogical and microstructural characteristics of the alkaline inorganic polymers obtained by alkaline activation. Using three source materials, Hua Xu and van Deventer (2002a) concluded that substantial reduction in reaction

time may be achieved along with a significant improvement in compressive strength when a calcined source material, such as fly ash, is added to the geopolymerisation of non-calcined materials such as kaolinite and albite.

In recent years, a substantial series of studies of fly ash-based geopolymer concrete has been carried out at Curtin University of Technology. Hardjito and Rangan (2005a) conducted experimental work to study the technology of making geopolymer concrete and the role of salient parameters on the strength and mechanical properties of fly ash-based geopolymer concrete. Long term properties of fly ash-based geopolymer concrete were investigated by Wallah and Rangan (2006) and the behavior of structural members like beams and columns made of fly ash-based geopolymer concrete was looked into by Sumajouw and Rangan (2006).

### **2.2.3 Alkaline Liquids**

It has been shown that alkali materials must be present to react with aluminum and silica in source materials to form geopolymeric gel. Different alkali liquids have been used by researchers to investigate the effect of alkali material on formation, strength and durability of geopolymers. Davidovits (1999a) used sodium hydroxide solution to react with kaolinite. Duxson et al. (2007a) studied the effect of alkali type on the development of mechanical properties of geopolymer. In their work, they used sodium and potassium hydroxide, and sodium silicate to activate metakaolin. The use of sodium hydroxide and potassium hydroxide has been reported by many researchers. Barbosa and MacKenzie (2003) reported the use of potassium hydroxide and metakaolin as the starting material to study the thermal stability of geopolymers. Testing 16 natural Al–Si minerals, Hua Xu and van Deventer (2000) concluded that the rate of dissolution of minerals is higher in NaOH than KOH. Other researchers used a mixture of sodium silicate and sodium

hydroxide to activate a combination of fly ash with metakaolin (Fernandez-Jimenez et al. 2008) or kaolinite (Swanepoel & Strydom 2002)

#### **2.2.4 Properties of Geopolymers**

**Acid Resistance** Compared to Portland cement binders, geopolymers are much more resistant when exposed to acid environments. Portland cement binders rely on lime and thus, are vulnerable to deterioration caused by acid environments (Davidovits 2002).

**Compressive strength** It has been shown that geopolymer concrete can be made employing the similar methods used for making conventional concrete (Hardjito & Rangan 2005a).

**Alkali-aggregate reaction** Under specific circumstances, alkalis present in ordinary Portland cement can react with certain forms of reactive silica in aggregates and cause deleterious alkali-aggregate reaction. Alkali-aggregate reaction results in expansion which can damage the concrete structures. As a consequence, the tendency has been to avoid using susceptible aggregate or high alkali cements in concrete. It is now known that using fly ash as a partial replacement for ordinary Portland cement can control the expansion caused by alkali-aggregate reaction. The effect of fly ash in preventing the expansion can be attributed to a number of factors, like the reduced alkalinity in pore solution of concrete or the lower calcium content in the system.

Although reaction can also take place in geopolymer matrix, the proneness of geopolymer concrete to deterioration caused by alkali-aggregate reaction is notably less (Garcia-Lodeiro, Palomo & Fernandez-Jimenez 2007). Other researchers have also studied the alkali-aggregate reaction in alkali-activated granulated slag and reported the slower rate of expansion in comparison with

Ordinary Portland Cement (OPC) mortars (Fernandez-Jimenez & Puertas 2002).

***Toxic waste management*** The contaminant migration derived from toxic waste materials can be inhibited by physical and geochemical barriers. The durability of the matrix is a key feature in determining the suitability of the barrier. Geopolymers have been shown to possess great potential to immobilize toxic wastes as well as to convert the semi-solid wastes to adhesive solid materials (Davidovits 1988). Furthermore, geopolymers, as durable compounds, encapsulate solidified waste and prevent the leaching of hazardous material into the environment.

***Geopolymeric cements and concretes*** Aside from the promising environmental benefits that geopolymer binders present, the good mechanical and durability features of geopolymer cements and concretes make them interesting construction materials. Geopolymer concrete mixtures with 7th day compressive strength over 70 MPA can be made employing the conventional method used for the production of ordinary concrete. Regarding durability features, geopolymer concrete exhibits low drying shrinkage and shows good resistance to acid and sulfate environments (Hardjito & Rangan 2005a)

***Fire-proof geopolymeric gements*** The resistance of concrete members to high temperature has been investigated for decades. It is always desirable to have fire-proof structural members that can withstand heat for a long time hence allowing residents the chance to escape. While conventional concrete may explode above 300 °C , geopolymer cements can be fire-resistant up to 1200 °c (Davidovits 1999b).

## **2.3 Red Sand**

### **2.3.1 Bauxite Residue**

As the world largest producer of alumina, Australia generates large amounts of bauxite residue each year. Approximately 15 million tones of bauxite residue are produced by three refineries in Western Australia. Red sand- the coarse fraction of this residue- makes up to one- half of the bauxite residue.

Alcoa World Alumina Australia (ALCOA) operates in Victoria, New South Wales and Western Australia and produces about 47% of Australia's alumina. Alcoa extracts alumina from bauxite ore at three refineries in Western Australia located at Kwinana, Pinjarra and Wajerup. They produce about 8 million tonnes of alumina each year which is 13% of world production (Alcoa World Alumina Australia (ALCOA)).

Aluminum is produced from alumina. Alumina is in turn produced from bauxite ore using the Bayer process, the widely used method for extraction of alumina. Initially developed by Karl Bayer in 1888, it consists of refining bauxite to extract alumina as a precursor to aluminum.

The Bayer process mainly comprises the digestion of bauxite in a sodium hydroxide solution. As a result, the aluminum content of bauxite ore is dissolved and a residue is generated, a highly alkaline slurry (pH 10-12.5) that exits the process. Bauxite residue is mainly composed of silica, aluminum, iron, calcium and titanium oxides and hydroxides. The iron present in the residue is responsible for the red color (Hind, Bhargava & Grocott 1999).

Disposal of this huge amount of residue requires vast areas of land. The disposal process should address environmental issues considering the fine

particles of red sand and prevent the residue from contaminating the ground water and soil. The other challenges are:

- The high alkalinity of the generated residue
- The costs associated with monitoring and storage

Attempts have been made for disposing bauxite residue in an environmentally friendly manner. One of the conventional methods is using bauxite residue in the construction of clay-lined dams or dykes. The residue is pumped into the dam and allowed to dry naturally (Hind, Bhargava & Grocott 1999).

Generally, bauxite residue can be disposed in two ways: wet disposal, which is the conventional method; and “dry stacking” which was adopted by Alcoa in 1985 for refineries in Western Australia:

Dry stacking utilizes a large diameter gravity thickener, called a superthickener, to de-water the fine tailings and produce thickened slurry. This slurry is then spread in layers over the storage areas to de-water by a combination of drainage and evaporative drying (Cooling 2007).

Figure 2-1 shows spreading and ploughing of bauxite residue in Alcoa’s Western Australian refineries (photo from Cooling (2007)).



Figure 2.1: Spreading the slurry to enhance the drying rate

Dry stacking bauxite residue is now conducted in all refineries in Western Australia as it has been shown to be environmentally friendly and cost effective way to dispose bauxite residue.

### **2.3.2 Applications of Bauxite Residue**

Although recovering alumina and soda from bauxite residue is possible, the costs are not justified. Generally, recovering the constituents of bauxite residue has been found to be costly and non-feasible. Another application of bauxite residue is in the ceramics industry. As a clay-like material, red mud can be used to make ceramics when fired. Bauxite residue has also been in use in agriculture. Because of its high alkalinity, it has been used in acidic soils and also as a treatment for iron deficient soils (Hind, Bhargava & Grocott 1999). It has also been used as a filtration medium to remove phosphorous and nitrogen from sewage effluent in domestic and industrial sewage systems. In the plastics industry, it is used as filler for plastics, to impart strength, resistance to UV, heat and chemicals. Other applications of bauxite residue are listed below (Cooling 2007):

- Used as road base, either the sand fraction directly, or the mud as a component of a composite with gypsum or fly ash.
- Used as raw materials for the production of cement alternatives, such as mineral polymers and ceramics.
- Used as a pigment for a range of applications in coatings and materials manufacture.

### 2.3.3 Red Sand and Red Mud

Bauxite residue can be separated into two fractions: the coarse fraction or “Red Sand” which has a particle size in excess of 90  $\mu\text{m}$  and the fine fraction called “Red Mud”.

Typically, red sand constitutes half of the bauxite residue. Based on bauxite composition, 1-2.5 tonne of residue is generated for each tonne of alumina. In Western Australia, three refineries generate 20,000 tonnes of Red Sand each day (Jamieson et al. 2006). Table 2.1 shows the constituents of the coarse and fine fraction of bauxite residue (Sri Wahyuni et al.).

Chemical Composition	Red Mud	Washed and Carbonated Red Sand
SiO <sub>2</sub> (%)	40.70	19-44
Fe <sub>2</sub> O <sub>3</sub> (%)	30.80	11-45
Al <sub>2</sub> O <sub>3</sub> (%)	19.50	11-26
TiO <sub>2</sub> (%)	2.97	1-4
CaO (%)	4.40	2-8
Na <sub>2</sub> O (%)	3.00	1-6

**Table 2.1: Chemical composition of bauxite residue**

Further processing red sand consists of neutralization via carbonation and washing. Washed and carbonated red sand can be further processed by magnetic separation to obtain three different classifications of sand. Magnetic separation creates the opportunity to obtain fractions of red sand with different properties. One fraction is called High Iron red sand which represents about 35% of coarse residue and has an iron content of around 56%. The High Silica Red sand is the non magnetic fraction of red sand is predominantly quartz. High Silica Red Sand has a SiO<sub>2</sub> content of about 90% and takes up to 40% of the coarse residue. There is also a middle fraction which can be separated after initial separation and represents up to 25% of red sand (Jamieson et al. 2006).

In recent years, there have been a number of studies carried out on Red sand utilization. A study in 2005, investigated the utilization of red sand as fine aggregate in traditional concrete (Sri Wahyuni et al.). The mechanical and durability features of concrete including red sand was tested and it was concluded that its inclusion did not affect the strength of concrete; however, the durability tests resulted in higher water permeability and chloride diffusion due to the “porosity” of concrete made with red sand. A key finding of the research was the lower workability of red sand concrete and the consequent high volume of pores. It was reported that “*Red Sand concrete had more pores due to the low, almost zero, slump value*”.

Recent research looked into use of red sand as a road base material in Western Australia. The soil stabilization technique, a pozzolanic-stabilized mixture, was used to improve the properties of red sand to satisfy minimum requirements of road bases (Jitsangiam, Nikraz & Jamieson 2007). The outcome of the study revealed that stabilized red sand is a viable option for use as a base course material in roads.

Research aimed at studying the behavior of concrete made with red sand in marine environment. In the laboratory work, washed and carbonated red and its derivatives i.e. high Iron sand and high silica sand were used along with red mud. The research studied various properties of concrete mixtures made with different types of fine aggregate. It concluded that concrete mixes incorporating red sand and low portions of red mud (10%) are likely to be useful in practice. However, in line with previous studies, the workability of the mixes containing washed and carbonated red sand was reported lower than control mixes using natural sand (Ghiafeh Davoodi, Nikraz & Jamieson 2007b).

### **3. EXPERIMENTAL PROGRAM**

#### **3.1 Experimental Approach**

An extensive series of laboratory tests was conducted throughout the project. At the early stage, a comparative approach was selected to study the properties of geopolymer concrete when red sand replaced natural sand. For this stage, compressive strength was selected as the indicator of mixtures' mechanical properties; afterwards, a trial-and-error method was adapted to identify salient parameters that are effective on mixture workability and strength. The aim was to optimize mixture proportions in terms of mechanical and durability features. Eventually, various properties of geopolymer concrete including red sand were studied. The experimental program used throughout the research is outlined in Figure 3.1 and the details of twelve types of concrete which were made during the laboratory work are given in Table 3.3.

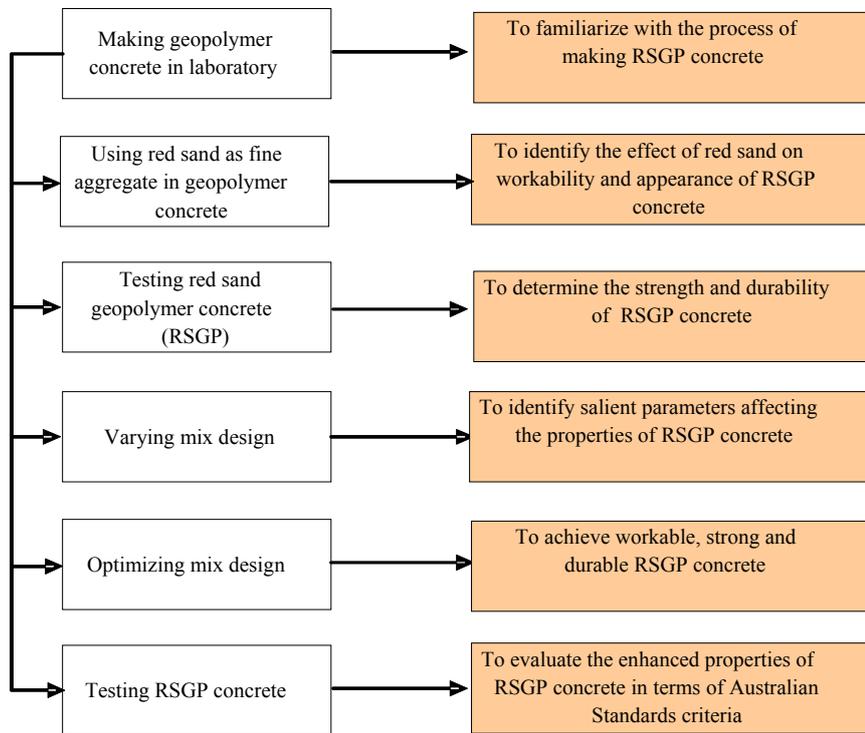


Figure 3.1: Experimental program

## 3.2 Materials

### 3.2.1 Fly Ash

Low Calcium Class F fly ash obtained from Collie power station, Western Australia was used as the sole source of alumina and silica. It was preferred to other types of fly ashes due to its wide use, availability and advantageous features. Authors have mentioned that the calcium content of fly ash affects the workability and setting of the mixture (van Jaarsveld, van Deventer & Lukey 2003). Furthermore, Gourley (2003) (cited by (Hardjito & Rangan 2005a) claimed that the “*high amount of calcium may interfere with the polymerization process and alter the microstructure*”. The X-Ray fluorescence (XRF) revealed the composition of fly ash which was used during the project the data are presented in Table 3.1. The XRF test was carried out at the Centre for Materials Research, Curtin University of Technology by Research Associate, Michael Rutledge.

Oxides	%	Oxides	%
SiO <sub>2</sub>	49.78	SrO	0.30
Al <sub>2</sub> O <sub>3</sub>	26.80	MnO	0.08
Fe <sub>2</sub> O <sub>3</sub>	13.40	ZnO	0.032
CaO	2.20	NiO	0.029
TiO <sub>2</sub>	1.60	V <sub>2</sub> O <sub>5</sub>	0.026
P <sub>2</sub> O <sub>5</sub>	1.56	Cr <sub>2</sub> O <sub>3</sub>	0.024
MgO	1.44	CuO	0.013
K <sub>2</sub> O	0.545	Rb <sub>2</sub> O	0.006
BaO	0.482		
Na <sub>2</sub> O	0.34		
SO <sub>3</sub>	0.30		

**Table 3.1: the chemical composition of fly ash**

As shown in Table 3.1 the ratio of  $\text{SiO}_2$ -to- $\text{Al}_2\text{O}_3$  is 1.86, the CaO comprises 2.2 percent of total mass and the Loss on Ignition is as low as 0.32. The source of fly ash was kept constant during the experimental program to rule out any change in mix properties.

### **3.2.2 Alkaline Liquid**

A combination of sodium hydroxide and sodium silicate solutions was used as the alkaline liquid to activate fly ash.

A sodium hydroxide solution was prepared by dissolving sodium hydroxide pellets in water. The degree of purity of the pellets was 97% and was taken into account to modify the quantities. Distilled water was used to dissolve the pellets to avoid affecting the solution by tap water contaminations.

Adding soluble silica has been shown to have positive effects on the properties and strength of geopolymers. It is also believed that using sodium silicate along with sodium hydroxide, enhances the formation of geopolymers (Xu & van Deventer 2000); therefore, a sodium silicate solution was used with the following composition:  $\text{Na}_2\text{O}$  (14.7%),  $\text{SiO}_2$  (29.4%) and water (55.9%). Sodium silicate was kept as solution in drums and pumped when needed during the project.

### 3.2.3 Aggregates

**Coarse aggregate:** Coarse aggregates used for this project consist of locally supplied granite type coarse aggregate. Although the grading of aggregates varied throughout the program, the coarse aggregate comprised only sizes 7 and 10mm. They were washed prior to use to ensure that dust and organic materials were not present. After that, the aggregate was soaked in water for two days, and placed on plastic sheets to dry naturally to reach the Saturated and Surface-Dried (SSD) condition. Aggregate was then kept in buckets until the day of mixing.



Figure 3.2: Coarse Aggregate

**Fine aggregate:** Natural sand or dune sand fine aggregates were used in this research. The natural sand was obtained from dunes in uncrushed form. Moisture content tests on the sand at SSD condition showed that the percentage of water by mass required to bring the dune sand to the SSD condition was 0.3%. Washed and carbonated red sand was used as the fine aggregate for experimental mixes. In Western Australia, red sand comprises half of the by-product from bauxite residue and can be processed through

standard mineralogical processes (Low Intensity Magnetic Separator– (LIMS) and Wet High Intensity Magnetic Separator – (WHIMS), to produce various fractions that have a higher potential value than the total sand fraction (Jamieson et al. 2006).



Figure 3.3: Fine aggregate

The washed and carbonated red sand used in this study was supplied by Alcoa from Western Australian refineries. Figure 3.3 shows particles of red sand under a Scanning Electron Microscope. Unlike natural sand, red sand comprises crushed particles in a wide range of shapes, angularity and size.

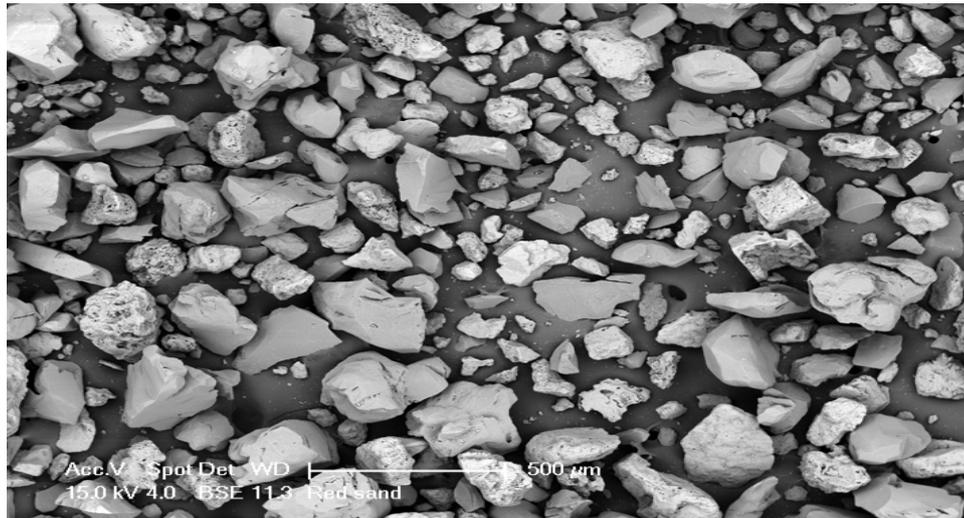


Figure 3.4: Red sand under the Electron Microscope  
(Photo taken by Chris Reid)

### 3.2.4 Superplasticizer

In order to achieve the desired workability, a naphthalene based superplasticizer was used as the water reducer. Previous studies have shown that the workability of fresh geopolymer concrete mixture can be increased by adding superplasticizers (Hardjito & Rangan 2005a).

