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Synthesis and mechanical properties of cotton fabric reinforced geopolymer composites

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Abstract

Geopolymer composites reinforced with different layers of woven cotton fabric are fabricated using lay-up technique. Mechanical properties, such as flexural strength, flexural modulus, impact strength and fracture toughness of geopolymer composites reinforced with 3.6, 4.5, 6.2 and 8.3 wt% cotton fibres are studied. The fracture surfaces of the composites are also examined using scanning electron microscopy. The results show that all the mechanical properties of the composites are improved by increasing the cotton fibre contents. It is found that the mechanical properties of cotton fabric reinforced geopolymer composites are superior to pure geopolymer matrix.

Keywords: A. Polymer-matrix composites, B. Microstructures, B. Mechanical properties, B. Fracture toughness.

1. Introduction

Geopolymers are inorganic compounds that can be cured and hardened at near-ambient temperatures to form materials that are effectively low-temperature ceramics with typical temperature resistance and strength [1]. In recent years, geopolymers have emerged as an alternative to cements [2,3]. However, despite their many desirable attributes, such as relatively high strength, elastic modulus and low shrinkage, geopolymers suffer from brittle failure like most ceramics. Nevertheless, this limitation can be overcome by the introduction of reinforcing materials, including short fibres or unidirectional long fibres into the geopolymer matrix. For this purpose, various inorganic fibres have been previously used as reinforcement in geopolymer composites [4-7].

However, contemporary concerns over environment and climate change have given rise to an increasing interest in natural materials to produce environmentally friendly composite materials for construction. Natural fibres are very attractive for composite materials as they have several useful characteristics, such as low cost, low density, availability, recyclability, and renewability. In addition, they possess excellent mechanical properties like high degrees of toughness, flexibility, specific modulus and specific strength [8,9].

For these reasons, natural fibres are more attractive to researchers and scientists as an alternative source of reinforcement to develop organic polymer composites. As an illustration, cellulose fibres have been used to reinforce various organic matrices like polyester, vinyl ester, and epoxy matrices [10-13]. However, at present there has been limited published research on the use of natural fibres to reinforce inorganic matrices despite the advantages they offer in terms of low cost, ready availability, low toxicity and good mechanical strength [14]. In a previous report, the authors studied the mechanical properties of short cotton fibre-reinforced geopolymer composites. Results showed that further increases

in short cotton fibre content beyond 0.5 wt% caused a reduction in the mechanical properties due to poor workability which led to formation of voids and fibre agglomerations [15,16] . In a follow-up work by the same authors [17], similar behaviour has been reported in cotton fabric-reinforced geopolymer composites, with the exception that the mechanical properties decreased beyond 2.1 wt% cotton fibres. This decrease was attributed to the poor bond between the cotton fibre and matrix due to insufficient amount of geopolymer matrix in the composites containing more than 2.1% wt% cotton fibres.

In the present study, woven cotton fabrics (CF) have been impregnated (wet out) with geopolymer paste, stacked, and compressed by a roller to force the paste to penetrate the fabric and to remove most of the trapped air. The aim is to investigate the possibility of applying above technique to manufacture geopolymer composites with cotton fabrics for structural applications, and to examine the basic mechanical properties through experimental testing. Useful results have been gathered for composites with various cotton fibres contents (0, 3.6, 4.5, 6.2 and 8.3 wt%) under the three-point bending test. The results show that the addition of cotton fibres improves the mechanical properties of geopolymer composites such as flexural strength, flexural modulus, impact strength and fracture toughness. Synchrotron radiation diffraction (SRD) and scanning electron microscopy (SEM) are used to characterise the phase composition, microstructure, and failure mechanisms of woven cotton fibre reinforced geopolymer composites.

2. Experimental investigation

2.1. Materials

Cotton fabric (CF) of 30 cm × 7.5 cm was used to reinforce the geopolymer. This fabric is made up of yarns with a density of 1.54 g/cm^3, tensile strength of 400 MPa, and Young's modulus of 4.8 GPa. Low calcium fly-ash (ASTM class F), collected from Collie power station in Western Australia, was used as the source material of the geopolymer matrix. The chemical composition of fly-ash (FA) is shown in Table 1. The alkaline activator for geopolymerisation was a combination of sodium hydroxide solution and sodium silicate grade D solution. Sodium hydroxide flakes with 98% purity were used to prepare the solution. The chemical composition of sodium silicate used was 14.7% Na_2O, 29.4% SiO_2 and 55.9% water by mass.

2.2. Preparation of geopolymer composites

To cast the composite samples, five wooden moulds with open tops were prepared and greased to avoid the samples sticking during de-moulding. The fabrics were pre-dried for 60 minutes at 70 °C. An 8 molar concentration of sodium hydroxide solution was prepared and combined with the sodium silicate solution one day before mixing. The fly ash and alkaline solution were mixed in a Hobart mixer to form a homogeneous paste. A thin layer of geopolymer paste was first spread in the wooden mould and the first layer of woven fabric was carefully laid on that layer. The fabric was then fully impregnated (wet out) with geopolymer paste by a roller and the process repeated for the desired number of cotton fibre layers. Each specimen contained different layers of cotton fabric (see Table 2) with final layer being geopolymer paste. The alkaline solution to fly ash ratio was fixed at 0.35 whereas the ratio of sodium silicate solution to sodium hydroxide solution was maintained at 2.5. The composite specimens were placed on a vibration table in order to ensure better penetration of the matrix between the fabric openings and to remove the entrapped air voids. Then, the composite specimens were pressed under 25kg load for 3 hours. Subsequently, the specimens

were covered with plastic film and cured at 80 °C in an oven for 24 hours. The samples were de-moulded and kept in room condition for 28 days before testing. The mechanical properties of unreinforced geopolymer used in this study are measured and used for comparison purpose. Typically, the compressive strength of the geopolymer paste is 21 MPa with density of 1.9 g/cm^3.