### 3.2.5 Water

Distilled water was used to make sodium hydroxide solution and to enhance the workability of fresh geopolymer concrete when needed.

### 3.3 Sieve Analysis

To ensure that the grading of the aggregate is kept constant during the project and also to investigate the effect of red sand inclusion on aggregate grading, numerous sieve analysis tests were carried out for various combinations of fine and coarse aggregates.



Figure 3.5: Sieves used for particle distribution test

Since the grading of fine aggregates has a considerable effect on workability, it was desired to conduct a series of tests to find the optimum proportion of used aggregates. Initially, a sieve analysis of all aggregate was carried out. Table 3.2 shows the particle size distribution of coarse and fine aggregates.

<b>Sieve size</b>	<b>10mm coarse aggregate</b>	<b>7mm coarse aggregate</b>	<b>%Natural sand</b>	<b>%Red sand</b>
<b>13.2</b>	100			
<b>9.5</b>	82	100		
<b>6.75</b>	12	76		
<b>4.75</b>	1	21		
<b>2.36</b>	0	4	100	100
<b>1.18</b>	0	3	100	100
<b>0.6</b>	0	2	84	95
<b>0.3</b>	0	2	18	64
<b>0.15</b>	0	1	1	16
<b>0.075</b>	0	0	0	4

**Table 3.2: Particle size distribution of aggregates used in the research**

It can be seen from the table that red sand consists of finer particles compared to natural sand. The difference between the two sands starts at sieve size 0.600mm. This is a well-known trend for natural sands. As Neville (1995) states:” *a large group of natural sands separate themselves at that size*”.

The particle size distribution of aggregates is illustrated in the following chart. A combination of 10mm (30% wt.), 7mm (35% wt.) and fine aggregate (35% wt.) was used.

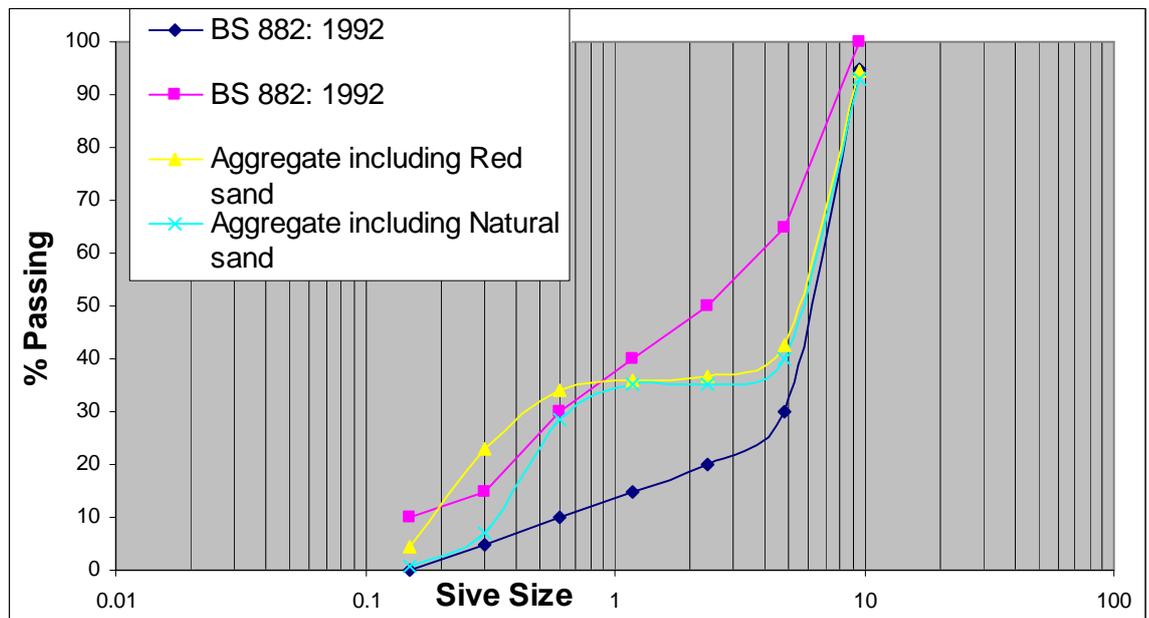


Figure 3.6: Particle size distribution of all-in aggregates

The upper and lower curves show the British Standard limits for “All-in aggregate” used in the mixture. It can be seen from the chart that the combination of a coarse aggregate and natural sand yields a mixture well between the limits. Replacing natural sand with red sand makes grading “finer” but considering a 10% standard deviation, the trend falls within the limits; hence red sand can be an appropriate substitute for natural sand for making concrete (A. S Wahyuni et al. 2006).

Another feature of red sand is high water absorption. This can be attributed to the relatively higher surface area of fine particles. In determining the grading of aggregates, the surface area is a governing factor which determines the water required to wet all the solids. The higher the surface area, the more water is required to wet the surface of fine particles in order to yield the same workability (Neville 1995).

Mixture No.	Aggregate				Fly Ash	NaOH		Sodium silicate	Water	Super plasticizer
	10mm	7mm	Natural sand	Red sand		Quantity	Molarity			
1	548	638	638	0	403	41	14M	103	20.8	6.1
2	548	638	0	638	403	41	14M	103	20.8	6
3	548	638	319	319	403	41	14M	103	20.8	6
4	548	638	160	478	403	41	14M	103	20.8	6
5	548	638	0	638	403	41	14M	103	20.8	12.2
6	548	638	0	638	403	41	14M	103	52	6
7	628	628	0	538	425	44	14M	110	20.8	6.3
8	620	620	0	531	442	46	14M	114	21.4	6.6
9	611	611	0	524	467	48	14M	120	22.7	7
10	548	638	638	0	403	41	8M	103	20.8	6
11	628	628	0	538	425	44	8M	109	20.8	6.3
12	620	620	0	531	442	46	8M	114	21.4	6.6

Table 3.3: Details of mixtures

## **4. EXPERIMENTAL METHODOLOGY**

### **4.1 The Moisture Content of Aggregates**

The moisture content of an aggregate plays a significant role in fresh concrete characteristics. A dry aggregate tends to absorb moisture from the mixture, leaving cementitious materials without sufficient water for the hydration process. On the other hand, an uncontrolled “wet” aggregate will introduce extra moisture to the mixture, causing higher water-to-cement ratio and consequently less strength. The saturated-and-surface-dried condition is referred to as the desirable condition on which aggregate neither gives moisture to the mixture, nor absorbs water from it.

In geopolymer concrete, as shown by Hardjito and Rangan (2005a), extra water substantially reduces the ultimate strength. In this particular project, it was important to measure the water absorption values for natural sand and red sand.

Measuring the water absorption was done by determining the weight of sand before and after saturation. The test was conducted according to AS 1141.5-2000. Initially, a 1kg sample of sand was taken. It was immersed in water for more than 24 hours. After draining the water off, it was placed on a flat surface under warm air generated by a small fan heater. The aim was to identify the point at which the fine aggregate loses its surface moisture but is still saturated with water. The sample was tested for surface moisture at 5 minute intervals. The test apparatus was a conical mould filled with sand. A tamper was used to compact the sand. The moisture content of aggregate was tested by lifting the mould. Once sand begun to slump, it was believed to be at saturated-and-surface-dried condition. The sample was then weighted before being transferred to an oven. The oven-dried sample was weighed after an

appropriate time and the difference between the two values was taken as the “absorption” of the sand.



Figure 4.1: the SSD test

## 4.2 Preparing Alkaline Liquids

Prior to the day of mixing, a sodium hydroxide solution was prepared by dissolving sodium hydroxide pellets in distilled water. According to the concentration, two types of solution were prepared: 8molar and 14molar. Each kg of 8molar solution consisted of 262 grams of pellets. Calculations were made to take into account the impurity percentage of pellets. Likewise, in one kg of 14molar solution, 404 grams pellets were used. After mixing, the solution was left in a fume cupboard in the Chemical Engineering Lab to cool down. Then it was completely mixed with a sodium silicate solution and extra water if needed. On the day of mixing, the required amount of superplasticizer was poured in the activator liquids mixture and was mixed thoroughly with the solution.



Figure 4.2: Preparing the sodium hydroxide solution

### 4.3 Mixing

Laboratory work was carried out using the facilities in the Concrete Lab, Curtin University of Technology. For mixing concrete, a rotary pan mixer was used (Figure 4.3).



Figure 4.3: Mixing geopolymer concrete

The method adapted for making geopolymer concrete had been used by many researchers (Hardjito & Rangan 2005a; Sofi et al. 2007) and generally consisted of mixing dry materials e.g. aggregate and fly ash for a few minutes followed by adding alkaline activators as the mixing for another couple of minutes. In this work, aggregates, including 10mm and 7mm coarse aggregates were poured into the mixer followed by fly ash and lastly fine aggregates; then dry materials were mixed for three minutes. The premixed alkaline activator, extra water and superplasticizer were added and wet mixing continued for an extra four minutes.

#### **4.4 Casting and Compaction**

After mixing, the geopolymer concrete mix was cast into cylinder moulds of 10cm diameter in cross section and 20cm height. The inside of the cylinder was coated with a thin layer of a water-based release agent to facilitate demoulding of the samples after curing.

The compaction of fresh concrete was carried out using sixty hits per layer with a rod (Figure 4.4). Fresh concrete was cast in three layers in each cylinder. Each layer was vibrated on a vibrator table for 10 seconds to achieve the best compaction. The moulds were then covered in plastic sheets in order to prevent loss of moisture. The covered moulds were then transferred to the curing room for 24 hours “preset period” (Neville 1995) before the start of curing at elevated temperatures.



Figure 4.4: Compacting red sand geopolymer concrete



Figure 4.5: Red sand geopolymer concrete after casting

## 4.5 Curing

High temperature curing was started on the day after casting. For steam curing, the moulds were taken to the steam curing room and placed under a thick plastic “tent” (Figure 4.6). A boiler produced steam to provide heat and humidity. In this way, samples were cured in a high humidity environment along with a high temperature. Dry curing was achieved by placing samples in an oven. Specimens were sealed with covers during curing to ensure no loss of moisture was possible. After curing, the specimens were left in the laboratory environment for 5-6 hours before demoulding.



Figure 4.6: the steam curing chamber

## 4.6 Mix Design

Mixture proportions were selected based on mix design process for conventional concrete and according to previous reports (Hardjito & Rangan 2005a). In their work, they showed that a sodium silicate-to-sodium hydroxide ratio of 2.5 by mass yields higher compressive strength. This was kept constant throughout the experimental work. The ratio of activator solution-to-fly ash was selected 0.36. Like conventional concrete mix design, aggregates

comprised 70-75 percent of the total mass. As the major objective of the research was to identify the role of red sand as a fine aggregate, this percentage was altered to give the optimum proportion (see table 3.3 for details). Also, the ratio of fine-to-coarse aggregate varied for some mixtures in order to investigate its effect on workability and strength of geopolymer concrete. To enhance the workability of geopolymer concrete, extra water was added to the mix. Likewise, the mass of superplasticizer was selected 1.5% of the fly ash. Throughout this research two sets of mixtures were prepared. Mixtures 1 to 9 comprised 14molar sodium hydroxide, whereas mixtures 10 to 12 used 8molar sodium hydroxide. Within each group, various ratios were used according to the experimental objectives. Each series was tested for mechanical and durability properties. The evaluation consisted of the following tests:

- Compressive Strength
  - Effect of concentration of alkaline activator
  - Effect of aggregate-to-fly ash ratio
  - Effect of extra water
  - Effect of superplasticizer
  - Effect of percentage of Red sand
  - Effect of curing method
  - Effect of curing temperature
  - Effect of preset period
- Indirect tensile strength
- Flexural strength
- Determining the modulus of elasticity and Poisson's ratio

- Determining the Apparent Volume of Permeable Voids (AVPV)
- Drying shrinkage
- Scanning Electron Microscopy (SEM)

#### 4.7 Compressive Strength

The compressive strength test was conducted according to AS1012.9-1999. A Farnell hydraulic testing apparatus available at the Concrete Lab, Curtin University of Technology was used to carry out the test (Figure 4.7)



Figure 4.7: The compressive strength test machine

The test specimens were 100×200 mm cylinders. The specimens were cured after a one day preset period. After curing, the samples were demoulded and transferred to the laboratory to be kept under high humidity conditions until the day of testing. The compressive strength test involved capping the samples with a restrained natural rubber capping system prior to placing the specimens in the testing machine.



Figure 4.8: A geopolymer concrete specimen under load

A load was then applied at the constant rate of  $20 \pm 2$  MPa/min until it reached the maximum force and the failure of sample occurred. The compressive strength of specimens was calculated from equation (4.1):

$$f_{cu} = P/A \quad (4.1)$$

Where:

$f_{cu}$  = compressive strength of specimen

P = maximum force applied by machine

A = cross-sectional area

For most of the mixes, testing was carried out 3, 7, 14, 28 and 56 days after casting. For each testing, three samples were tested and their average was considered as compressive strength.

#### **4.8 Indirect Tensile Strength**

Indirect tensile tests were conducted in accordance with AS 1012.10-2000. The specimens were 150×300 mm cylinders. Three specimens were tested for each mixture 28 days after casting.

The tests process started with aligning two hardboard bearing strips between the top and bottom platen of the cylinder when the cylinder was placed in the centre of the testing jig. Force was applied at the constant rate of  $1.5 \pm 0.15$  MPa / min until no further increase in load was achieved. The indirect tensile strength of the specimen was calculated based on equation (4.2):

$$T = \frac{2000P}{\pi LD} \quad (4.2)$$

Where:

$T$  = indirect tensile strength, in MPa

$P$  = maximum applied force indicated by the testing machine, in KN

$L$  = length, in mm

$D$  = diameter, in mm

#### 4.9 Modulus of Rupture (Flexural Strength)

The flexural strength of geopolymer concrete specimens was determined with regard to AS 1012.11-2000. Beams were made 400 mm length in length and  $100 \times 100$  mm cross-section, steam cured on the day after casting for 24 hours, then transferred to the curing room, demoulded and left there until the day of testing. The specimens were tested at the age of 28 days.



Figure 4.9: The flexural strength test

The testing apparatus was a frame containing two supporting rollers and two loading rollers. The sample was placed on its side with respect to its position as molded and then was centered on the supporting rollers. A sitting load was applied to bring loading rollers into contact with the top of the specimen. The load was applied without shock and increased continuously at a rate equivalent to  $1 \pm 0.1$  MPa/min extreme fiber stress until no increase in force was achieved. With regard to the fraction location on the specimen, three situations may occur:

If the fracture occurs in the tension surface within the middle third of the span length, the modulus of rupture is calculated according to equation (4.3):

$$R = \frac{PL}{bd^2} \quad (4.3)$$

Where:

$R$  = modulus of rupture, MPa,

$P$  = maximum applied load indicated by the testing machine, N

$L$  = span length, mm

$b$  = average width of specimen, mm

$d$  = average depth of specimen, mm

If the fracture occurs in the tension surface outside of the middle third of the span length by not more than 5 % of the span length, the modulus of rupture is calculated by equation (4.4):

$$R = \frac{3Pa}{bd^2} \quad (4.4)$$

Where:

$a$  = average distance between the line of fracture and the nearest support measured on the tension surface of the beam, mm

If the fracture occurs in the tension surface outside of the middle third of the span length by more than 5 % of the span length, no calculation is made and the sample is rejected.

#### **4.10 Modulus of Elasticity and Poisson's Ratio**

Determining the modulus of elasticity and Poisson's ratio was carried out in accordance with AS 1012.17 – 1997. The specimens, 100 × 200 mm cylinders like the compressive strength specimens, were subjected to a one day preset period after casting. After that, they underwent steam curing for 24 hours and transferred to curing room to be kept in high humidity until the day of testing. Before the day of testing, samples were capped with a layer of sulfur mixture as in this way, the effect of surface roughness was removed and the load could be applied evenly and continuously.

Two linear variable displacement transducers (LVDT) were attached vertically to the sides of the specimen to measure the longitudinal displacement. Another LVDT was attached horizontally to the middle of the sample measuring the lateral displacement. The LVDTs were connected to a computer to transfer the data during the test.

According to AS 1012.17 – 1997, the test load was selected as 40 percent of the compressive strength of the samples. Three specimens were tested for compressive strength prior to a modulus test and the average was used to determine the test load. Three other samples were tested to determine the modulus of elasticity and Poisson's ratio.

#### 4.11 Apparent volume of permeable voids

The test for determining the water absorption and Apparent Volume of Permeable Voids (AVPV) in hardened geopolymer concrete was carried out according to AS 1012.21 – 1999.

The test specimens were  $100 \times 200$  mm cylinders which were cast and cured according to compressive strength specimens and subsequently cut into four equal slices. The top surface of the samples was trimmed to 3 mm to eliminate any irregularities. The average of four slices was taken as the result.

The test procedure started by weighing the samples to the nearest 0.1 g, followed by drying in an oven in the concrete laboratory. The samples were dried for a few days in the oven at a temperature between 100 to 110°C until the subsequent weights showed no difference in specimen weight. After removal from the oven, they were left to cool down in a desiccator containing silica gel to the temperature of  $23 \pm 2^\circ\text{C}$  and weighed ( $M_1$ ). Afterwards, the specimens were immersed in a water bath at a temperature of  $23 \pm 2^\circ\text{C}$  for more than two days and weighed ( $M_{2i}$ ). After two successive weighings of surface-dried samples there was no increase. The procedure was followed by weighing the specimens after boiling them in a water bath for 6 hours ( $M_{3b}$ ) and determining the suspended weight of samples ( $M_{4ib}$ ). The calculation of AVPV results was based on the following equations:

For specimens tested for immersed absorption:

$$Ai = \frac{(M_{2i} - M_1)}{M_1} \times 100\% \quad (4.5)$$

For specimens tested for boiled absorption:

$$Ab = \frac{(M_{3b} - M_1)}{M_1} \times 100\% \quad (4.6)$$

For specimens tested for apparent volume of permeable voids:

$$AVPV = \frac{(M_{3b} - M_1)}{M_{3b} - M_{4ib}} \times 100\% \quad (4.7)$$

#### **4.12 Drying shrinkage**

The drying shrinkage of geopolymer concrete was studied based on AS 1012.13 – 1992. For each mix, three specimens were made. The specimens were 75×75×285 mm prisms which were steam cured for twenty four hours after a one day preset period. Moulds were treated with a water-based release agent before casting. Each mould was filled in two layers, each layer receiving thirty strokes with a tamping bar. The fresh geopolymer concrete mixture was then vibrated for ten seconds on a vibrating table. The specimens underwent a one day preset period before being transferred to the steam curing chambers. The samples were then kept on racks in the curing room. Free circulation of air was provided using a wire mesh and spacing the specimens. Temperature and humidity with laboratory was kept within the AS1012.13-1992 criteria at all times.

The test consisted of measuring the length difference between the specimen and a reference bar at specific periods after demoulding. The drying shrinkage (indicated as micro strains) is the ratio of length change to the initial length of the sample. For measuring the length, a horizontal comparator in the Concrete Laboratory was used. A reference bar was used each time a sample was tested to check the precision of the apparatus. Each specimen was tested at least three times for each testing and the average of readings was taken. For each mixture, three specimens were made and samples tested for a period up to six months.



Figure 4.10: the drying shrinkage test

#### 4.13 Scanning Electron Microscopy

Mixtures 1, 2, 7 and 8 were selected to be examined by the Scanning Electron Microscope (SEM) available at the Electron Microscopy laboratory at the Centre for Materials Research, Curtin University of Technology.

SEM was conducted to investigate the microstructure of geopolymer concrete paste and to examine the porosity and crack formation of red sand geopolymer concrete.

SEM analysis was performed on polished specimens which were cut to 5 mm and then transferred to the laboratory to be cured and prepared before testing.

An energy dispersive X-Ray spectrometer was also used for elemental analysis.

## **5. RAPID HARDENING**

The major challenge which needed to be addressed during the laboratory work was rapid setting. Lack of literature mentioning this phenomenon was surprising. On numerous occasions throughout the experimental work, geopolymer mixtures set a short time after mixing had begun or even inside the mixer. On some occasions, the mix was so stiff that it prevented the pan mixer from rotating.

The rapid setting did not only occur in mixes with red sand but also the geopolymer mixtures comprising natural sand. However, it was noticeable that the mixtures with higher amount of water - such as mixtures containing lower concentrations of sodium hydroxide solution - did not show any sign of rapid setting. Although adding extra water to the mixture appeared to be advantageous in terms workability, it was ruled out as an option as extra water in the mixture would have affected the mix design and strength.

Rapid hardening of geopolymer concrete mixtures resulted in the disposal of numerous concrete batches that were prepared for this research. Figure 5.1 shows the rapid hardening phenomenon that occurred at the time of mixing in a geopolymer concrete mixture.



Figure 5.1: Rapid hardening at the time of mixing



Figure 5.2: A compacted mixture underwent hardening

Rapid hardening resulted in stiff concrete. Mixtures suffered from rapid hardening showed extremely low workability and the compaction process could not be achieved. Figure 5.3 shows the slump test carried out for a mixture which became stiff just after mixing.



Figure 5.3: Rapid hardening at the time of the slump test

The literature review did not shed light on the possible reasons of hardening because the situation had not been reported in the past. Therefore, a trial-and-error method was adopted to investigate the factors involved in making and casting geopolymer concrete mixtures. The following parameters were identified as possible reasons for rapid hardening:

- Moisture content of mixtures
- Temperature
- Superplasticizer
- Sodium Silicate
- Sodium hydroxide
- Fly ash

A range of experiments and tests was carried out in order to try and identify the cause of rapid hardening.

## 5.1 The Effect of Temperature

Temperature was identified as a major contributor to the rapid hardening of geopolymer concrete. The effect of temperature on rapid hardening was investigated by means of a series of experiments. As the problem was first encountered in the winter months, cold weather was considered as a possible factor. In the first experiment, a gas patio heater was set up close to the mixer (Figure 5.4). The heater was used to warm up the pan mixer before commencement of mixing. However, it did not affect the temperature of aggregates and other constituents prior to mixing. The number of geopolymer concrete batches that underwent rapid hardening decreased significantly after using the heater; however, it did not eliminate the problem entirely.

Since warmer ambience did not stop rapid hardening altogether, it was concluded that the ambient temperature is not the only governing factor. The temperature of the aggregates was also considered as a contributor to rapid hardening. As it was crucial for aggregate to be in saturated-surface-dry (SSD) condition, heating aggregate was ruled out as a solution.



Figure 5.4: Warming up the pan mixer before mixing

## 5.2 The Effect of Sodium Silicate Conditions

The quality of sodium silicate used in geopolymer concrete is also believed to be a factor in the hardening. Throughout this project, three different drums of sodium silicate were used, all from the same supplier. Although the three drums of sodium silicate were obtained from the same source, they each had a different visual appearance. The original drum contained a sodium silicate that was transparent and clear. The other two drums contained cloudy sodium silicate solution. It was observed that hardening only occurred when the cloudy solution was used.

Theoretically, the cloudy nature of sodium silicate solution indicated that it has segregated into different layers of concentration. It was thought that the bottom of the tank from which the pump draws the chemicals, contains cloudy sodium silicate due to the heavier particles settling gradually to the bottom of the drum.

The settlement of the sodium silicate solution could be explained by the fact that the drums sit in one position for an extended period of time. The extra concentration of the segregated sodium silicate could, hypothetically, account for the rapid hardening.

To test the settlement theory, the samples of sodium silicate were taken from the top, middle and bottom of the drums of sodium silicate and were left to settle over time. The samples, however, showed no indications of settling or segregation of the chemical after three months (Figure 5.5).



Figure 5.5: Sodium silicate solution samples

### 5.3 The Effect of Sodium Hydroxide Conditions

To investigate the effect of sodium hydroxide on rapid hardening, sodium hydroxide was supplied from a different supplier. The new sodium hydroxide was premixed before delivery and contained 50% water. Appropriate calculations were made to take into account the degree of impurity. Cubic moulds were used measuring  $7.5 \times 7.5 \times 7.5$  to cast geopolymer concrete mortars. The aim was to compare the mortars made with a different type of sodium hydroxide (i.e. premixed solution) with the samples that used the sodium hydroxide made with pellets. The mix design shown in Table 5.1 was adopted to make geopolymer mortar based on previous studies (Wallah & Rangan 2006).

Materials	Mass (kg/m <sup>3</sup> )
Natural sand	1052
Sodium silicate solution	196
Sodium hydroxide solution	78
Superplasticizer	12
Fly ash	774

**Table 5.1 materials used for preparing mortar specimens**

Two moulds were prepared for each type of sodium hydroxide; all other constituents were kept constant. The experiment outcome was inconclusive. Figure 5.6 shows the mixtures. It can be seen that one mould from each mixture underwent hardening, while the other one set normally.

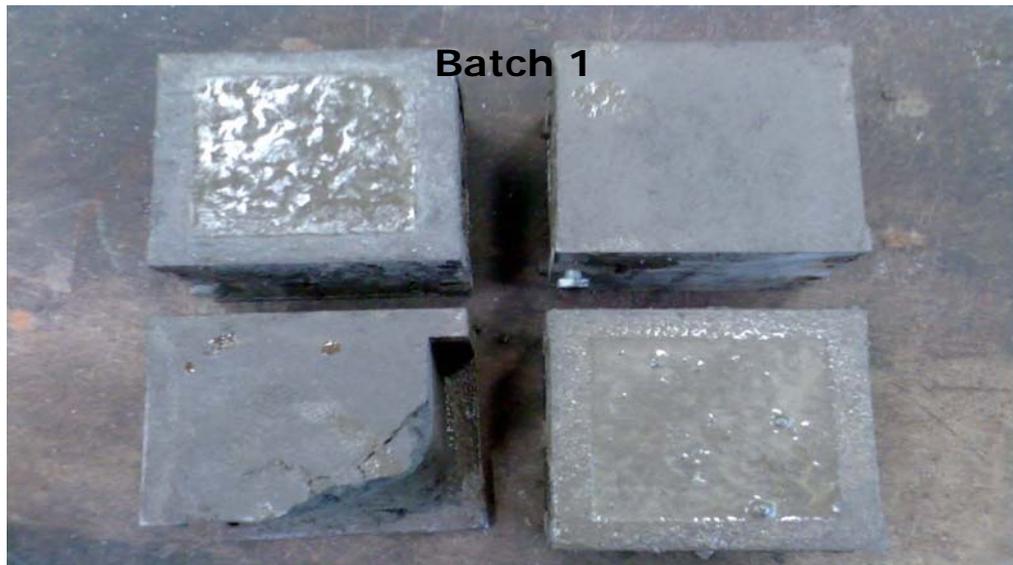


Figure 5.6: Mixtures using different sodium hydroxide solution sources



Figure 5.7: Geopolymer concrete mortars used different sodium hydroxide solution sources

## 5.4 The Effect of Water Content

Water content is also a major variable in the rapid hardening problem. Several experiments with different mix designs made it clear that mixtures with a lower water-to-solid ratio underwent hardening more often than the “lean” mixtures. Although adding extra water to the mix can prevent rapid hardening, this was ruled out as a method to increase the workability as tests during this project showed that the extra water had negative effect on the strength of geopolymer concrete.

## 5.5 Mortar experiments

Mortars were made using two of the different sodium silicates to test whether or not the source of sodium silicates was the cause of the hardening. The mortars for the tests contained fly ash, sodium hydroxide, sodium silicate, free water and superplasticizer – with the same ratios as in the geopolymer concrete batches.



Figure 5.8: Geopolymer binder samples

Two samples from each batch were taken. One sample from each type of mortar was placed in a cool environment (bucket of water), and one sample from each type was placed in a warmer environment (wrapped in blankets).

Hardening was measured by a simple penetration test with a tamping rod dropped from a distance of 20mm from the surface of the geopolymer mortar.

The findings from these tests were inconclusive. The cooler temperature showed no difference in hardening due to the warmer environment over a period of 24 hours. Although one of the mortars set in a shorter time than the other; none of the samples underwent anything close to what had been previously observed when “rapid hardening” occurred.

The temperatures of these mortars would have been warmer than the concrete batches that experienced hardening. The presence of SSD aggregates and a steel mixing pan are both major contributors to the cold temperatures that are believed to be part of the hardening problem.

In the last series of experiments, a sodium silicate solution was obtained from a different supplier. The new batch contained a transparent solution, however, after using it, rapid hardening did not cease.

## 6. RESULTS AND DISCUSSION

### 6.1 Compressive Strength

#### 6.1.1 The Effect of Concentration of the Alkali Activator

As previously stated, two different categories of mixtures were made during the experimental program with respect to the concentration of sodium hydroxide solutions: 8molar and 14molar mixtures. The primary objective of the test was to study the effect of sodium hydroxide solution on the compressive strength of red sand geopolymer concrete.

Mixtures selected from the 14molar category consisted of: 1, 7 and 8. Mixtures 10, 11 and 12 were selected from the 8molar category to be compared with the mixes above respectively. Figure 6.1 shows the results of the compressive strength tests:

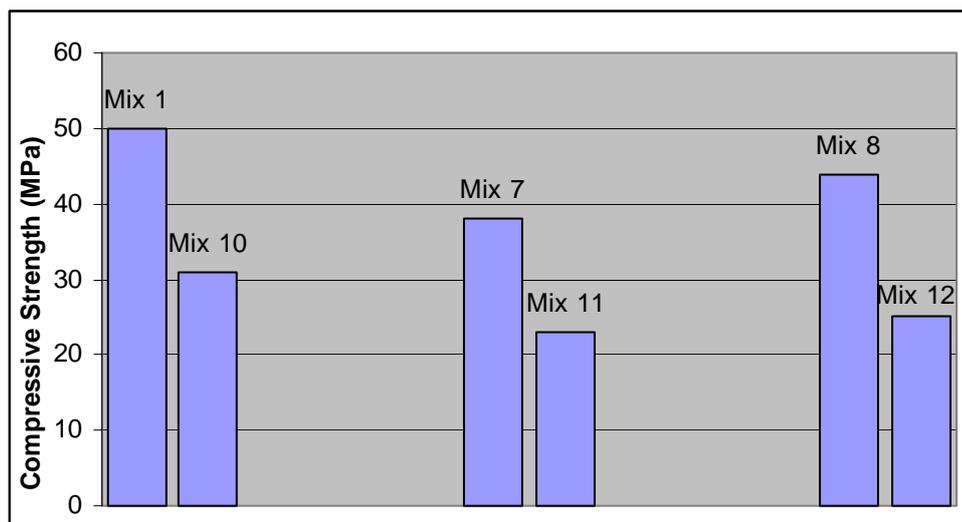


Figure 6.1: The effect of concentrations of alkaline liquids on compressive strength

It can be seen from the figure that the concentration of sodium hydroxide has a considerable effect on the compressive strength of red sand geopolymer concrete. The variation in strength is particularly significant as the characteristic strength increases and can be up to 60%. The results of the compressive strength for Red sand geopolymer concrete mixes were completely analogous to the previous finding by Hardjito and Rangan (2005a). The authors concluded that the concentration of sodium hydroxide is a governing factor in determining compressive strength of geopolymer concrete.

### **6.1.2 The Effect of Red Sand Inclusion**

Mixtures 1 to 4 were made with different percentages of red sand: mixture 4 used red sand as 25 percent of the total fine aggregate, whereas mixture 3 was made with a 50:50 ratio of red sand to natural sand. Mixture 2 used red sand as the sole fine aggregate and mixture 1 comprised 100% natural sand. All other constituents were kept constant.

For each mix, specimens were tested for compressive strength after 3, 7, 14, 28 and 56 days after casting. The average of three cylinders was taken as the mean compressive strength. At this stage, the aim was to understand the manufacturing process of geopolymer concrete incorporating red sand and to compare its workability and strength with correspondent natural sand mixture.

The slump test was carried out for mixtures 1 to 4. Table 6.1 shows the results.

<b>Mix No.</b>	<b>Slump (mm)</b>
<b>Mix 1</b>	250
<b>Mix 2</b>	10
<b>Mix 3</b>	155
<b>Mix 4</b>	180

**Table 6.1: Slump test results**

As the slump test results indicate, geopolymer concrete with natural sand has a reasonable slump value. In the laboratory, it did not show segregation at the time of mixing and compaction was achieved easily.

The test showed a 10mm slump for mixture 2, which comprised 100% red sand. The low slump and workability indicated that replacing natural sand with red sand noticeably decreases the workability of the mix. Other researchers have also mentioned the low slump values of red sand concrete (Ade Sri. Wahyuni 2005). The mixture seemed “dry” and stiff at the time of mixing and compaction was achieved with extra effort.

Two more mixtures were prepared that included 50, and 25 percent red sand - 50 and 75 percent natural sand respectively. The aim was to investigate the behavior of mixtures incorporating both types of sands and to study the effect of the inclusion of red sand as partial replacement to natural sand.

Mixtures 3 and 4 showed more workability than mixture 2 and less than mixture 1. The slump values for mixtures 3 and 4 were 155 and 180mm respectively. It is evident that the use of red sand as a fine aggregate reduces

the slump value: as the percentage of incorporated red sand reduces the workability and slump increase. However, the correlation between the slump value and red sand amount does not follow a well-defined trend (Figure 6.2).

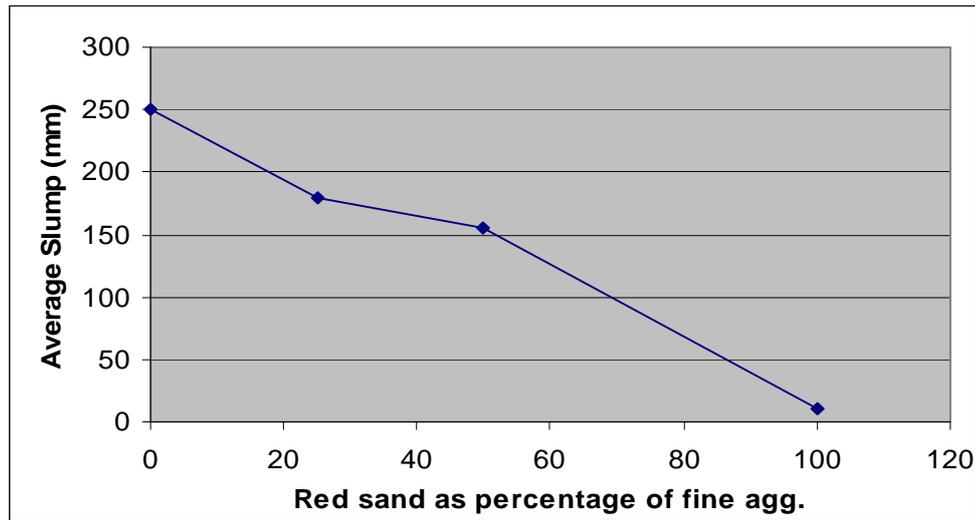


Figure 6.2: The effect of Red sand on workability of geopolymer concrete

The compressive strength test was done for mixtures 1 to 4 to investigate the effect of red sand on the strength of the geopolymer concrete mixture. The results are shown in Table 6.2.

	<b>DAY 3</b>	<b>DAY 7</b>	<b>DAY 14</b>	<b>DAY 28</b>	<b>DAY 56</b>
<b>Mix 1</b>	38	43	42	50	50
<b>Mix 2</b>	19	25	25	29	28
<b>Mix 3</b>	13	21	24	30	33
<b>Mix 4</b>	28	28	30	32	35

Table 6.2: Compressive strength results (MPa)

The data revealed that direct replacement of natural sand by red sand affects the compressive strength of geopolymer concrete. When natural sand was entirely replaced by red sand (Mix 2), the 56 day compressive strength decreased from 50 MPa to 28 MPa. As the percentage of red sand was reduced, the final compressive strength increased for mixtures 3 and 4. However, this increment is not considerable compared to the compressive strength of natural sand-geopolymer concrete.

The high surface area and water absorption of red sand can be responsible for lowering the final compressive strength. In conventional concrete, the amount of moisture which is absorbed by red sand can be compensated by extra water which is added to the mix; in this way, the actual water-to-cement ratio remains unchanged. In the case of red sand geopolymer concrete, the situation is more complicated. Since the alkaline activator is a combination of chemical solutions, adding water to the mixture does not keep the actual activator-to-fly ash ratio constant; on the contrary, the ingress of H<sub>2</sub>O ions affects the strength of the mix. It is, therefore, essential to consider other governing factors to increase the compressive strength of red sand geopolymer. Figure 6.3 shows the compressive strength results of mixes 1 to 4.