2.3. Characterisation

2.3.1. Synchrotron Radiation Diffraction (SRD)

The Powder Diffraction beamline at the Australian Synchrotron was used to collect the diffraction patterns of fly-ash and the geopolymer composites. The diffraction pattern of each sample was collected using a wavelength of 0.825 Å over the 2θ range of 5° - 45°.

2.3.2. Scanning Electron Microscopy (SEM)

The microstructures of geopolymer composites were examined using a Zeiss Evo 40XVP scanning electron microscope. The specimens were mounted on aluminium stubs using carbon tape and then coated with a thin layer of platinum to prevent charging before the observation.

2.4. Mechanical properties

2.4.1. Flexural strength

Rectangular bars with a length of 60 mm were cut from the fully cured samples and subjected to three-point bend tests to evaluate their flexural strength. The tests were performed in a LLOYD Material Testing Machine (50 kN capacity) with a displacement rate of 0.5 mm/minute. Five specimens of each composition were tested. The flexural strength (σ_F) was determined using the following equation [9]:

$$\sigma_F = \frac{3}{2}\frac{P_m S}{BW^2} \quad (1)$$

where P_m is the maximum load at crack extension, S is the span of the sample, B is the specimen width and W is the specimen thickness (depth).

Values of the flexural modulus (E_F) were computed using the initial slope of the load–displacement curve, $\Delta P / \Delta X$, using the following formula [9]:

$$E_F = \frac{S^3}{4BW^3}\left(\frac{\Delta P}{\Delta X}\right) \quad (2)$$

2.4.2. Impact strength

The impact strength was determined using a Zwick Charpy impact tester with a 7.5 J pendulum hammer. Five bars of 60 mm long were utilised. The impact strength (σ_i) was calculated using the following equation [18]:

$$\sigma_i = \frac{E}{A} \quad (3)$$

where E is the impact energy required to break a sample with a ligament of area A.

2.4.3. Fracture toughness

Rectangular bars of 60 mm long and cross-sectional dimension of 20 x 20 mm were used in the fracture toughness measurements. A crack with a length to thickness (depth) (a/W) ratio of 0.4 was introduced into the specimen using a 0.4 mm diamond blade to evaluate the fracture toughness. The fracture toughness K_{IC} was calculated using the following equation:

$$K_{IC} = \frac{p_m S}{BW^{3/2}} f\left(\frac{a}{W}\right) \quad (4a)$$

where P_m is the maximum load at crack extension, S is the span of the sample, B is the specimen width, W is the specimen thickness (depth), a is the crack length where $f(a/W)$ is the polynomial geometrical correction factor given by [9]:

$$f\left(\frac{a}{W}\right) = \frac{3(a/W)^{1/2}[1.99 - (a/W)(1 - a/W) \times (2.15 - 3.93a/W + 2.7a^2/W^2)]}{2(1 + 2a/W)(1 - a/W)^{3/2}} \quad (4b)$$

3. Results and discussion

3.1. Synchrotron radiation diffraction

The synchrotron radiation diffraction (SRD) patterns of class F fly ash, cotton fibres, and geopolymer composites containing 0, 3.6, 4.5, 6.2 and 8.3 wt% of cotton fibres, are shown in Fig.1. The crystalline phases present were indexed using Powder Diffraction Files (PDFs) from the Inorganic Crystal Structure Database (ICSD). The diffraction pattern of cotton fibres shows typical characteristic peaks, indicating the presence of cellulose. The SRD pattern of fly ash shows that the major crystalline phases are quartz, mullite and hematite. These crystalline phases are the main component of fly ash. When comparing the SRD pattern of the original fly ash with those of the hardened geopolymeric materials shown in Fig. 1, it is seen that the crystalline phases of quartz, mullite and hematite remain unchanged and have not been visibly altered by the activation reaction. This finding confirms that the crystalline phases are not reactive or involved in geopolymerisation, but simply present as inactive fillers in geopolymer network [18-21]. In this case**,** however, amorphous aluminosilicate phases are more reactive and dissolvable in alkaline solution during the formation of a geopolymer [22-24].

3.2. Mechanical properties

3.2.1. Flexural strength and modulus

Flexural tests are often used to characterise the mechanical properties of layered materials since they provide a simple means of determining the bending response. This provides useful information on the performance of layered fabric-based composites [25]. The test results show that the flexural strength of cotton fabric reinforced composite increases as wt% of cotton fibres increase (see Fig. 2). The composite containing 8.3 wt% woven cotton fibres exhibited the highest flexural strength among all composites. The flexural strength of the composites increased from 8.2 MPa to 31.7 MPa compared to pure geopolymer. This indicates that increasing the number of woven cotton fibres leads to considerable improvement in flexural strength in the composite. This finding can be justified from the fact that the flexural strength is controlled by the number of reinforcement layers. The lower weight of cotton fabrics allows multiple layers of fabric in the composite, to resist the shear failure and contribute in sustaining the applied load to the composites. This permits greater stress transfer between the matrix and the cotton fibres, resulting in improved flexural strength [26].

In previous studies [15,16], the authors studied the flexural behaviour of short cotton fibre reinforced geopolymer composites and observed minimum improvement in the flexural strength over that of the unreinforced specimens due to poor dispersion of short cotton fibres in the matrix. In fact, agglomerations of cotton fibres were noticed which degraded the interfacial adhesion between the fibre and the matrix as shown in Fig. 6. In present study, the utilisation of continuous fibres as reinforcement for geopolymer composites has shown better mechanical properties than short cotton fibres, owing to their ability to effectively bridge the cracks due to their alignment in the direction of tension resulted in greater stress transfer at the interface of the composites. The improved performance of cotton fabric-geopolymer composite can be explained by observing the SEM microscope images as seen in Fig. 7F.