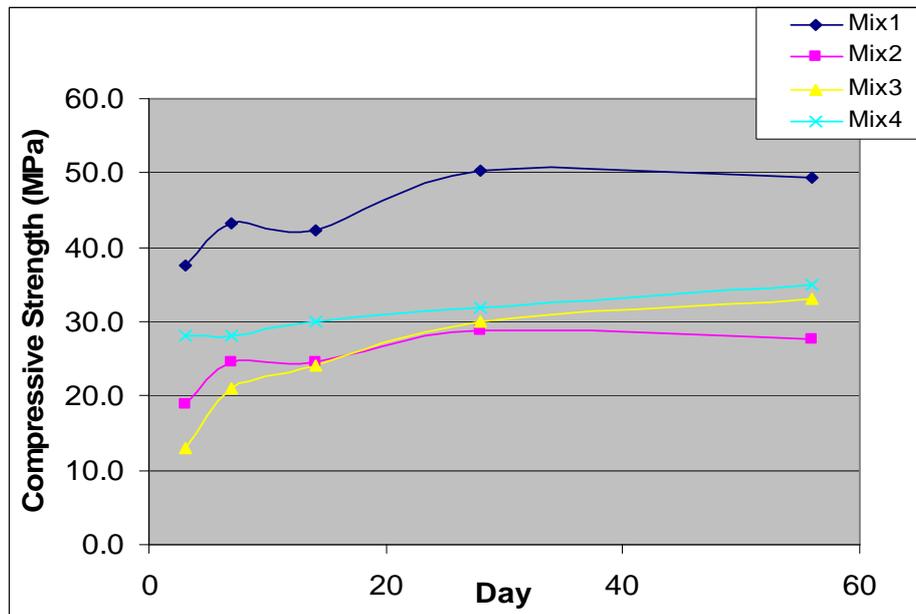


Figure 6.3: The effect of red sand on the compressive strength of geopolymer concrete samples

The other reason for the lower compressive strength of mixes 2 to 4 can be insufficient compaction. Although the grading of aggregate is not considered as effective on the compressive strength of concrete, it can affect the workability of the mixture. Compaction is not completely achieved in the case of non-workable mixtures as leaving behind micro pores indirectly affects the strength of the mixture. The strength of concrete is the function of the volume of voids in it (Neville 1995). A less compacted mixture means less material in a given volume and consequently less strength.

As the workability of red sand geopolymer concrete was not sufficient to achieve satisfactory compaction, attempts were made to enhance the workability and strength of geopolymer concrete incorporating red sand. The role of various parameters was investigated throughout the research to identify the governing factors.

### 6.1.3 The Effect of Superplasticizer and Extra Water

As the low workability of mixture was deemed as the main factor affecting the compaction process and final strength, the aim was to study the efficiency of conventional methods for increasing workability.

Mixture 5 was made using a 3% superplasticizer-to-fly ash ratio. The amount of superplasticizer was doubled in order to investigate its effect on the workability and strength of mixture. Mixture 6 was prepared comprising 2.5 times more water to identify the effect of extra water in a red sand geopolymer mixture on workability and strength.

The compressive strength results of mixtures 5 and 6 with mixture 2 are presented in Table 6.3 and Figure 6.4.

	<b>DAY 3</b>	<b>DAY 7</b>	<b>DAY 14</b>	<b>DAY 28</b>	<b>DAY 56</b>
<b>Mix 5</b>	16	19	19	20	23
<b>Mix 6</b>	11	11	13	13	12

**Table 6.3: Compressive strength results (MPa)**

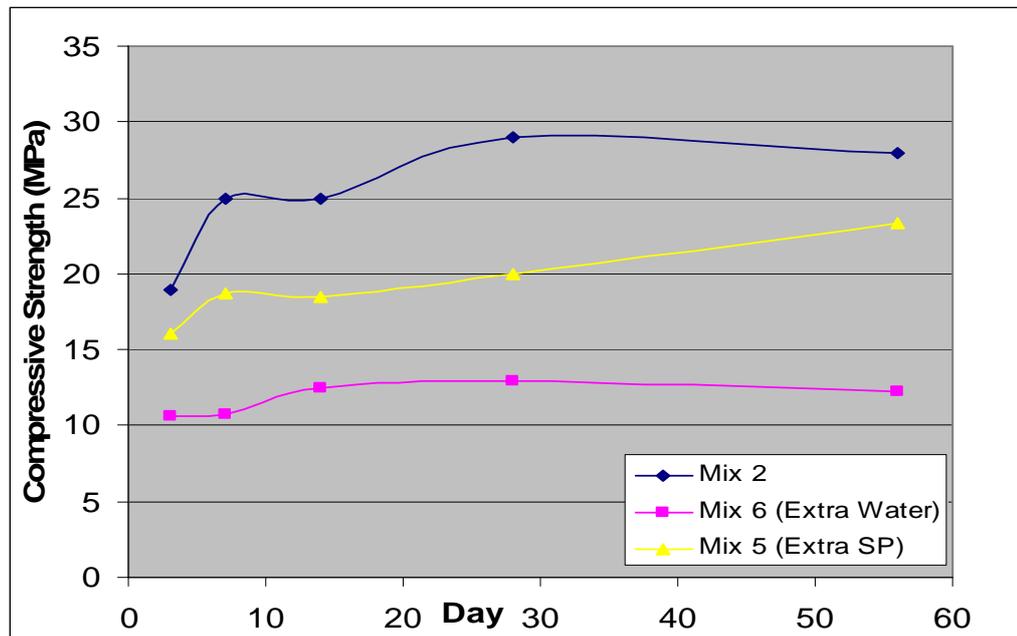


Figure 6.4: Effect of extra water and superplasticizer on compressive strength

The compressive strength of mix 5 was 23 MPa after 28 days. Comparing this to mix 2 which used the same constituents except for superplasticizer shows a 5MPa reduction. Although the workability of the mixture was higher, it was still not enough to yield the desirable compaction and to make up for the negative effect of extra superplasticizer on compressive strength. The result shows that increasing superplasticizer is not an efficient method to increase the behavior of fresh concrete and it also can decrease the strength of hardened mix.

In order to investigate the effect of extra water on the workability and strength of geopolymer concrete mixtures, mixture 6 was made using 2.5 times more water than mixture 2. All other proportions were kept constant.

The mixture was workable at the time of mixing, however, segregation occurred at the time of handling and compaction and the mix did not show cohesiveness due to excessive amounts of water. The 28 day compressive

strength, which was 12 MPa, showed a substantial degradation in comparison with mixture 2. The reduction in strength was completely in line with previous literature. As early studies suggest, unlike conventional concrete, the water has no role in geopolymerisation and is, in fact, expelled from the mix (Davidovits 1988; Xu & van Deventer 2002b). Furthermore, excessive water decreases the final strength of a mixture.

The water-to-solid ratio (introduced by Hardjito and Rangan (2005a)) is defined as the ratio of the sum of water which is present in sodium silicate, sodium hydroxide and extra water; to the sum of the geopolymer solids i.e. fly ash, pellets in sodium hydroxide and solid portion of sodium silicate. This is similar to the well-known “water-to-cement ratio” which is always considered to have the primary effect on the strength of conventional concrete. Similarly, the water-to-solid ratio affects the strength of geopolymer mixtures. The calculations of water-to-solid ratios are given in Appendix 1. The results for mixtures 1 to 6 are presented in Table 6.4.

<b>Mix</b>	<b>Water-to-solid ratio</b>
<b>Mix 1</b>	0.22
<b>Mix 2</b>	0.22
<b>Mix 3</b>	0.22
<b>Mix 4</b>	0.22
<b>Mix 5</b>	0.22
<b>Mix 6</b>	0.29

**Table 6.4: Water-to-solid ratios**

It can be seen from the Table 5.4 that mix 6 has the highest water-to-solid ratio. It is not unexpected that the lowest compressive strength belongs to this mixture

The results lead to the conclusion that the compressive strength of geopolymer concrete is highly dependent on the free water in the mix.

#### **6.1.4 The Effect of Aggregate-to-Fly Ash Ratio**

Mixtures 7, 8 and 9 were made using different aggregate gradings. When all mix designs remained identical to other mixes, red sand incorporation decreased from 35 percent to 30 percent of the total aggregate, but the 10mm coarse aggregate increased by five percent. The change in aggregate proportions was conducted to take into account the fineness of red sand. According to Neville (1995), the ratio of coarse to fine aggregate should be higher when finer sand is used. The particle size distribution of total aggregates shows that the recent grading conforms further to the British Standards and hence, it was believed that the workability of the mixture would improve (Figure 6.5 and Table 6.5)

	<b>10mm (%)</b>	<b>7mm (%)</b>	<b>Fine aggregate (%)</b>
<b>Grading 1</b>	30	35	35
<b>Grading 2</b>	35	35	30

**Table 6.5: Aggregate gradings used in this research**

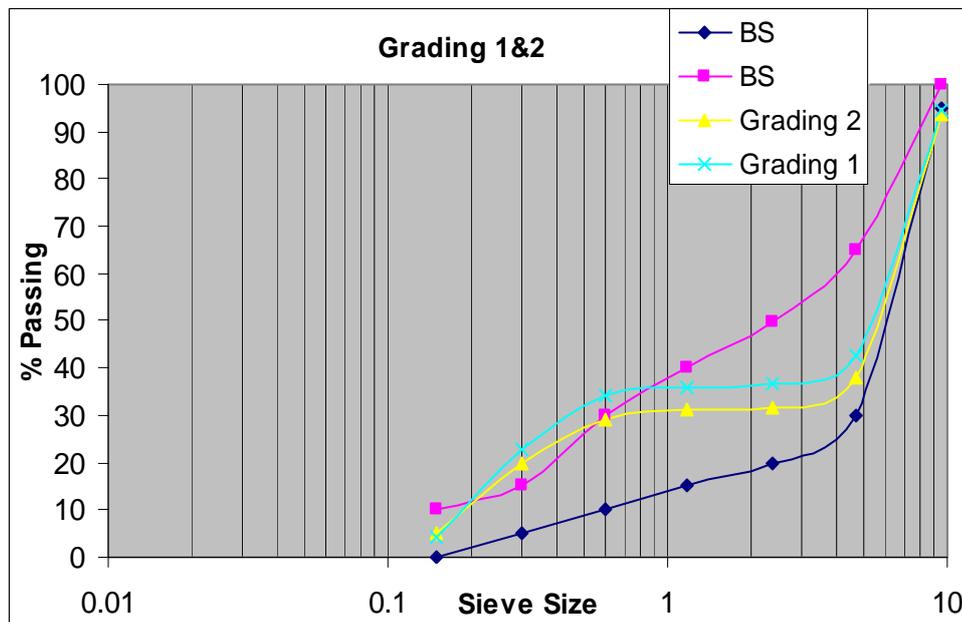


Figure 6.5: Particle size distributions of different gradings

It is shown in Figure 6.5 that the second grading – which was used for mixes 7, 8 and 9 –complies more with the limits set by the British Standard. Particularly, the percentage of aggregate passing through 0.600mm is completely within the limits and the deviation for material passing through 0.300mm is less.

The term “aggregate-to-fly ash ratio” was defined for geopolymer concrete which resembled the “aggregate-to-cement ratio” used in conventional concrete literature. For mixtures 1 to 9, aggregate-to-fly ash ratio values are given in Table 6.6 with other properties of mixtures. For mixes 1 to 6, the aggregate-to-fly ash value was kept constant and equal to 4.53. From mix 7, the ratio reduces to 4.23, 4 and 3.74 for mixes 7, 8 and 9 respectively.

Decreasing the ratio of aggregate-to-cement is an acceptable method in concrete mix design to enhance workability. The method is especially useful when particularly fine sand is in use and the high surface area of the aggregate

leaves insufficient moisture for cementitious materials. As shown in Table 6.6, except for mixture 5 which was made with extra water, the water-to-solid ratio of the mixes did not change. Also, as other proportions did not vary, a comparison could be made among the mixtures.

<b>Mixture</b>	<b>Water/solid ratio</b>	<b>Alkali activator/ Fly ash ratio</b>	<b>Sodium Silicate/ Sodium Hydroxide</b>	<b>Molarity of Sodium Hydroxide</b>	<b>Aggregate/Fly ash</b>
<b>1</b>	0.22	0.36	2.5	14	4.53
<b>2</b>	0.22	0.36	2.5	14	4.53
<b>3</b>	0.22	0.36	2.5	14	4.53
<b>4</b>	0.22	0.36	2.5	14	4.53
<b>5</b>	0.29	0.36	2.5	14	4.53
<b>6</b>	0.22	0.36	2.5	14	4.53
<b>7</b>	0.22	0.36	2.5	14	4.23
<b>8</b>	0.22	0.36	2.5	14	4.00
<b>9</b>	0.22	0.36	2.5	14	3.74

**Table 6.6: Details of 14molar mixtures**

The test results of the new mixtures showed a promising increase in mechanical properties of geopolymer concrete with red sand. The compressive strength results of mixtures 1, 7, 8 and 9 are given in Table 6.7:

	<b>DAY 3</b>	<b>DAY 7</b>	<b>DAY 14</b>	<b>DAY 28</b>	<b>DAY 56</b>
<b>Mix 1</b>	38	43	42	50	50
<b>Mix 7</b>	34	36	38	38	40
<b>Mix 8</b>	38	38	41	44	45
<b>Mix 9</b>	37	42	42	47	47

**Table 6.7: Compressive strength results of 14molar mixtures (MPa)**

It can be seen that mixtures 7 and 8 that used red sand as the sole fine aggregate yielded comparable results with mixture 1 which used natural sand only. Mix 9 showed the highest compressive strength amongst the Red sand mixtures. The mixtures were also workable and compaction was achieved easily. High strength and good workability of mixtures can be attributed to the new mix proportions. The mix design used for mixes 7 to 9 contained more “paste” compared to mixes 1 to 7. In other words, there was enough paste for coating aggregate and solid constituents of concrete; hence, higher workability resulted in better compaction. Furthermore, the liquid activators were consumed by cementitious materials and provided higher compressive strength.

The effect of aggregate-to-fly ash ratio on the workability and strength of Red Sand geopolymer concrete was further examined by making additional mixtures 10 to 12. The new series of mixtures used 8molar sodium hydroxide. Details of mixes 10 to 12 are given in Table 6.8. The 8molar sodium hydroxide was used to make medium strength geopolymer concrete as previous research results showed that the concentration of sodium hydroxide has a substantial effect on the compressive strength of geopolymer concrete (Hardjito & Rangan 2005a).

Mix 10 was made as a control mix. The fine aggregate comprised only natural sand and the mix proportions were identical to mixes 1 to 6. The 8molar sodium hydroxide was prepared by dissolving 262 grams of sodium hydroxide pellets in one kg of solution.

Mixes 11 and 12 were prepared with red sand as the fine aggregate. The mix design was similar to mixes 7 to 9 since this mix was found appropriate for red sand geopolymer concrete.

The details of mixtures 10 to 12 are given in Table 3.3 and other features of mixes 10, 11 and 12 are shown in Table 6.8.

Mixture	water/solid ratio	Alkali activator/ Fly ash ratio	Sodium Silicate/ Sodium Hydroxide	Molarity of Sodium Hydroxide	Aggregate /Fly ash
10	0.24	0.36	2.5	8	4.53
11	0.24	0.36	2.5	8	4.22
12	0.24	0.36	2.5	8	4.00

**Table 6.8: Details of 8molar Mixtures**

In the new set of mixtures, like mixes 7 and 8, the ratio of aggregate-to-fly ash decreased from 4.53 to 4.22 in mix 11 and to 4.00 in mix 12. The aim was to find out whether the method used for making mixes 7, 8 and 9 is useful for making medium strength geopolymer concrete with red sand.

The compressive strength of mixtures was determined 3, 7, 14, 28 and 56 days after casting. The results are presented in Table 6.9.

	DAY 3	DAY 7	DAY 14	DAY 28	DAY 56
Mix 10	25	28	27	31	32
Mix 11	18	21	19	23	24
Mix 12	21	23	23	25	26

Table 6.9: The compressive strength of 8molar mixtures (MPa)

The results are illustrated in Figure 6.6.

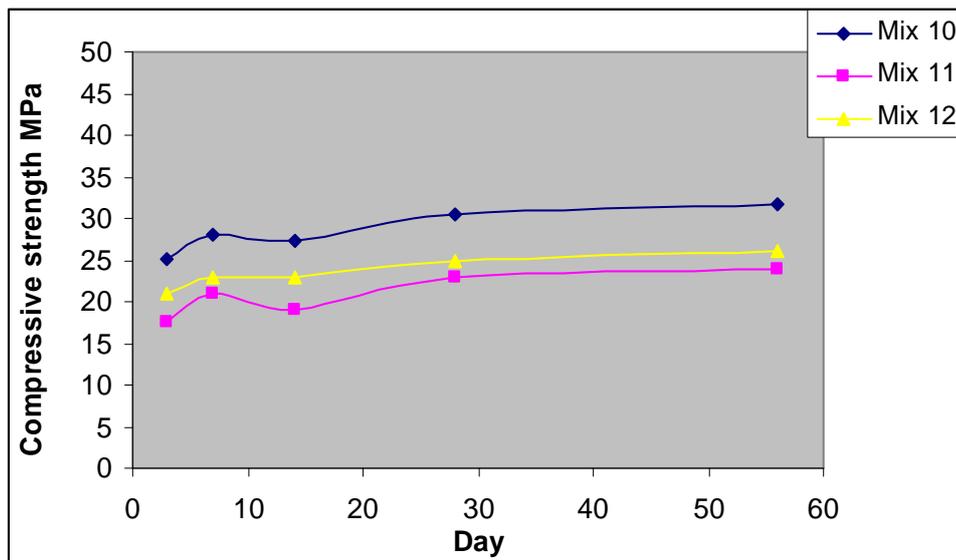


Figure 6.6: Compressive strength results

The data presented in Table 6.9 are analogous to compressive strength results of mixes 7 and 8. The results show that when natural sand is replaced with red sand, decreasing the aggregate to fly ash ratio is an effective method to enhance the mechanical properties of geopolymer concrete. The ratios of the mass of aggregate-to-total mass of mixture are given in Table 6.10.

<b>Mixture No.</b>	<b>Aggregate/ Total mass %</b>
<b>1</b>	76.1
<b>7</b>	74.8
<b>8</b>	73.8
<b>9</b>	72.4
<b>10</b>	76.1
<b>11</b>	74.8
<b>12</b>	73.8

**Table 6.10: Ratios of aggregate-to-total mass**

This shows that a slight decrease in aggregate portion is needed when natural sand is replaced with red sand. Figure 6.7 shows the compressive strength of red sand geopolymer concrete at different aggregate-to-fly ash ratios. The data are presented as a percentage of compressive strength of mixture 2 with aggregate-to fly ash ratio of 4.53.

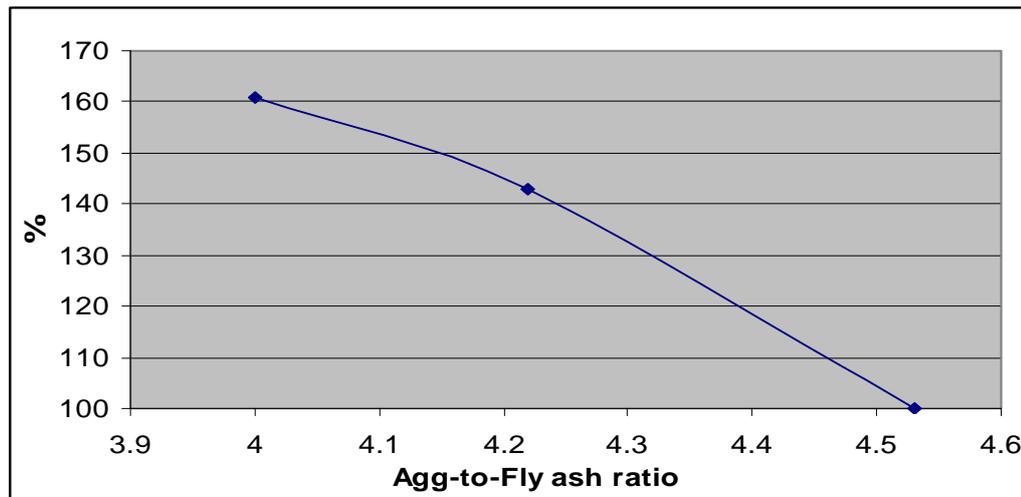


Figure 6.7: Compressive strength as a percentage according to mix2

It is noticeable that a reduction in aggregate-to-fly ash ratio can increase the strength of an individual mix by up to 60 percent. The development in strength is especially notable when one considers that the mix design has not changed and effective factors are kept constant.