This shows good penetration of geopolymer paste into the filament of the cotton bundle making up the fabric, providing improved bonding between the fabric and the geopolymer matrix and leading to an improvement in flexural strength.

The flexural modulus of geopolymer composites is shown in Fig. 3 and indicates similar trends to flexural strength values. In reality, the addition of woven cotton fibres in the geopolymer matrix increases the flexural modulus over plain geopolymer matrix. The flexural modulus is a measure of resistance to deformation of the composite in bending. It was observed that none of the specimens are completely broken at peak load. This could be due to the crack bridging by long continuous fibres, which makes their flexural modulus higher than un-reinforced geopolymer. Such fibres are able to withstand a higher load and are capable of undergoing multiple cracks throughout the loading process, thus prevent brittle failure of the specimens. Similar results have been reported by Low et al. [9] when testing the mechanical properties of cellulose fibre-reinforced epoxy laminates using the three point bending test. They report an increase in both flexural strength and modulus as the fibre contents increase.

The increase in woven cotton fibre content was exceptionally useful in terms of improving the flexural strength and modulus of this inorganic polymer matrix.

3.2.2. Impact strength

Impact strength is an essential dynamic property of engineering material that gives an indication of its resistance against sudden impact. The impact strength of fibre reinforced polymer is governed by the matrix-fibre interfacial bonding, and properties of matrix and fibres. When the composites undergo a sudden force, the impact energy is dissipated by the

combination of fibre pull outs, fibre fracture and matrix deformation [27, 28]. The experimental results presented in Fig. 4 indicate that the impact strength of the composites increases as cotton fibre content increases. The impact strength of the neat geopolymer increased from 2.1 to15.6 kJ/m^2 after the addition of 8.3 wt% woven cotton fabric to the geopolymer composite. This significant enhancement in impact strength could be the result of pressing the top surface of the geopolymer composites, which expelled the trapped air from the sample and forced the geopolymer paste into the voids and pore spaces. As a result, the bonding between the fabrics and the matrix is enhanced, and results in increased impact strength.

This improvement in impact strength may also be attributed to the fibrillation of cotton layers that creates branches in the fibre (see Fig. 7G), resulting in the formation of micro fibrils which increase the fibre specific surface area [29, 30]. This results in an enhanced fibre-matrix interaction, and a strong bonding between the micro fibrils and the geopolymer matrix. Therefore, better stress transfer from the matrix to the micro fibrils results in increased fibre-matrix bonding. Similar remarkable improvements in impact strength was reported by Graupner [31]. Based in his observation, the addition of cotton fibre increased the impact strength of pure poly (lactic acid) (PLA) matrix. He concluded that the increase was due to greater elongation of cotton fibres at break. Fibres containing much cellulose generally have high elongation at break values. Cotton has a cellulose content of about 88–96%. Elongation at break and impact strength are directly correlated. The high elongation at break of cotton fibres increased the elongation at break in the composites, leading to higher impact strength.

3.2.3. Fracture toughness

Generally, crack deflection, debonding and bridging of fibres slows down the crack propagation in fibre reinforced composites and increase the fracture energy [32-37].

Fig. 5 shows the influence of cotton fabric content on the fracture toughness of the composites, the composite containing higher cotton fibre content exhibits higher fracture toughness. The greatest improvement in fracture toughness (from about 0.6 $MPa.m^{1/2}$ in the unreinforced matrix to about 1.8 $MPa.m^{1/2}$) was obtained with 8.3 wt% cotton fibre reinforcement. This extraordinary enhancement is due to the unique properties of woven cotton fibre to resist fracture resulted in increased energy dissipation from crack-deflection at fibre–matrix interface, fibre-debonding, fibre-bridging, fibre pull-out and fibre-fracture. The high values of fracture toughness obtained in geopolymer composites with woven cotton fibres were due to better interaction between fibre and matrix as shown in Fig. 7. The improved interfacial adhesion enabled higher stress transfer between the fibres and matrix and reduced the chance of fibre de-bonding. Accordingly, the load required to break the sample increases when the content of cotton fibre is increased. Therefore, the fracture toughness of geopolymer composites increases with increasing wt% of cotton fibres.

Fig. 7E demonstrates selected scanning electron micrographs of fracture surface of geopolymer composite and explains the fracture toughness behaviour. It can be seen from Fig. 7E that small pieces of geopolymer paste were attached to the fibre surface of cotton fibre/geopolymer composites. Hence, retention of the matrix on the fibre surfaces shows the good adhesion between cotton fibres and geopolymer matrix. Additionally, it was observed that geopolymer composites with woven cotton fibres did not completely break into two pieces due to close spacing of woven cotton fabric which lead to fibres bridging the cracks

and enhancing the crack propagation resistance. The tortuous pathway of the crack propagation indicates that high energy is absorbed by the cotton fibre layers (see Fig. 8).

3.3. Microstructure characteristics

The fracture surfaces of the woven cotton fibre reinforced geopolymer composites have been studied under SEM and are shown in Fig. 7. Generally, fibre pullout, fibre-debonding, fibre breakage and matrix fracture are observed after the fracture test of all composites. In fact, such toughening mechanisms increased the fracture properties of samples reinforced with woven cotton fibres. The effect of fibre content on the fracture surface is clearly seen in Figs. 7A and B. Composites filled with lower fibre content (3.6 and 4.5) wt% show an increase in matrix-rich regions compared to composites filled with higher fibre content. An increase in matrix rich regions means that the matrix is not reinforced by enough fibres. Therefore, there are insufficient fibres to transfer the load from the matrix [37]. Due to this reason, the geopolymer composites with low fibre content exhibited low fracture toughness and mechanical properties. However, Figs. 7C and D illustrate the fracture surfaces of the geopolymer composites with higher fibre content. It can be seen that there are higher fibre-rich regions of composites filled with 6.2 and 8.3 wt% cotton fibres. An increase in fibre-rich regions means greater stress-transfer from the matrix to the cotton fibres thereby resulting in the improvement of mechanical properties.