### 6.1.5 The Effect of the Curing Method

In order to investigate the effect of the curing method on the compressive strength of specimens, a series of tests was conducted in the Concrete Laboratory. Two main methods of curing consisted of oven curing and steam curing. Steam curing was performed using the steam chamber in the steam curing room. Dry curing was carried out by putting the specimens in the oven when the surfaces of the cylinders were covered by plastic sheets. Mixture 2 was selected for the test. After casting, vibrating and compaction, specimens underwent a 24 hour preset period before curing. The objective of the test was to find out which method of curing is more effective.

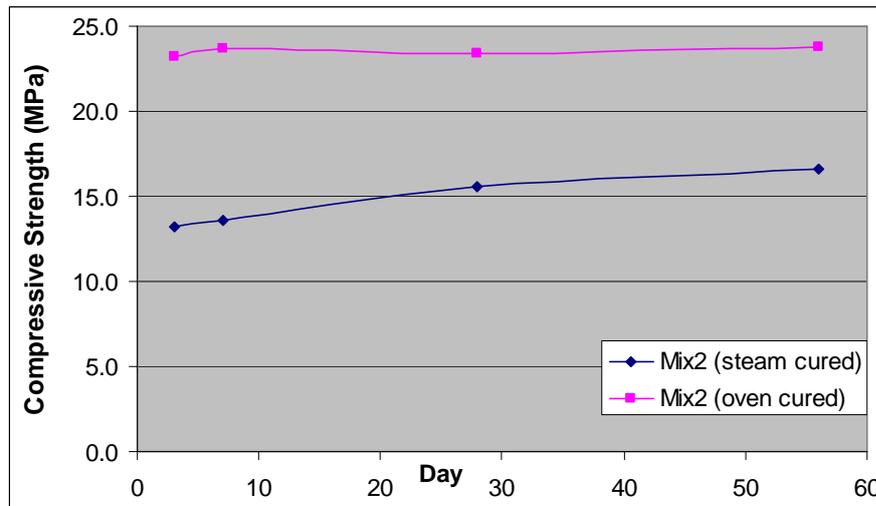


Figure 6.8: The effect of the curing method on compressive strength

It can be seen that the specimens which were cured in the oven showed higher compressive strength. The increase in compressive strength is fairly substantial for a mixture with 17MPa strength. Although the reason is not clear, it could be attributed to the constant performance of the oven that creates a controlled environment in which samples can be cured in an elevated temperature. In comparison, the steam chamber uses a “cycle” to produce high temperature steam. On the other hand, the steam chamber is not a completely closed environment as it can have heat transfer to the ambient through the unsealed plastic cover.

### 6.1.6 The Effect of the Curing Temperature

The curing temperature plays a significant role in the compressive strength of geopolymer concrete samples. Geopolymer concrete, like traditional concrete, gains strength when cured in elevated temperatures.

Mix 2 was selected to test the effect of curing temperature on compressive strength of red sand geopolymer concrete. A batch was made using the similar mixing and compaction procedure. Following compaction, samples were

divided into three groups with each group being transferred to a separate oven. The oven's temperatures were set at 60, 80 and 100 ° C. the objective was to compare the compressive strength of the samples that used a similar mix design but were cured in different temperatures. Table 6.11 shows the compressive strength results of the samples.

Curing temperature	Compressive Strength (MPa)		
	3 days	7 days	28 days
60 ° C	23	23	24
80 ° C	32	32	32
100 ° C	30	33	33

**Table 6.11: Compressive Strength values (MPa)**

It can be seen that increasing the curing temperature is an effective method to enhance the compressive strength of red sand geopolymer concrete. The results, graphically presented in Figure 6.9, show that the samples cured at 100° C were stronger than the rest of the samples; however, the gain was not substantial.

Therefore, curing at elevated temperatures up to 80 ° C was proved to be advantageous.

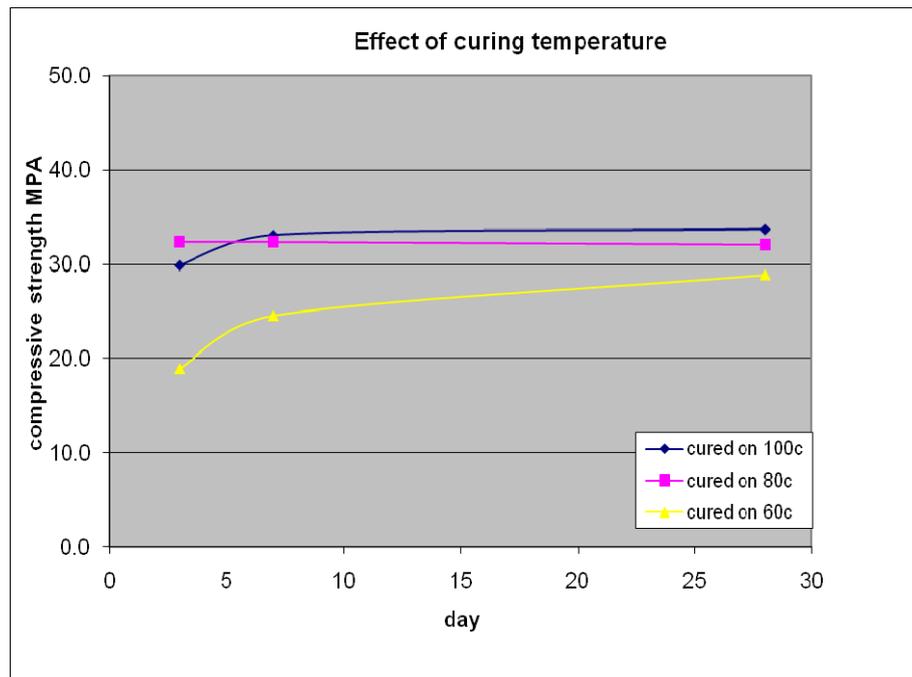


Figure 6.9: The effect of curing temperature on compressive strength

### 6.1.7 The Effect of the Preset Period

A “delay time” between casting and the start of curing concrete has been found to be advantageous to make it more durable and to increase strength. It has been stated that a “preset period” is beneficial before steam curing of conventional concrete (Neville 1995).

Alike traditional concrete, geopolymer concrete has been shown to gain more strength if it undergoes a delay time before being exposed to high temperature (Hardjito & Rangan 2005a).

The effect of a preset period on the strength of Red sand geopolymer concrete was studied by testing mix 2. Three mixes were made with the same mix design and cured at an elevated temperature. The mixes underwent 0, 1 and 2 day

delay time. Table 6.12 shows the results of the compressive strength of the mixtures:

Preset period	Compressive Strength (MPa)		
	3days	7days	28days
(No preset period)	17.4	17	19
One day	21.5	21.3	19.9
Two days	21.8	22.6	25.5

**Table 6.12: Compressive strength values (MPa)**

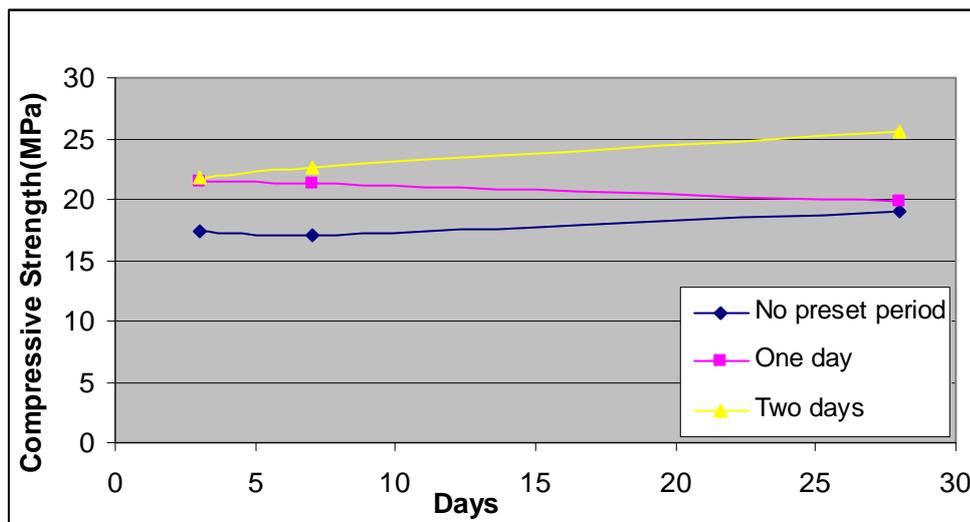


Figure 6.10: The effect of a preset period on compressive strength

Figure 6.10 shows that samples that underwent a two days preset period gained up to a considerable amount of 34% more compressive strength. Samples that were cured after a one day preset period did not show a significant increase in compressive strength on the 28th day; however, this could be attributed to test circumstances since the difference was considerable in the short term.

Figure 6.11 shows the development of compressive strength as mixtures experienced different preset periods.

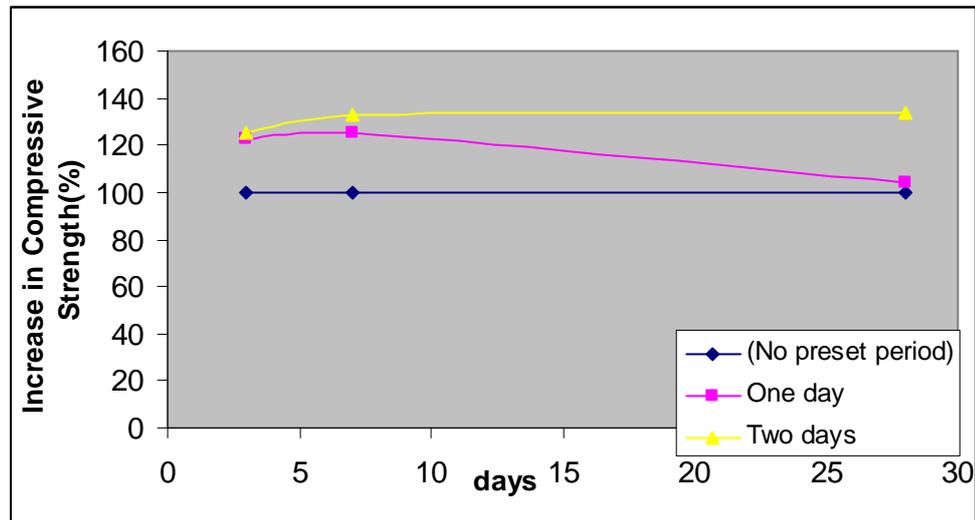


Figure 6.11: Increases in compressive strengths due to various preset periods

## 6.2 Indirect Tensile Strength

Although concrete design is normally based on compressive strength, knowing the tensile strength of hardened concrete is a matter of interest in many cases. Concrete tends to crack in tension leaving the reinforcement more susceptible to corrosion. Also, the propagation of cracks may lead to higher stress in steel. The tensile strength is also important in concrete structures like dams and highways (Neville 1995).

The tensile strength of concrete can be evaluated by different methods: a direct tension test, a flexure test, and a splitting test. However, the splitting test showed it gives more uniform results than other tension tests and the results are believed to be close to the direct tensile test (Neville 1995).

Splitting strength was carried out for mixes 1, 8, 10, 11 and 12. Mix1 used natural sand as the sole fine aggregate and mix8 incorporated red sand without any natural sand. Both mixtures 1 and 8 were made with 14molar sodium hydroxide. On the other hand, mixtures 10, 11 and 12 used 8molar sodium hydroxide. Mix10 was made with natural sand whereas mixes11 and 12 used 100% red sand as a fine aggregate. The objective of the test was to evaluate the effect of red sand on the splitting strength of geopolymer concrete and to enhance the splitting strength by using different aggregate-to-fly ash ratios.

An indirect tensile test was conducted in accordance with AS 1012.10-2000 at the Concrete Laboratory, Curtin University.

Mixture 8 shows comparable results with control mix. It indicates that by replacing natural sand by red sand, the indirect tensile strength of geopolymer concrete is not significantly affected.

Standards Australia offers the equation (6.1) to relate the splitting strength to the compressive strength:

$$f'_{ct} = 0.4\sqrt{f_{cm}} \quad (6.1)$$

While Neville (1995)suggested the following equation to express the relation between the two (equation 6.2):

$$f'_{ct} = 0.3f_{cm}^{(2/3)} \quad (6.2)$$

A comparison between the two equations and measured splitting strength values is given in Table 6.13.

	<b>Compressive Strength</b>	<b>Splitting Strength (measured)</b>	<b>Splitting Strength (equation 6.1)</b>	<b>Splitting Strength (equation 6.2)</b>
<b>Mix 1</b>	50	3.82	2.83	4.07
<b>Mix 8</b>	44	3.52	2.65	3.74

**Table 6.13: Indirect tensile strength (MPa)**

It can be seen that measured splitting strength values fall between two equations that relate the indirect tensile strength to compressive strength. For mixes 1 and 8, the measured splitting strength is higher than that of the equation (6.1), whereas, equation (6.2) predicts a higher splitting strength.

Table 6.14 shows the results of mixes 10, 11 and 12. It is notable that both mixes that used red sand as a fine aggregate yielded higher indirect tensile strength than a corresponding natural sand mix (mixture 10).

As with mixes 1 and 8, a comparison is made for mixes 10 to 12 between the measured values of the splitting test and the relevant equations (Table 6.14)

	<b>Compressive Strength</b>	<b>Splitting Strength (measured)</b>	<b>Splitting Strength (equation 6.1)</b>	<b>Splitting Strength (equation 6.2)</b>
<b>Mix 10</b>	31	2.43	2.23	2.96
<b>Mix 11</b>	23	2.62	1.92	2.43
<b>Mix 12</b>	25	2.43	2	2.56

**Table 6.14: Indirect tensile strength (MPa)**

Mixes 10 and 12 showed identical splitting strengths despite the fact that the compressive strength of mix 10 was higher than mix 12. Comparing the measured results with the equations, the data are analogous to the measured values of mixes 1 and 8. While Mix 12 possessed the highest compressive strength amongst the group, mix 11 showed a slightly higher splitting strength. The higher indirect tensile strength of mix 11 could be due to the fact that properly centering the specimen under the load is extremely difficult as there is no means to make sure that the load is applied without any eccentricity (Neville 1995).

In view of the data obtained from mixes 1, 8, 10, 11 and 12, it can be concluded that replacing natural sand with red sand does not significantly affect the splitting strength of the 14M mixtures but in fact enhances the strength of specimens made with 8M concentration (low strength). The higher strength of red sand mixtures can be attributed to the angularity of particles. The crushed particles create a stronger bond with paste that leads to higher strength in splitting.

### 6.3 Modulus of Rupture (Flexural Strength)

The flexural strength of the geopolymer beam specimens was determined in two categories: mixes 1, 2, 3, 4, 7, and 8 were selected from 14M mixes; and mixes 10, 11 and 12 that used 8M sodium hydroxide. For each mixture, three beams were tested to give the average flexural strength. The values of flexural strength for mixes 1 to 8 are given in Figure 6.12.

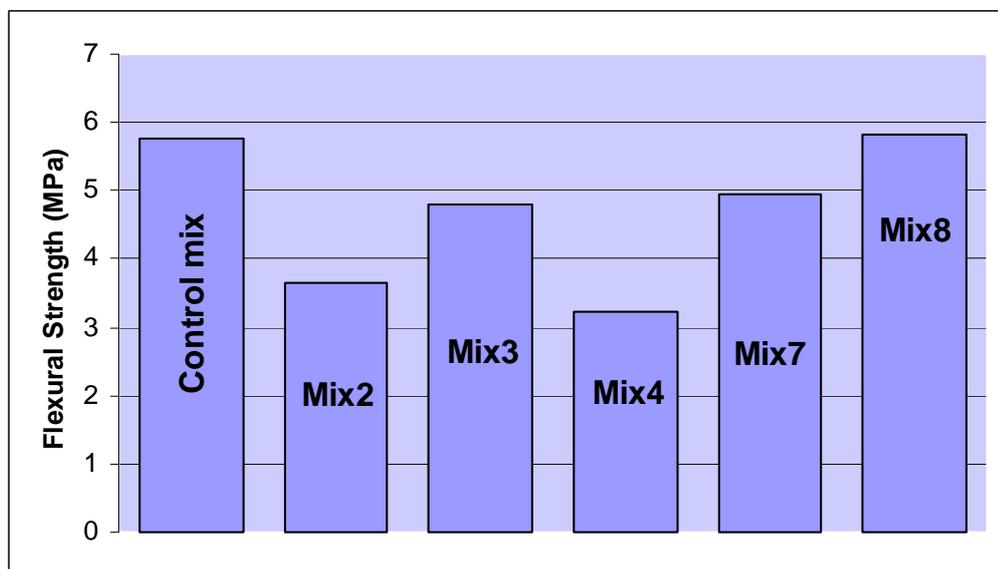


Figure 6.12: The flexural strength of 14molar mixtures

Mix 2 showed a relatively low strength compared to the control mix. As the percentage of red sand decreased from 100 in mix 2 to 50% in mix 3, the strength increased. Considering that mixes 2 and 3 used the same mix design as the control mix, it again indicates that replacing natural sand with red sand requires a reduction in aggregate-to-fly ash ratio. Mix 8, which used 100% red sand but a lower aggregate-to-fly ash ratio, showed the highest flexural strength indicating that using red sand can increase the modulus of rupture of the geopolymer beams.

The lowest strength amongst the group belonged to mix 4. Using the same proportions of materials, the only difference between mix 4 and mix 2 was the doubled quantity of superplasticizer. In line with a previous study at Curtin University (Hardjito & Rangan 2005a), using extra superplasticizer seems to have negative effect on the flexural strength of geopolymer concrete as well as its compressive strength.

The flexural strength of mixes 10, 11 and 12 are shown in Figure 6.13.

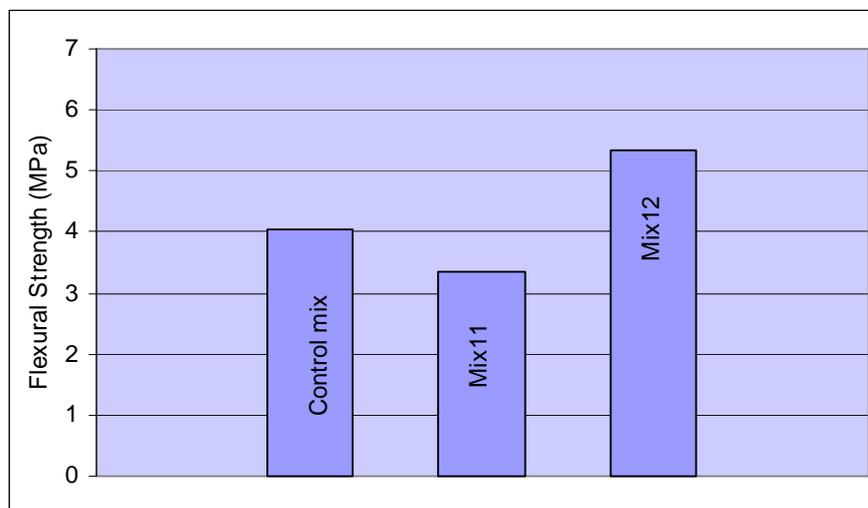


Figure 6.13: Flexural strength of 8molar mixtures

Mix 12 showed the highest flexural strength amongst the 8M mixtures. Although the data are not conclusive in terms of the effect of red sand on flexural strength, a similar trend is observed by comparing the results of the two groups (8M and 14M). Within both groups, the highest flexural strength is shown by the mixture with least aggregate-to-fly ash ratio.