Therefore, these observations indicate that the woven cotton fabrics can be used as potential material to reinforce geopolymer composites due to their good mechanical properties.

4. Conclusions

This study on mechanical properties of cotton fabric-reinforced geopolymer composite shows that the presence of cotton fabric layers in the geopolymer paste significantly increases all mechanical properties (flexural strength, flexural modulus, impact strength and fracture toughness) compared to neat geopolymer. This remarkable enhancement is due to the unique properties of cotton fibre in withstanding greater bending and fracture forces than the more brittle geopolymers. SEM micrographs show a number of toughness mechanisms, such as crack bridging, fibre pullout and fibre fracture, and matrix fracture. These toughening mechanisms of cotton fabric-reinforced geopolymer composites are the major factors contributing to the enhanced mechanical properties of samples reinforced with cotton fabric. Cotton fibres appear to be uniquely suited to reinforce the geopolymer composites since they process easily under conventional manufacturing techniques to yield a product with good mechanical properties at low cost. They can be classified as desirable performing composites that offer benefits to engineers, particularly in less developed countries or countries that need low-cost construction materials. Possible applications for cotton fibre-reinforced geopolymer composites include slabs or shingles for siding, certain types of roofing, and some interior uses in the building structure. They may also be used for other applications such as pipes and cooling towers.

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Figure Captions

1. Synchrotron radiation diffraction patterns of (A) cotton fibres (CF), (B) fly-ash , and geopolymer composite with (C) 3.6 wt% CF, (D) 4.5 wt% CF, (E) 6.2 wt% CF and (F) 8.3 wt% CF. [Legend: 1 = mullite (PDF 15–0776), 2 = quartz (PDF 05–0490), 3 = hematite (PDF 13–0534), 4= cellulose (PDF 00–060–1502)].
2. Flexural strength of geopolymer composites as a function of fibre content
3. Flexural modulus of geopolymer composites as a function of fibre content.
4. Impact strength of geopolymer composites as a function of fibre content.
5. Fracture toughness of geopolymer composites as a function of fibre content.
6. SEM showing agglomeration of short fibres in cotton fibre/geopolymer composites loaded with 1.0 wt%.
7. SEM images of the fracture surface for geopolymer composites reinforced with varying content of cotton fibres (A) 3.6, (B) 4.5, (C) 6.2 and (D-E) 8.3wt%. The micrographs of (F) penetration of the geopolymer matrix into cotton fabrics and (G) micro fibrillation of cotton fibers.
8. Optical image of crack propagation in cotton fabric/geopolymer composites loaded with 8.3 wt% fibre.

List of Tables

Table1: Chemical composition of fly ash.

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	LOI
50%	28.25%	13.5%	1.78%	0.89%	0.38%	0.32%	0.46%	1.64%

Table 2: Formulations of samples.

Sample	Fabric layers	Fibre content (wt%)
Composite 0	0	0
Composites 1	5	3.6
Composites 2	10	4.5
Composites 3	20	6.2
Composites 4	40	8.3