The behavior of red sand geopolymer concrete under a flexural test is analogous to an indirect tensile test: in both tests, geopolymer concrete mixture containing red sand showed higher strength compared to the control mixes containing natural sand. As Neville (1995) suggests, the interlocking of

the aggregate and cementitious paste is an important factor in flexural strength. The stronger bond between crushed particles of red sand and geopolymer paste provides higher strength compared to the bond between rounded-shape natural sand particles and geopolymer paste. The good flexural and indirect tensile strength of red sand geopolymer concrete may suggest this material can be used in areas like pavement slabs, where the flexural strength of concrete is of great interest.

#### **6.4 Drying Shrinkage**

A drying shrinkage test was conducted for four mixtures: 1, 6, 7 and 8. The objective of the test was to identify the effect of red sand on the drying shrinkage of geopolymer concrete. Mixture 1 was tested as a control mix as it contained natural sand as fine aggregate. Mixtures 6, 7 and 8 were then selected with red sand as the sole fine aggregate. The effect of extra water on drying shrinkage was investigated by using mixture 6 and mixtures 7 and 8 provided data regarding the effect of different aggregate-to-fly ash ratio on the drying shrinkage of geopolymer concrete beams.

For each mixture, the average of three specimens was selected as the mean drying shrinkage. The specimens were visually examined before storing to ensure that no voids were present as samples with voids or cracks on the surface must be rejected according to AS 1012.13 – 1992. Figure 6.14 shows the drying shrinkage test results for the 14M category.

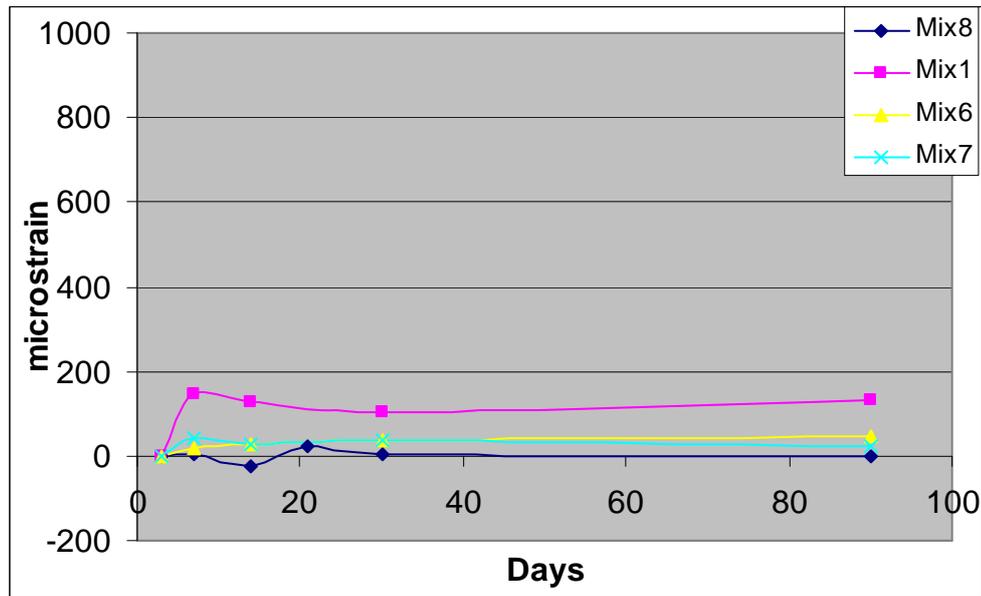


Figure 6.14: The drying shrinkage of mixtures

It can be seen that the drying shrinkage of red sand geopolymer concrete is considerably less than Portland Cement Concrete (AS 1379-2007 sets the limit of 1000 micro strain drying shrinkage after 56 days for normal concrete). In the geopolymerisation process water is expelled from the mixture. The removal of pore water in geopolymer concrete occurs mainly at the time of heat curing. With less water present in mixture pores after curing, the susceptibility of the mixture to drying shrinkage is less. As the water-to-cement ratio is believed to be a factor influencing drying shrinkage in traditional concrete (Neville 1995), insubstantial drying shrinkage of geopolymer concrete can be attributed to less amounts of evaporable water.

In the early stages, the samples undergo a cycle of swelling and shrinking. This is analogous to other researchers' findings (Hardjito & Rangan 2005a). At this period, the movement of water from and to the mixture can happen in the humid environment of a laboratory, but as time passes by, the hydration

process is more complete and withdrawal of water from the mix becomes steady.

All mixes showed very small shrinkage. Although the difference between the measured values among the mixes was insignificant, it was noticeable that those that used red sand suffered less shrinkage than the control mix that used natural sand.

The results for mixes 7 and 8 show that decreasing the aggregate-to-fly ash ratio from 4.23 to 4.00 has a negligible effect on the drying shrinkage of geopolymer concrete samples made from red sand. On the other hand, from the results of mix 6, it can be deduced that increasing the amount of added water does not increase the drying shrinkage values significantly. It is mainly because the extra water in the mixture, which is used to facilitate higher workability, would be expelled from the mixture at the curing stage. The remaining mixture has less water to lose; hence lower drying shrinkage values.

## 6.5 The Modulus of Elasticity and Poisson's Ratio

The modulus of elasticity and Poisson's ratio was determined for two groups: 14 molar and 8 molar mixtures. The objective of the experiment was to identify the effect of red sand on the modulus of elasticity and Poisson's ratio of geopolymer concrete. Standards Australia suggests the equation (6.3) for determining the modulus of elasticity:

$$E_c = \rho^{1.5} \times (0.043\sqrt{f_{cm}}) \quad (6.3)$$

Where,

$E_C$  is the Modulus of Elasticity,  $\rho$  is the average density of concrete in  $\text{kg/m}^3$  (which was taken as  $2350 \text{ kg/m}^3$ ); and  $f_{cm}$  is compressive strength in MPa. The equation described in AS 3600-2001 gives a value with a range of  $\pm 20\%$ .

Table 6.15 shows the measured values of the modulus of elasticity in the laboratory and the calculated values based on equation (6.3).

Mixture	Compressive Strength (MPa)	Modulus of Elasticity (equation 6.3)	Modulus of Elasticity (Measured)	Poisson's Ratio
Mixture 1	50	$34.6 \pm 7$	26.6	0.15
Mixture 7	40	$31 \pm 6.3$	22.6	0.16
Mixture 8	45	$32.9 \pm 6.7$	22.5	0.16

**Table 6.15: Modulus of elasticity and Poisson's ratios**

Mixtures 10, 11 and 12 were tested, the results of which are shown in Table 6.16.

Mixture	Compressive Strength (MPa)	Modulus of Elasticity (equation 6.3)	Modulus of Elasticity (Measured)	Poisson's Ratio
Mixture 10	32	$27.7 \pm 5.5$	20.7	0.14
Mixture 11	24	$24 \pm 4.8$	18	0.19
Mixture 12	26	$25 \pm 5$	18.4	0.15

**Table 6.16: Modulus of Elasticity and Poisson's ratios**

The test results show that measured values of the modulus of elasticity were less than values calculated by the AS equation. Mixtures that contained red sand showed the same trend as control mixes. Measured values of Poisson's

ratios ranged from 0.14 to 0.19 similar to conventional concrete. Results were also consistent with a previous research by Hardjito and Rangan (2005a).

## 6.6 Apparent Volume of Permeable Voids (AVPV)

The volume of pore space in geopolymer concrete mixtures was determined by an AVPV test according to AS 1012.21. This measures the volume of voids in concrete "as distinct from the ease with which a fluid can penetrate it" (Neville 1995). AVPV was carried out for seven mixtures. Table 6.17 shows the results.

	<b>Immersed Absorption</b>	<b>Boiled Absorption</b>	<b>AVPV</b>
<b>Mix 1</b>	4.3	4.8	11.0
<b>Mix 2</b>	4.9	5.4	12.3
<b>Mix 7</b>	5.0	5.4	12.4
<b>Mix 8</b>	5.1	5.7	12.8
<b>Mix10</b>	4.31	4.53	10.25
<b>Mix11</b>	4.60	4.84	11.00
<b>Mix12</b>	4.58	4.77	10.90

**Table 6.17: Apparent Volume of Permeable Voids**

Vicroads provides a series of test methods for assessing the durability features of conventional concrete. Table 6.18 shows the values presented by Vicroads for concrete with various values of characteristic strength.

<b>Concrete grade</b>	<b>Test Cylinders (compacted by vibration)</b>	<b>Test Cylinders (compacted by rodding)</b>	<b>Test cores</b>
<b>VR330/32</b>	14	15	17
<b>VR400/40</b>	13	14	16
<b>VR450/50</b>	12	13	15
<b>VR470/55</b>	11	12	14

**Table 6.18: AVPV values recommended by Vicroads**

A comparison made between the measured values of AVPV test and values suggested by Vicroads showed that red sand geopolymer concrete comprises lower permeable voids than conventional concrete. The results suggested that interconnected pores within geopolymer concrete mixtures are within limits set by standards. The permeable voids inside a mixture comprise capillary

pores, gel pores, air voids and micro cracks that can absorb water and other fluids and subsequently reduce concrete durability. As the amount of water that can be absorbed into concrete is a function of the permeable voids of the concrete, it is considered that concrete with less permeable pores or voids should better withstand an aggressive environment than concrete with more permeable pores or voids (VicRoads 2007).

A comparison was also made between control mixes that used natural sand (mixtures 1 and 10) and mixes that used red sand (mixtures 2, 7, 8, 11 and 12). In all cases, the volume of permeable voids was within accepted values. Even red sand mixtures that exhibit lower workability showed good durability in terms of volume of permeable voids.

## 6.7 Scanning Electron Microscopy (SEM)

Geopolymer samples were also examined under a Scanning Electron Microscope. Samples were selected from mixtures 1, 2, 7 and 8. Figure 6.15 shows the SEM micrograph of a specimen from mixture 1.

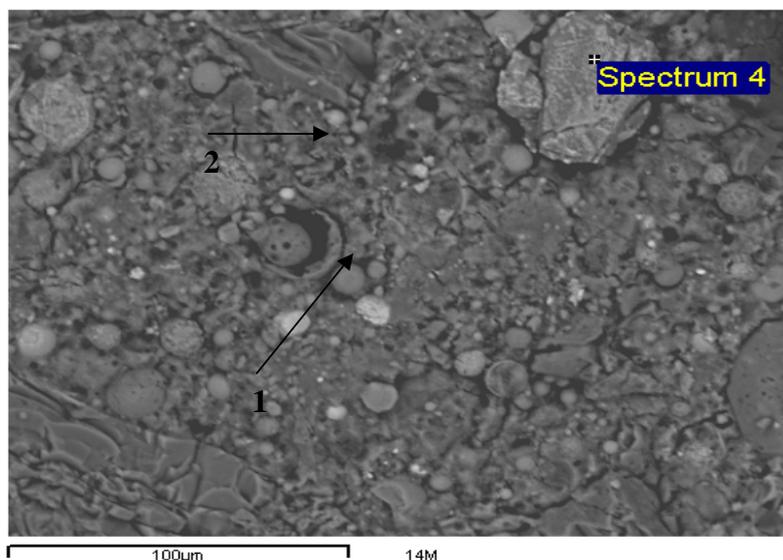


Figure 6.15: SEM micrograph of mixture 1

The picture reveals that mixture 1 consisted of a paste with a combination of porous and dense areas. At the bottom left side of the picture, a more compacted area is visible. Arrow 1 indicates a non-reacted fly ash grain and 2 shows a micro crack. The crack can have occurred at the time of cutting. Mixture 1 incorporated natural sand and yielded 50MPa compressive strength after 28 days.

Figure 6.16 shows SEM micrograph from mix 2 which used red sand as a fine aggregate. A combination of porous and well-compacted areas is also notable.

A good example of geopolymer paste is shown in Figure 6.17. The SEM picture was taken from mix 7 that used red sand as the sole aggregate. The high density of produced geopolymer gel is responsible for compressive strength of the mixture.

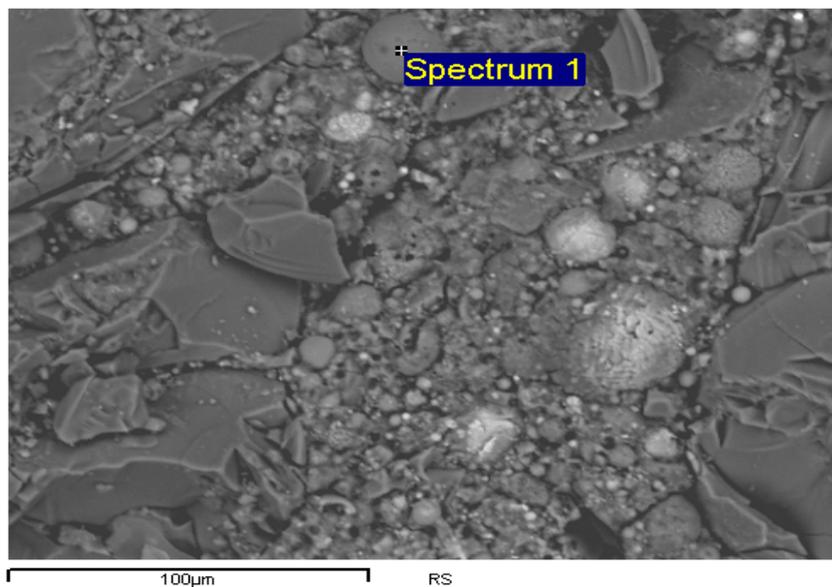


Figure 6.16: SEM micrograph of mixture 2

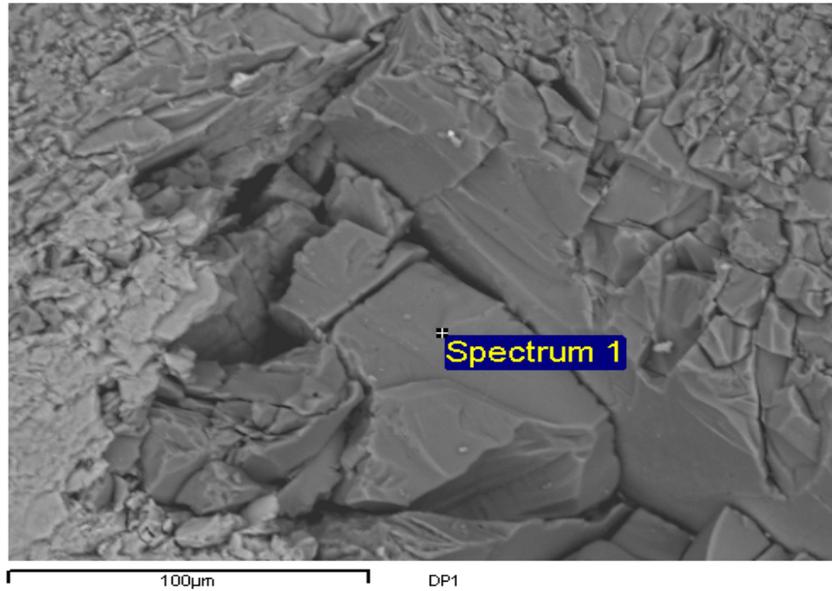


Figure 6.16: SEM micrograph of mixture 7

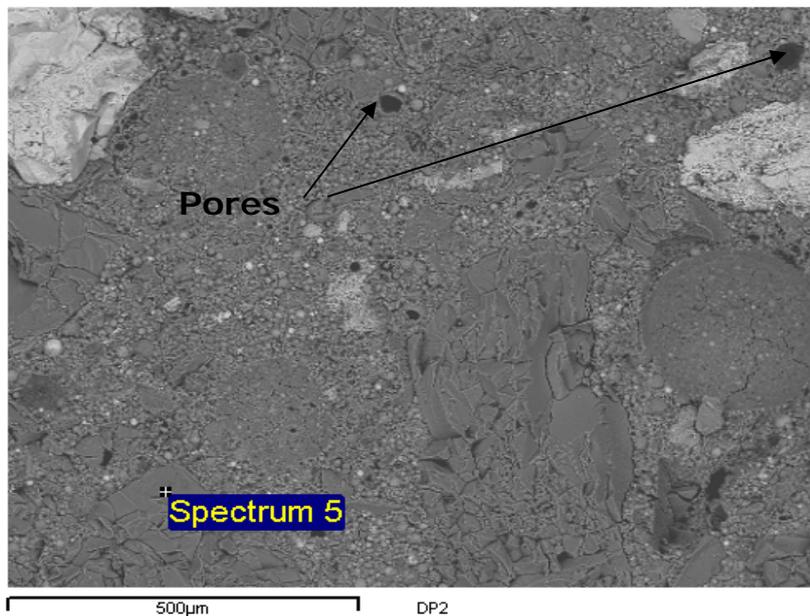


Figure 6.18: SEM micrograph of mixture 8

A SEM micrograph was also taken for mixture 8. As figure 6.18 illustrates, a porous portion of the specimen was investigated. Considering the high compressive strength of mix 8, the results of the SEM test showed that an even

higher compressive strength can be obtained by enhancing the density of red sand geopolymer concrete.

## 7. CONCLUSIONS

- Red sand is an ultra-fine highly absorptive fine aggregate. The high surface area and crushed shape of its particles are the main factors resulting in the low workability of geopolymer concrete.
- The moisture content of red sand prior to use has a great influence on the workability of geopolymer concrete mixture: in order to achieve the desired workability, red sand must be in SSD condition.
- The concentration of a sodium hydroxide solution is a governing factor affecting the strength of red sand geopolymer concrete: 14 molar sodium hydroxide proved to increase the compressive strength of a mixture up to 67% more than 8 molar sodium hydroxide.
- The preset period (the delay time between casting and curing of specimens), up to two days, has been shown increase the strength of red sand geopolymer concrete.
- Dry cured specimens have been found to yield higher compressive strength than samples cured by steam.
- Curing at elevated temperature has been shown to have advantageous effects on strength of red sand geopolymer concrete. Curing at 60 °C is recommended as higher temperatures have not significantly increased the strength.