Fig. 1

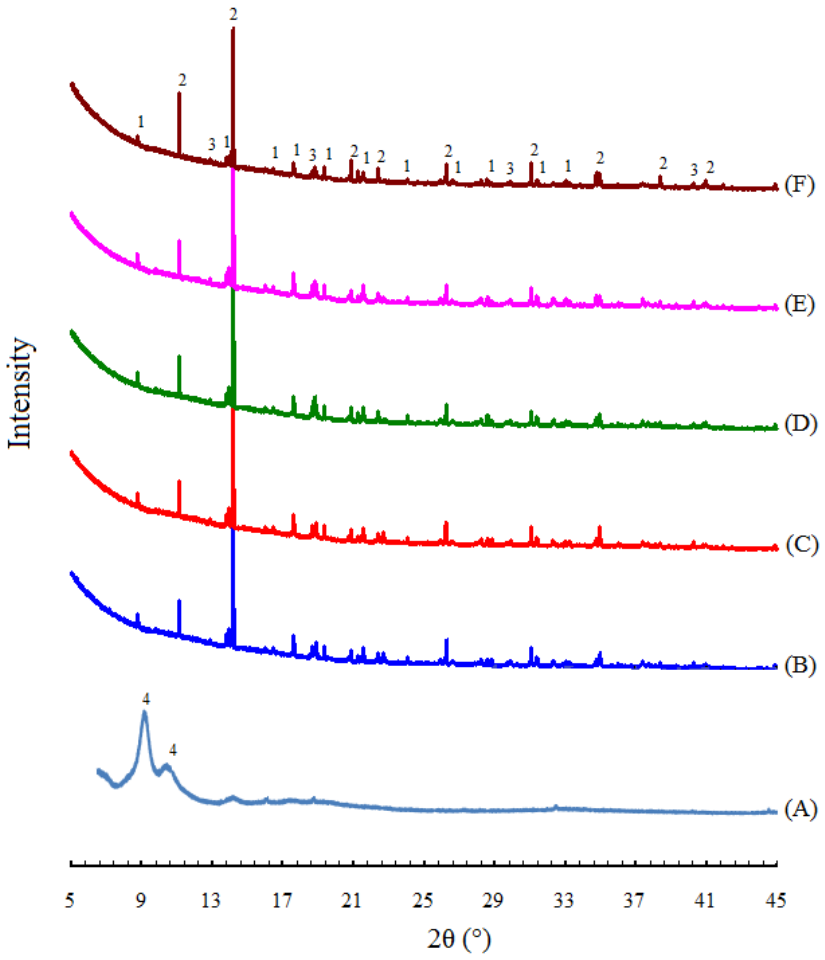


Fig. 2

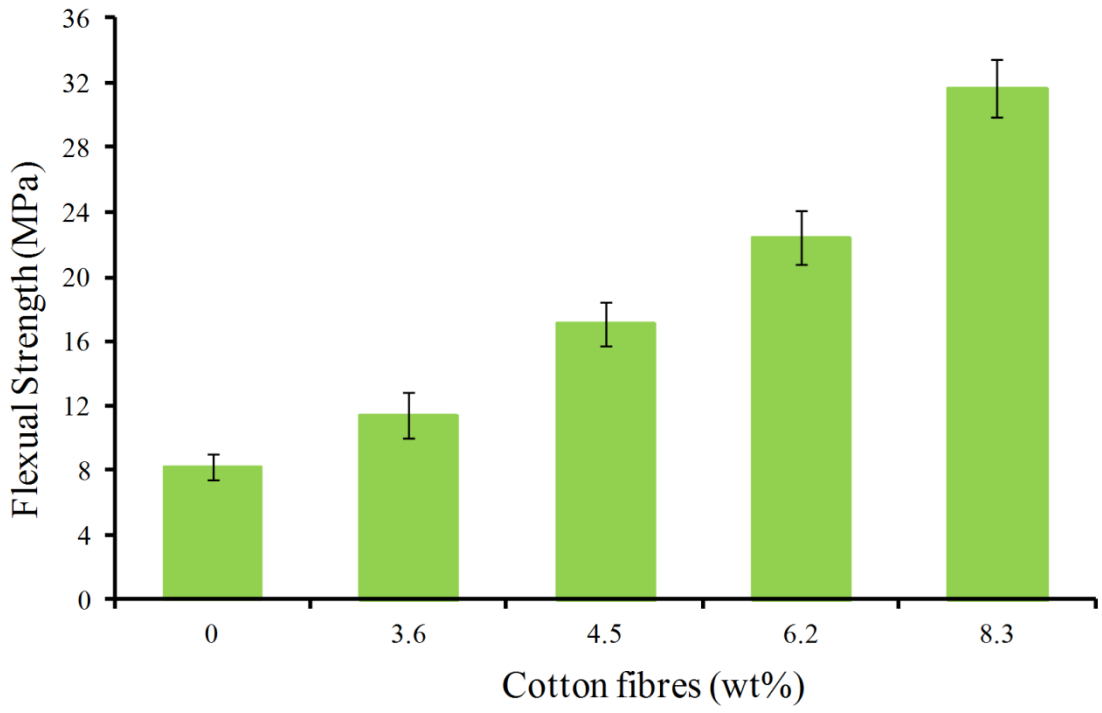

Flexual Strength (MPa)
36
32
28
24
20
16
12
8
4
0
0
3.6
4.5
6.2
8.3
Cotton fibres (wt%)

Fig. 3

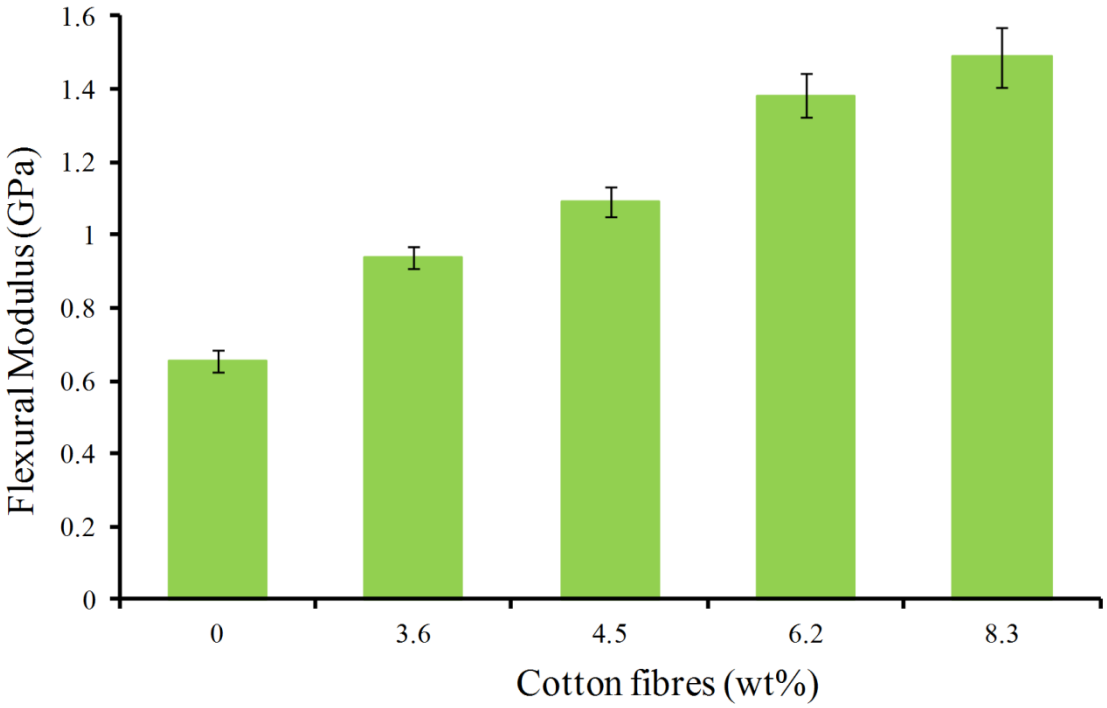

Flexural Modulus (GPa)
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
0
3.6
4.5
6.2
8.3
Cotton fibres (wt%)

Fig. 4

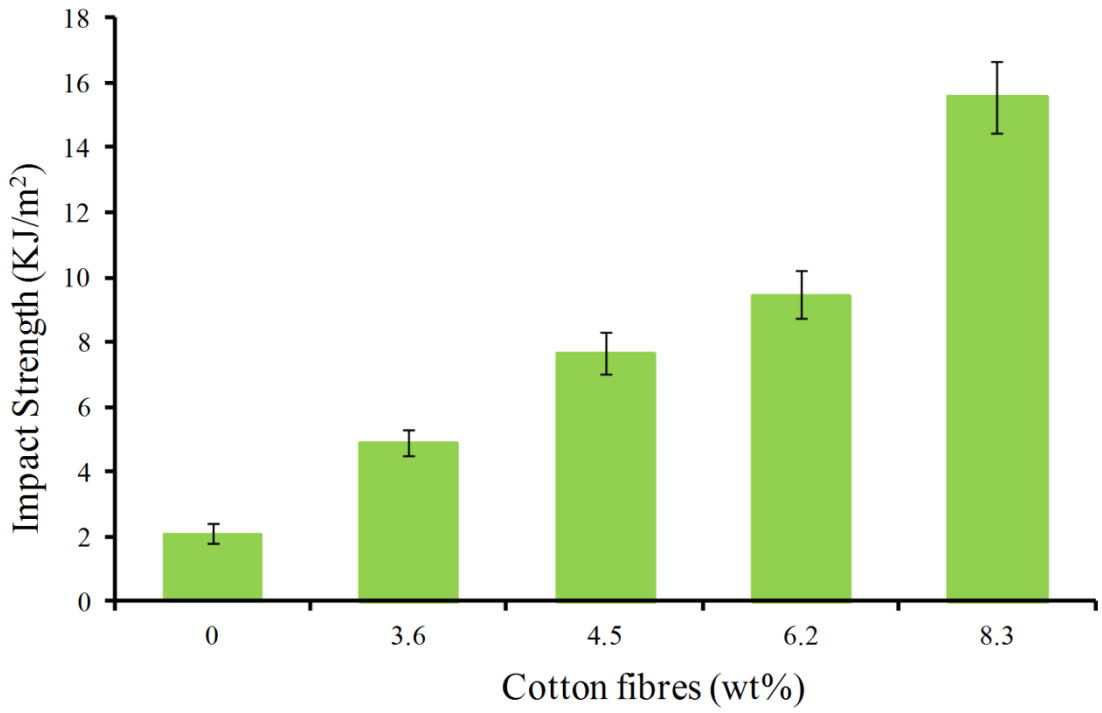

Impact Strength (KJ/m²)
18
16
14
12
10
8
6
4
2
0
0
3.6
4.5
6.2
8.3
Cotton fibres (wt%)

Fig. 5

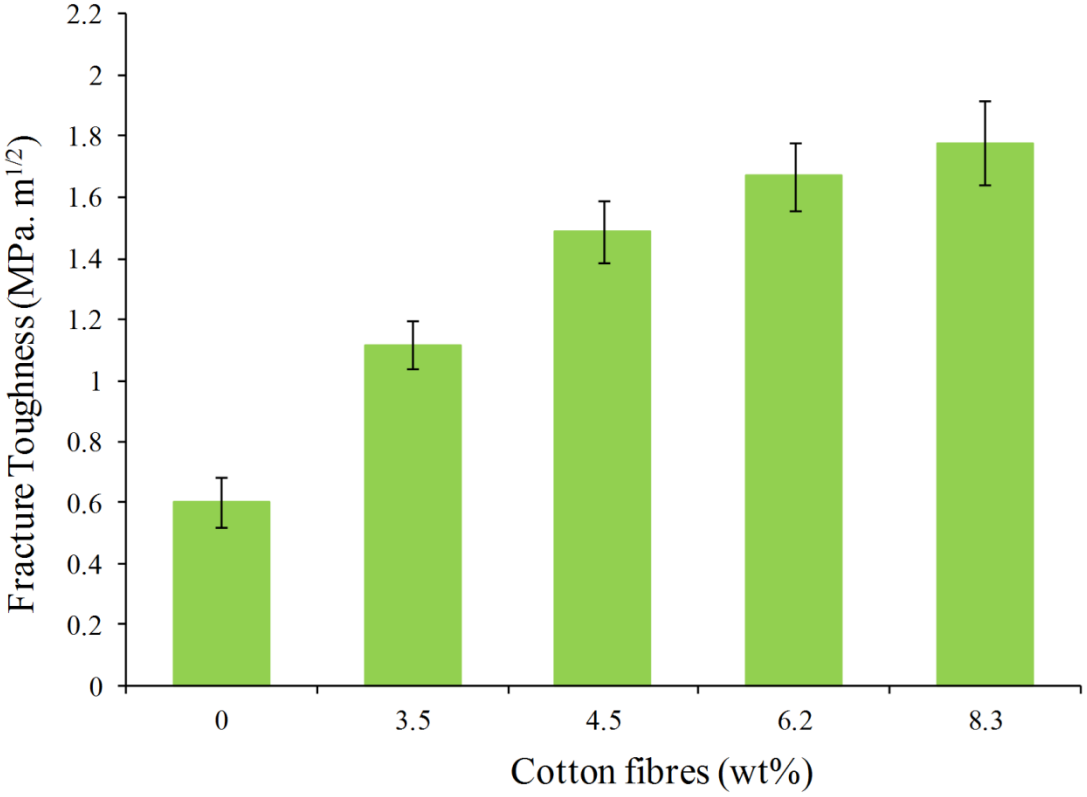

Fracture Toughness (MPa. m^1/2)
2.2
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
0
3.5
4.5
6.2
8.3
Cotton fibres (wt%)

Fig. 6

agglomeration
100 µm
EHT = 15.00 kV
Spot Size = 400
Signal A = CZ BSD
Signal B = SE1
WD = 12.0 mm