- Extra water present in the mixture significantly decreases the strength of red sand geopolymer concrete.
- Previous studies have shown that super plasticizer can increase the workability of the mixture; however, in this research it was observed that extra super plasticizer has a negative effect on compressive strength of red sand geopolymer concrete. Therefore, using super plasticizer up to 1.5% of fly ash in the mixtures is recommended.
- The inclusion of red sand has been found to reduce the compressive strength of geopolymer concrete. The most effective method to maintain high compressive strength is altering the aggregate-to-fly ash ratio. Once this was changed, a red sand geopolymer concrete mixture was achieved compatible with relevant standards and comparable to an identical mixture containing natural sand.
- The indirect tensile strength, flexural strength and modulus of elasticity and Poisson's ratio of red sand geopolymer concrete have been found to be higher or comparable to natural sand geopolymer concrete.
- The Apparent Volume of Permeable Voids (AVPV) was measured in red sand geopolymer concrete and results revealed that red sand geopolymer concrete falls into the category of "good" or "excellent" according to the criteria suggested by Vicroads.
- The drying shrinkage of red sand geopolymer concrete has been found to be considerably below values suggested by Standards Australia for concrete structures.

- SEM micrographs showed that a geopolymer concrete mixture comprises well-compacted areas as well as voids and porous areas. Cracks were also observed on the surface of laboratory specimens. Therefore, it is concluded that compaction of red sand geopolymer concrete is a governing factor affecting the microstructure of samples.
- Rapid hardening, experienced throughout the project, is believed to be the result of various parameters. The cold temperature, free water in the mix and conditions of alkaline liquids were identified as major contributing factors.

## **8. RECOMMENDATIONS**

- As curing of red sand geopolymer concrete is carried out at high temperatures, it is recommended that a dedicated series of tests be conducted to investigate the new methods of curing at ambient temperatures.
- It is recommended that red sand geopolymer concrete be made using different alkali liquids such as various combinations of potassium hydroxide and potassium silicate solutions.
- It is recommended that different types of coarse aggregate be used to produce geopolymer concrete with red sand.
- It is recommended that the interface between red sand particles and surrounding paste is investigated thoroughly in terms of microstructure and bond between the particles and the binder.
- It is recommended that the resistance of red sand to abrasion is made the subject of a separate research.
- Throughout the project, the rapid hardening of geopolymer concrete mixtures was experienced occasionally. An extensive series of tests was conducted to find the cause of the hardening; however, it is believed that rapid hardening is the result of a combination of parameters. Therefore, it is recommended that a dedicated series of experiments is conducted to investigate the factors affecting the setting time of geopolymer concrete.

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**APPENDIX A: DETAILS OF MIXTURES****Details of Mixtures**

Mix No.	Aggregate (kg)					Sodium Hydroxide		Sodium Silicate (kg)	Water (kg)	Super Plasticizer (kg)
	10mm	7mm	Natural sand	Red sand	Fly Ash	Quantity(kg)	Molarity			
<b>1</b>	548	638	638	0	403	41	14M	103	20.8	6.1
<b>2</b>	548	638	0	638	403	41	14M	103	20.8	6.0
<b>3</b>	548	638	319	319	403	41	14M	103	20.8	6.0
<b>4</b>	548	638	0	638	403	41	14M	103	20.8	12.0
<b>5</b>	548	638	0	638	403	41	14M	103	52.0	6.0
<b>6</b>	548	638	160	478	403	41	14M	103	20.8	6.0
<b>7</b>	628	628	0	538	425	44	14M	109	20.8	6.3
<b>8</b>	620	620	0	531	442	46	14M	114	21.4	6.6
<b>9</b>	611	611	0	524	467	48	14M	120	22.7	7.0
<b>10</b>	548	638	638	0	403	41	8M	103	20.8	6.0
<b>11</b>	628	628	0	538	425	44	8M	109	20.8	6.3
<b>12</b>	620	620	0	531	442	46	8M	114	21.4	6.6

**Calculating water-to-solid ratio for the mixtures**

Mixture No.	Sodium Hydroxide		Sodium Silicate		Added water	Fly Ash	water/solid ratio
	water	solids	water	solid			
1	24.44	16.56	57.577	45.42	20.8	403	0.22
2	24.44	16.56	57.577	45.42	20.8	403	0.22
3	24.44	16.56	57.58	45.42	20.8	403	0.22
4	24.44	16.56	57.58	45.42	20.8	403	0.22
5	24.44	16.56	57.58	45.42	52.0	403	0.29
6	24.44	16.56	57.58	45.42	20.8	403	0.22
7	26.33	17.85	60.93	48.07	20.8	425	0.22
8	27.42	18.58	63.59	50.17	21.4	442	0.22
9	28.61	19.39	67.08	52.92	22.7	467	0.22
10	30.26	10.74	57.71	45.53	20.8	403	0.24
11	32.61	11.58	60.93	48.07	20.8	425	0.24
12	33.95	12.05	63.5902	50.17	21.4	442	0.24

**Geopolymer Constituents: Molar Concentrations**

Mix No.	Sodium Hydroxide			Sodium Silicate		Added water	Fly Ash	H2O/Na2O
	Flakes		Water	H2O	Na2O	H2O	Na2O	
	H2O	Na2O	H2O					
1	207.05	207.05	1357.56	3198.72	244.19	1153.46	24.04	12.45
2	207.05	207.05	1357.56	3198.72	244.21	1155.56	24.05	12.45
3	207.05	207.05	1357.56	3198.72	244.21	1155.56	24.05	12.45
4	207.05	207.05	1357.56	3198.72	244.21	1155.56	24.05	12.45
5	207.05	207.05	1357.56	3198.72	244.21	2888.89	24.05	16.10
6	207.05	207.05	1357.56	3198.72	244.21	1155.56	24.05	12.45
7	223.13	223.13	1462.99	3385.06	258.44	1155.56	25.34	12.28
8	232.30	232.30	1523.11	3532.79	269.71	1190.07	26.40	12.26
9	242.40	242.40	1589.33	3726.67	284.52	1262.88	27.85	12.30
10	134.28	134.28	1681.00	3206.33	244.79	1153.46	24.04	15.32
11	144.70	144.70	1811.56	3385.06	258.44	1155.56	25.34	15.16
12	150.65	150.65	1886.00	3532.79	269.71	1190.07	26.40	15.13

## APPENDIX B: TEST RESULTS

### Compressive Strength (KN)

#### Mixture 1

<b>3 Day</b>	<b>7 Day</b>	<b>14 Day</b>	<b>28 Day</b>	<b>56 Day</b>
189.6	226.2	226.1	240	260.3
205.4	203.8	206	246.3	247.1
191.7	232.2	212.2	234	242

#### Mixture 2

<b>3 Day</b>	<b>7 Day</b>	<b>14 Day</b>	<b>28 Day</b>	<b>56 Day</b>
163.7	201.7	176.8	242.3	223.1
191.8	167.2	222.2	224.6	212.6
89.9	209.6	179.4	213.3	215.3

**Mixture 3**

<b>3 Day</b>	<b>7 Day</b>	<b>14 Day</b>	<b>28 Day</b>	<b>56 Day</b>
87.2	159	186.7	236.8	255
91.7	172.5	173.4	219.9	265.9
130.8	166.1	208.3	250.5	242.2

**Mixture 4**

<b>3 Day</b>	<b>7 Day</b>	<b>14 Day</b>	<b>28 Day</b>	<b>56 Day</b>
223.8	231.5	251.6	237.9	258.7
210.6	218.2	227.7	247.8	264
226	211	243.3	257.2	261

**Mixture 5**

<b>3 Day</b>	<b>7 Day</b>	<b>14 Day</b>	<b>28 Day</b>	<b>56 Day</b>
141.4	144.4	144.3	149.4	188.5
134.2	129.6	138.6	165.6	188.4
104	167.9	151.7	157.1	172.7

**Mixture 6**

<b>3 Day</b>	<b>7 Day</b>	<b>14 Day</b>	<b>28 Day</b>	<b>56 Day</b>
78.4	77.7	95.7	92.4	84.8
79.3	84.3	97.3	114.1	100.8
92.2	93.3	101.4	97.3	101

**Mixture 7**

<b>3 Day</b>	<b>7 Day</b>	<b>14 Day</b>	<b>28 Day</b>	<b>56 Day</b>
274.8	292.9	297.4	314.3	314
287.7	289	290	291.8	315
246.3	269.7	273.8	277	312

**Mixture 8**

<b>3 Day</b>	<b>7 Day</b>	<b>14 Day</b>	<b>28 Day</b>	<b>56 Day</b>
302	302	309	345	344
308	308	321	340	350
279.6	304.5	323.3	350	355

**Mixture 9**

<b>3 Day</b>	<b>7 Day</b>	<b>14 Day</b>	<b>28 Day</b>	<b>56 Day</b>
301.2	330.3	334.3	378.4	382.1
304.6	320.3	322.4	384.3	382.3
265.2	334.8	334.3	354.3	345.7

**Mixture 10**

<b>3 Day</b>	<b>7 Day</b>	<b>14 Day</b>	<b>28 Day</b>	<b>56 Day</b>
189.6	226.2	226.1	240	260.3
205.4	203.8	206	246.3	247.1
191.7	232.2	212.2	234	242

**˘ Mixture 11**

<b>3 Day</b>	<b>7 Day</b>	<b>14 Day</b>	<b>28 Day</b>	<b>56 Day</b>
134	164	155	188	188
154	170	149	177	180
124	158	160	182	183

**Mixture 12**

<b>3 Day</b>	<b>7 Day</b>	<b>14 Day</b>	<b>28 Day</b>	<b>56 Day</b>
160	177	178	205	204
176	183	180	190	195
160	190	191	197	200

**Mix 2 (Curing Method test)**

<b>Age</b>	<b>3 Day</b>	<b>7 Day</b>	<b>28 Day</b>	<b>56 Day</b>
<b>Steam cured</b>	<b>105</b>	<b>116</b>	<b>118</b>	<b>127</b>
	<b>100</b>	<b>102</b>	<b>124</b>	<b>138</b>
	<b>107</b>	<b>102</b>	<b>125</b>	<b>126</b>

<b>Age</b>	<b>3 Day</b>	<b>7 Day</b>	<b>28 Day</b>	<b>56 Day</b>
<b>Oven cured</b>	177	185	187	194
	190	176	185	184
	181	197	179	184

**Mix 2 (Curing Temperature test)**

<b>Age</b>	<b>3 Day</b>	<b>7 Day</b>	<b>28 Day</b>
<b>Cured at 100 ° C</b>	235.1	274.5	257.7
	253.6	234.3	261.3
	216.2	271.7	275

<b>Age</b>	<b>3 Day</b>	<b>7 Day</b>	<b>28 Day</b>
<b>Cured at 80 ° C</b>	252	256.5	257.2
	251.4	260.3	250.6
	261.2	248.1	248.3

<b>Age</b>	<b>3 Day</b>	<b>7 Day</b>	<b>28 Day</b>
<b>Cured at 60 ° C</b>	176.9	184.9	186.7
	190.3	175.7	185.2
	181.2	196.8	178.9

<b>Age</b>	<b>3 Day</b>	<b>7 Day</b>	<b>28 Day</b>
<b>1 day Preset period</b>	171.7	165.2	157.2
	164	163.1	134.5
	169.8	196.8	176

<b>Age</b>	<b>3 Day</b>	<b>7 Day</b>	<b>28 Day</b>
<b>2 day Preset period</b>	173	179.6	168.2
	180	182.3	225.8
	159.3	169.8	207.1

<b>Age</b>	<b>3 Day</b>	<b>7 Day</b>	<b>28 Day</b>
<b>No Preset period</b>	135.9	140.6	148
	134.5	124.8	143.2
	140	135.3	155.9

Indirect Tensile Strength  
(KN)

	<b>1st trial</b>	<b>2nd trial</b>	<b>3rd trial</b>
<b>Mix 1</b>	292.5	258.2	260.6
<b>Mix 8</b>	238	248	260

	<b>1st trial</b>	<b>2nd trial</b>	<b>3rd trial</b>
<b>Mix 10</b>	188.8	184	177
<b>Mix 11</b>	167.6	195	168
<b>Mix 12</b>	158.7	180	175

## Flexural Strength (KN)

### 14 Molar mixtures

<b>Mix 1</b>	<b>Mix 2</b>	<b>Mix 3</b>	<b>Mix 4</b>	<b>Mix 7</b>	<b>Mix 8</b>
19.2	12.35	16.3	9.8	15.45	19
21	11.3	16.9	11.6	17.9	19.35
18.2	12.95	14.75	10.8	16.1	20

### 8 Molar mixtures

<b>Mix 10</b>	<b>Mix 11</b>	<b>Mix 12</b>
14	11.3	18.5
13	11.1	17.1
13.6	11.3	17.8

AVPV Data

**8 Molar Mixtures (Weight in grams)**

		<b>Oven dried</b>	<b>SSD</b>	<b>Boiled</b>	<b>Immersed</b>
<b>Mix 10</b>	1	836.1	873.1	874.9	506.5
	2	846.1	882.3	883.9	510.6
	3	892.1	929	931.2	537.4
	4	837.7	874.7	876.4	504.2
<b>Mix11</b>	1	818	856.3	858.2	495.6
	2	836.9	875.7	877.6	509
	3	887.8	928.7	931	543.3
	4	852.6	890.9	892.8	518.1
<b>Mix12</b>	1	845.2	884.3	886	515.5
	2	889.3	930.5	932.6	545.6
	3	858.8	898.2	899.5	521.1
	4	853	891	892.7	519.6

**14 Molar Mixtures (Weight in grams)**

		<b>Oven dried</b>	<b>SSD</b>	<b>Boiled</b>	<b>Immersed</b>
<b>Mix 1</b>	1	871	909.4	914.6	531.1
	2	828.3	863.8	868.8	
	3	778.2	811.8	815.9	476
	4	861.2	898.5	900.9	523.6
<b>Mix2</b>	1	850.5	892.9	896.2	526.2
	2	921	965.8	970.3	570.6
	3	806.1	843.9	849.7	499.6
	4	813.1	853.5	856.3	503.7
<b>Mix7</b>	1	777	817	819	476
	2	817.7	858.3	862.2	502
	3	842.7	883.5	889.5	519.5
	4	896.6	942.1	944.3	552.3
<b>Mix8</b>	1	778.3	819.1	825.4	476.1
	2	841.9	883.7	889.1	517
	3	917.6	964.9	969.4	561
	4	836	878.2	881.1	514

## Shrinkage Test Results

### Mixture 1

**Sample 1**

	<b>3 DAY</b>	<b>7 DAY</b>	<b>14 DAY</b>	<b>28 DAY</b>	<b>90 DAY</b>
<b>1st trial</b>	0.5381	0.5363	0.5363	0.5364	0.5362
<b>2nd trial</b>	0.5378	0.5363	0.5364	0.5364	0.5361
<b>3rd trial</b>	0.5378	0.5364	0.5364	0.5362	0.5363
<b>4th trial</b>	0.5379	0.5363	0.5363	0.5363	0.5362
<b>5th trial</b>	0.5379	0.5364	0.5364		
<b>Reference</b>	0.2024	0.2026	0.2023	0.2022	0.2021

**Sample 2**

	<b>3 DAY</b>	<b>7 DAY</b>	<b>14 DAY</b>	<b>28 DAY</b>	<b>90 DAY</b>
<b>1st trial</b>	0.5175	0.5162	0.5162	0.5163	0.516
<b>2nd trial</b>	0.5171	0.5162	0.516	0.5159	0.5162
<b>3rd trial</b>	0.517	0.5159	0.516	0.5162	0.5161
<b>4th trial</b>	0.5174		0.516	0.516	0.5161
<b>5th trial</b>	0.517		0.5162		0.516
<b>Reference</b>	0.2022	0.2025	0.2023	0.2021	0.2023

**Sample 3**

	<b>3 DAY</b>	<b>7 DAY</b>	<b>14 DAY</b>	<b>28 DAY</b>	<b>90 DAY</b>
<b>1st trial</b>	0.5267	0.525	0.5253	0.5254	0.5253
<b>2nd trial</b>	0.5264	0.5252	0.525	0.5253	0.525
<b>3rd trial</b>	0.5268	0.5254	0.5264	0.5259	0.5252
<b>4th trial</b>	0.5263	0.525	0.526		
<b>5th trial</b>	0.5265	0.525			
<b>Reference</b>	0.2022	0.202	0.2024	0.202	0.2022

## Mixture 6

**Sample 1**

	<b>3 DAY</b>	<b>7 DAY</b>	<b>14 DAY</b>	<b>28 DAY</b>	<b>90 DAY</b>
<b>1st trial</b>	0.2715	0.2713	0.2714	0.2710	0.4025
<b>2nd trial</b>	0.2717	0.2713	0.2715	0.2713	0.4026
<b>3rd trial</b>	0.2717	0.2713	0.2715	0.2713	0.4026
<b>4th trial</b>	0.2715	0.2714	0.2716	0.2714	
<b>5th trial</b>	0.2713	0.2713	0.2713	0.2713	
<b>Reference</b>	0.0710	0.0711	0.0709	0.0710	0.2024

**Sample 2**

	<b>3 DAY</b>	<b>7 DAY</b>	<b>14 DAY</b>	<b>28 DAY</b>	<b>90 DAY</b>
<b>1st trial</b>	0.2723	0.2724	0.2724	0.2717	0.4037
<b>2nd trial</b>	0.2726	0.2723	0.2725	0.2717	0.4037
<b>3rd trial</b>	0.2720	0.2725	0.2724	0.272	0.4038
<b>4th trial</b>	0.2722	0.2726	0.2724	0.2719	
<b>5th trial</b>	0.2721	0.2725	0.2723	0.2720	
<b>Reference</b>	0.0713	0.0712	0.0709	0.0707	0.2024

**Sample 3**

	<b>3 DAY</b>	<b>7 DAY</b>	<b>14 DAY</b>	<b>28 DAY</b>	<b>90 DAY</b>
<b>1st trial</b>	0.2895	0.2896	0.2894	0.2894	0.4209
<b>2nd trial</b>	0.2896	0.2896	0.2896	0.2895	0.4206
<b>3rd trial</b>	0.2896	0.2896	0.2896	0.2891	0.4205
<b>4th trial</b>	0.2896	0.2894	0.2896	0.2892	
<b>5th trial</b>	0.2894	0.2895	0.2894	0.2891	
<b>Reference</b>	0.0712	0.0710	0.0713	0.0708	0.2024

## Mixture 7

**Sample 1**

	<b>3 DAY</b>	<b>7 DAY</b>	<b>14 DAY</b>	<b>28 DAY</b>	<b>90 DAY</b>
<b>1st trial</b>	0.3987	0.3986	0.3988	0.3986	0.3985
<b>2nd trial</b>	0.3985	0.3986	0.3991	0.3986	0.3982
<b>3rd trial</b>	0.3988	0.3989	0.3987	0.3988	0.3988
<b>4th trial</b>	0.3987	0.3987	0.3989		0.3986
<b>5th trial</b>	0.3986	0.3988	0.3990		
<b>Reference</b>	0.0705	0.071	0.0711	0.0708	0.0707

**Sample2**

	<b>3 DAY</b>	<b>7 DAY</b>	<b>14 DAY</b>	<b>28 DAY</b>	<b>90 DAY</b>
<b>1st trial</b>	0.4253	0.425	0.4258	0.4260	0.5568
<b>2nd trial</b>	0.4256	0.4252	0.4256	0.4256	0.557
<b>3rd trial</b>	0.4251	0.4249	0.4259	0.4251	0.5569
<b>4th trial</b>	0.4258	0.4254	0.4254	0.4257	0.5567
<b>5th trial</b>	0.4253	0.4255	0.4255	0.4255	0.5563
<b>Reference</b>	0.0707	0.0709	0.0712	0.0713	0.2022