Fig. 7A

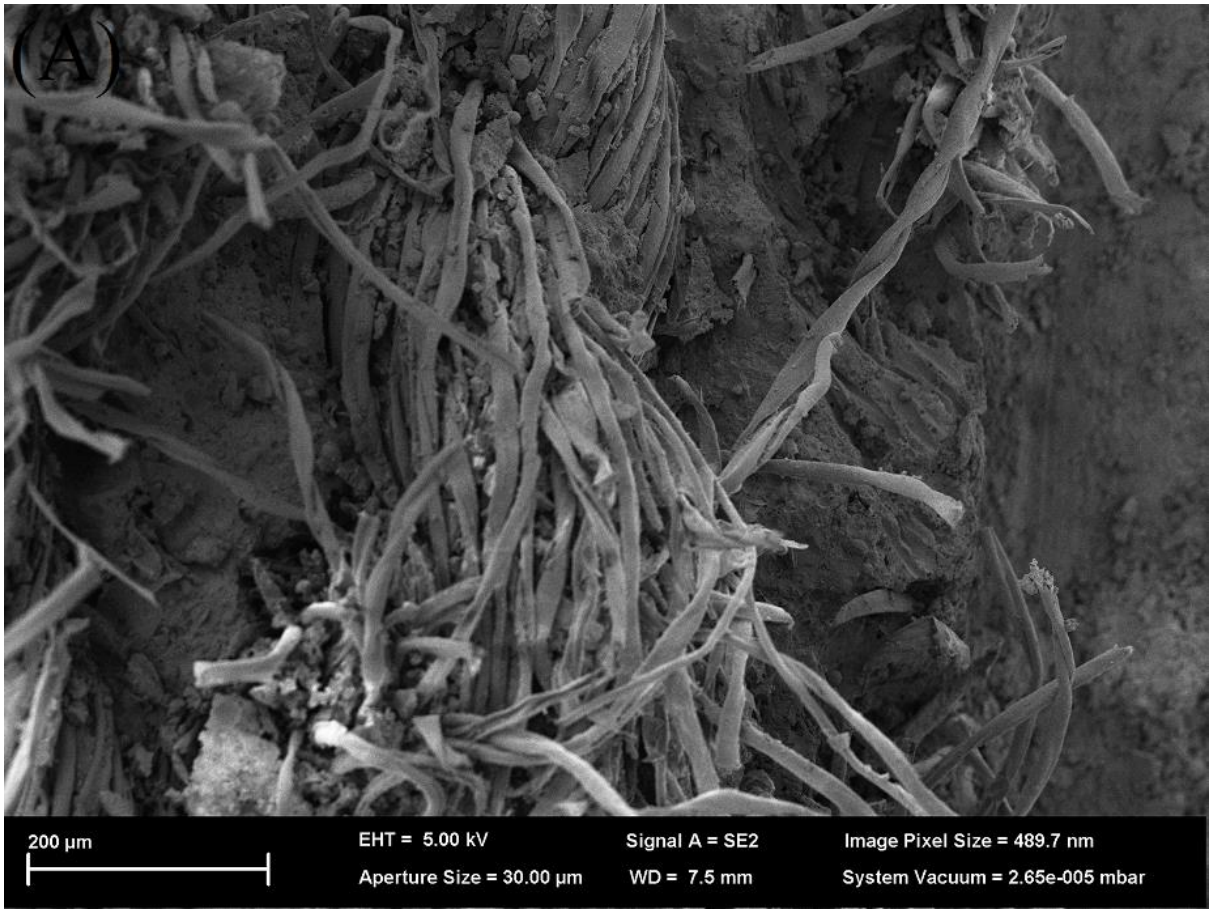

(A)
200 µm
EHT = 5.00 kV
Aperture Size = 30.00 µm
Signal A = SE2
WD = 7.5 mm
Image Pixel Size = 489.7 nm
System Vacuum = 2.65e-005 mbar