**Sample 3**

	<b>3 DAY</b>	<b>7 DAY</b>	<b>14 DAY</b>	<b>28 DAY</b>	<b>90 DAY</b>
<b>1st trial</b>	0.4174	0.4173	0.4173	0.4173	0.5485
<b>2nd trial</b>	0.4176	0.4177	0.4178	0.4176	0.5482
<b>3rd trial</b>	0.4174	0.4175	0.4176	0.4177	0.5486
<b>4th trial</b>	0.4175	0.4172			
<b>5th trial</b>					
<b>Reference</b>	0.0708	0.0711	0.0711	0.0713	0.2020

## Mixture 8

**Sample 1**

	<b>3 DAY</b>	<b>7 DAY</b>	<b>14 DAY</b>	<b>28 DAY</b>	<b>90 DAY</b>
<b>1st trial</b>	0.2561	0.256	0.2559	0.2562	0.3875
<b>2nd trial</b>	0.2558	0.256	0.2559	0.2558	0.3871
<b>3rd trial</b>	0.2560	0.256	0.2560	0.256	0.3870
<b>4th trial</b>			0.2558	0.2561	0.3873
<b>5th trial</b>					0.3872
<b>Reference</b>	0.0706	0.0707	0.0707	0.0709	0.2023

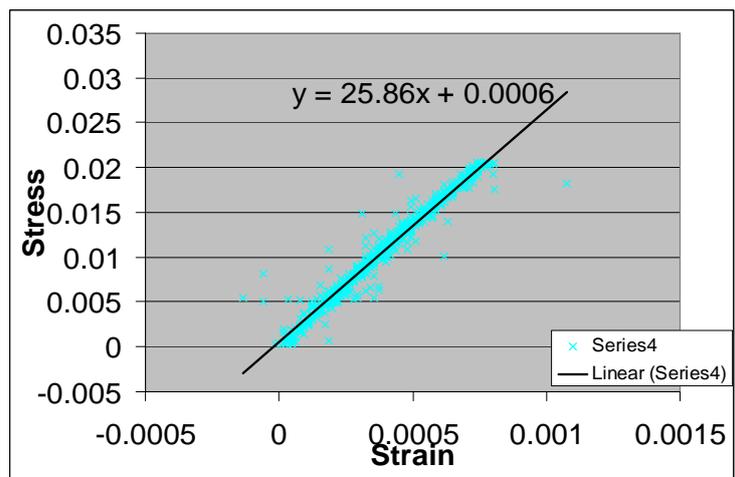
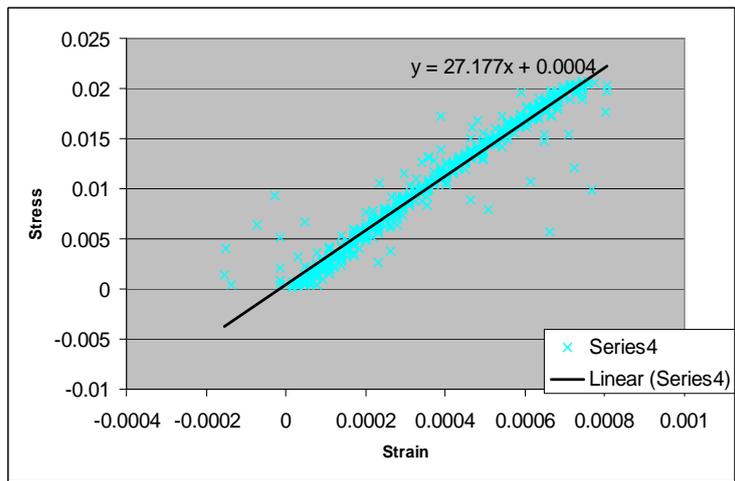
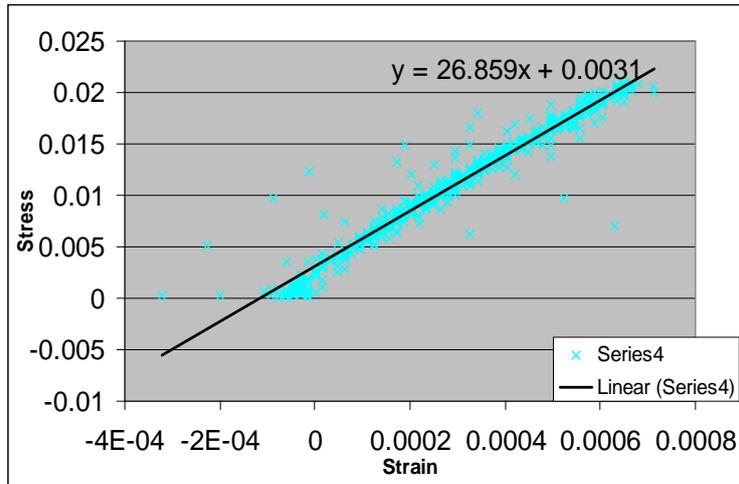
**Sample 2**

	<b>3 DAY</b>	<b>7 DAY</b>	<b>14 DAY</b>	<b>28 DAY</b>	<b>90 DAY</b>
<b>1st trial</b>	0.1557	0.1557	0.1557	0.1555	0.2868
<b>2nd trial</b>	0.1553	0.1557	0.1555	0.1553	0.2868
<b>3rd trial</b>	0.1554	0.1556	0.1554	0.1555	0.2868
<b>4th trial</b>	0.1555		0.1554	0.1554	
<b>5th trial</b>					
<b>Reference</b>	0.0706	0.0709	0.0708	0.0707	0.2023

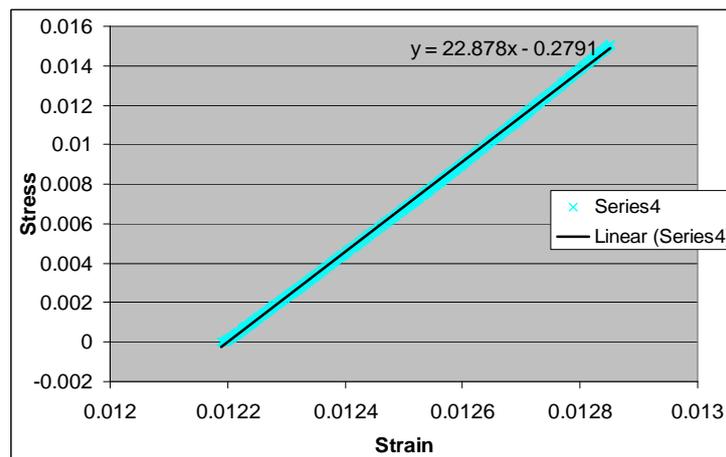
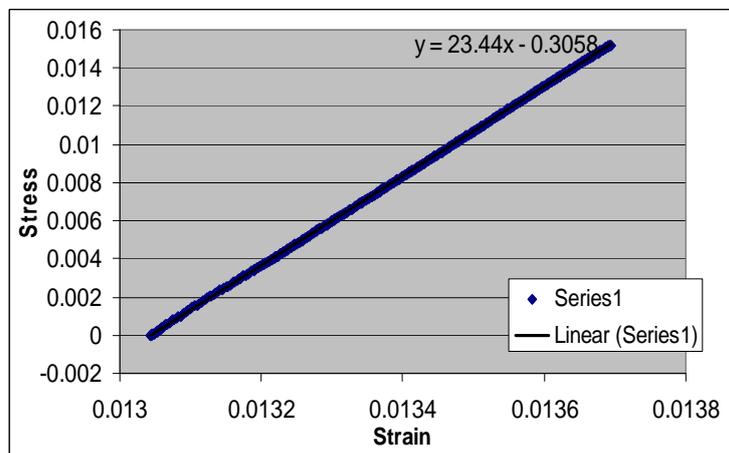
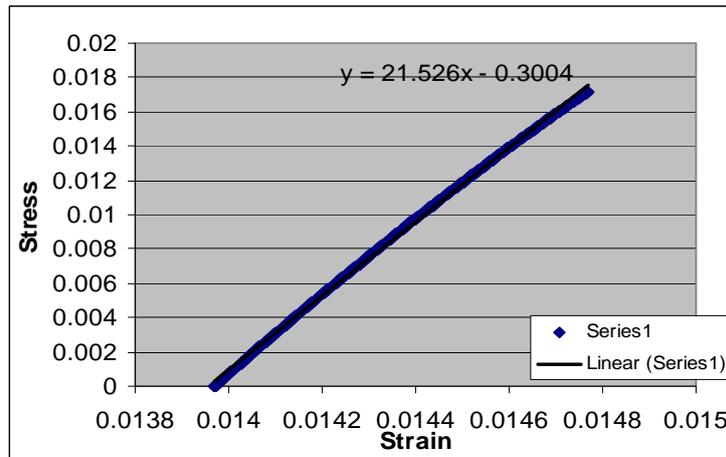
**Sample 3**

	<b>3 DAY</b>	<b>7 DAY</b>	<b>14 DAY</b>	<b>28 DAY</b>	<b>90 DAY</b>
<b>1st trial</b>	0.2157	0.2157	0.2155	0.2155	0.2155
<b>2nd trial</b>	0.2158	0.2156	0.2158	0.2156	0.2154
<b>3rd trial</b>	0.2155	0.2159	0.2156	0.2157	0.2157
<b>4th trial</b>	0.2157				
<b>5th trial</b>	0.2158				
<b>Reference</b>	0.0705	0.0708	0.0708	0.0709	0.0709

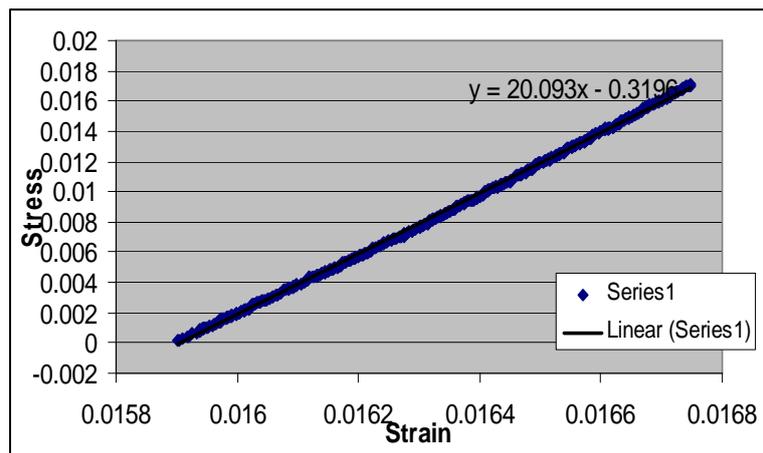
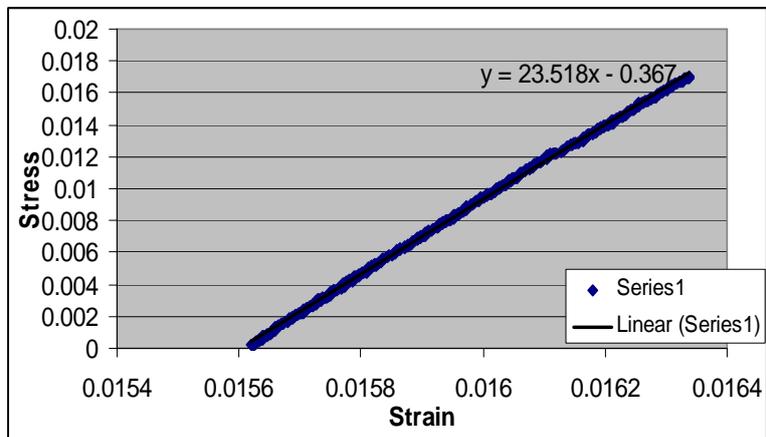
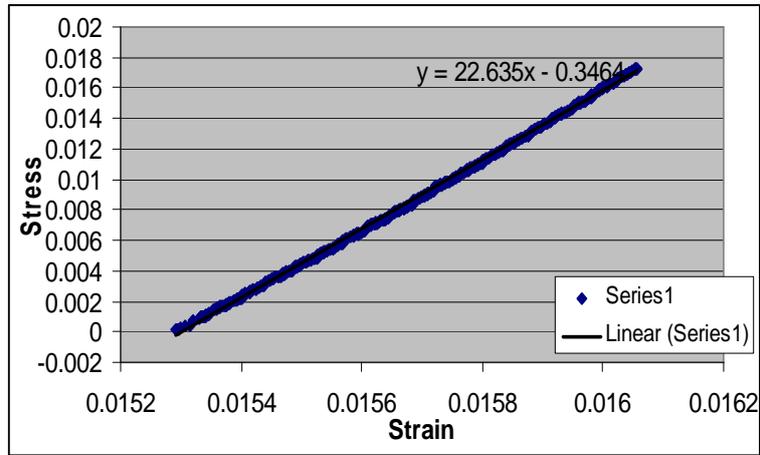
### Mixture 1



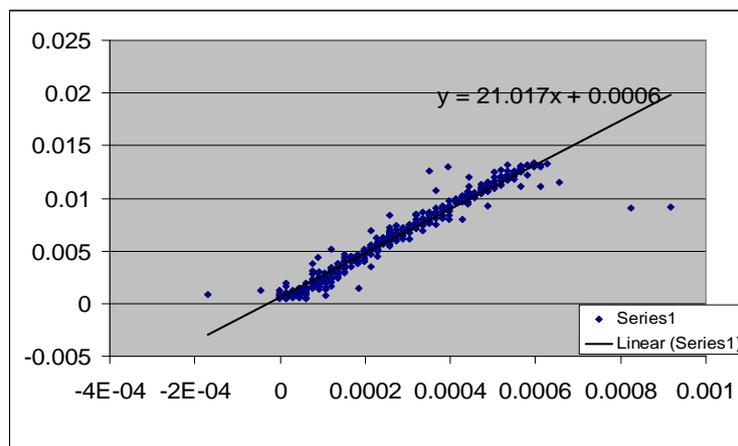
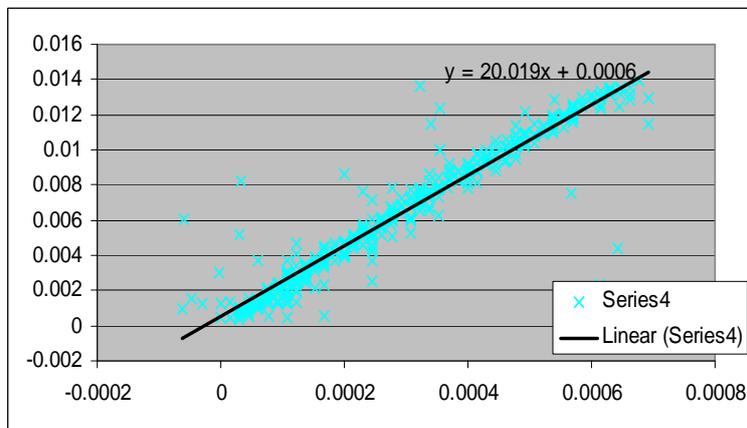
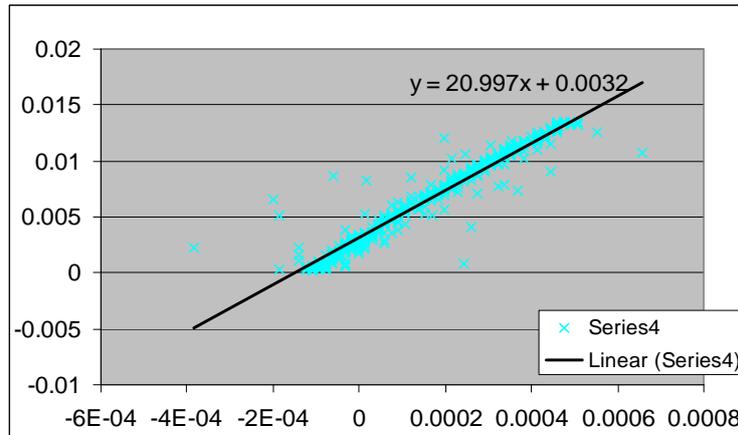
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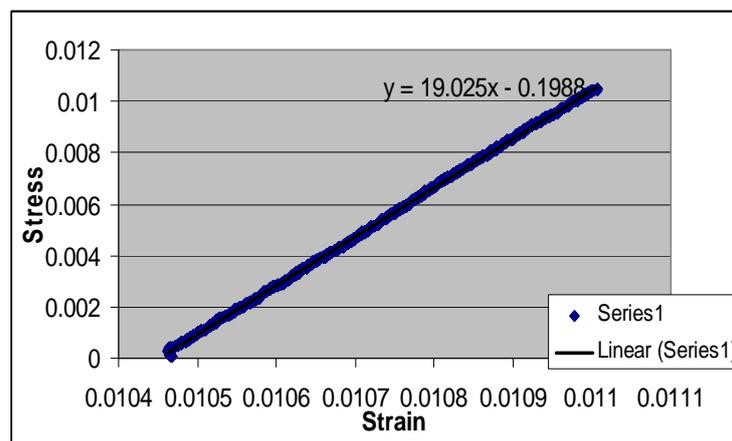
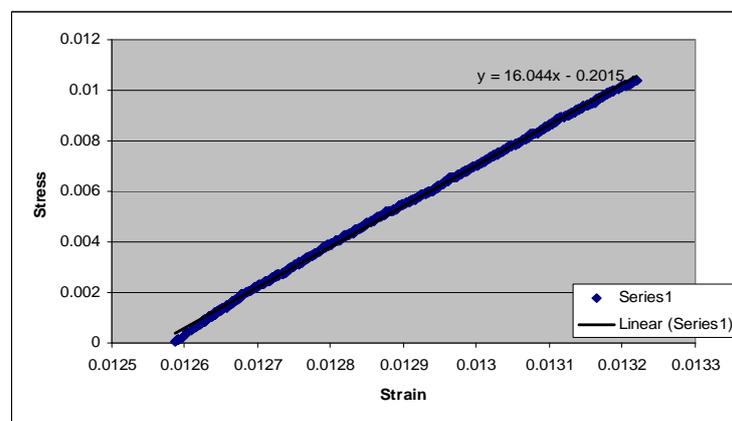
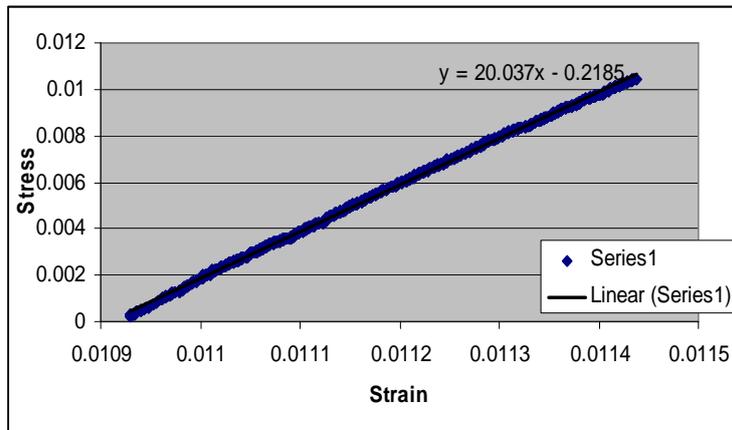
### Mixture 8



### Mixture 10



### Mixture 11



### Mixture 12