Fig. 7B

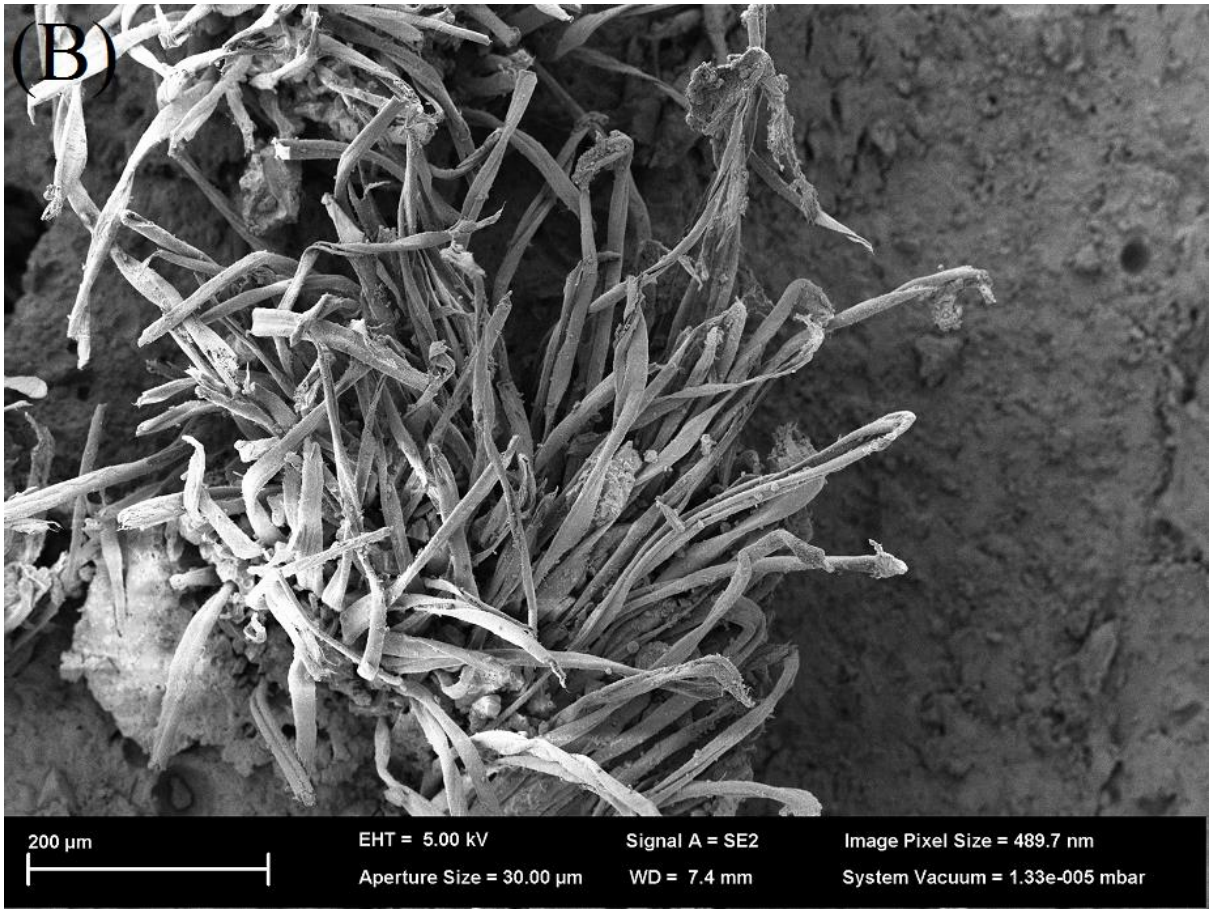

(B)
200 µm
EHT = 5.00 kV
Aperture Size = 30.00 µm
Signal A = SE2
WD = 7.4 mm
Image Pixel Size = 489.7 nm
System Vacuum = 1.33e-005 mbar

Fig. 7C

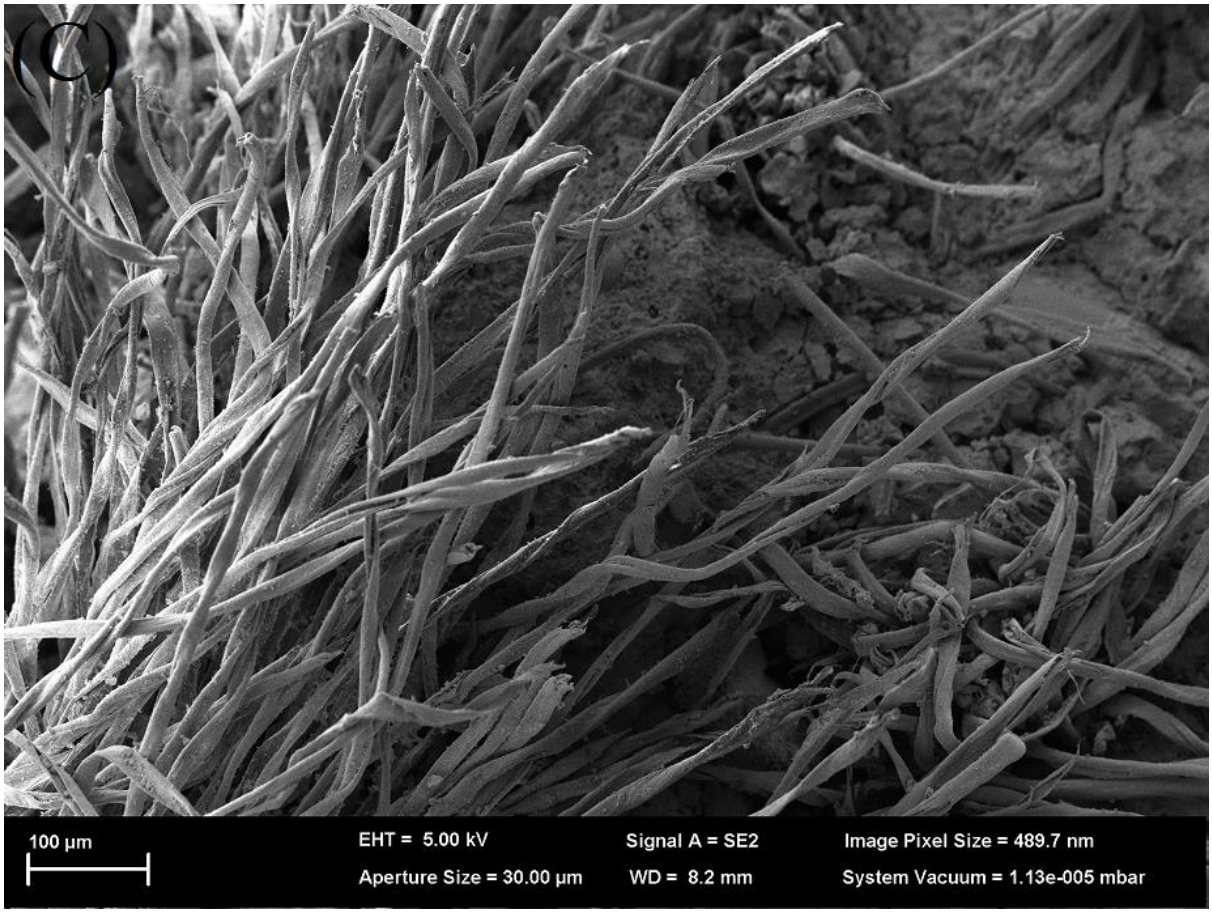

(C)
100 µm
EHT = 5.00 kV
Aperture Size = 30.00 µm
Signal A = SE2
WD = 8.2 mm
Image Pixel Size = 489.7 nm
System Vacuum = 1.13e-005 mbar

Fig. 7D

(D)
100 µm
EHT = 5.00 kV
Aperture Size = 30.00 µm
Signal A = SE2
WD = 7.3 mm
Image Pixel Size = 485.5 nm
System Vacuum = 9.52e-006 mbar

Fig. 7E

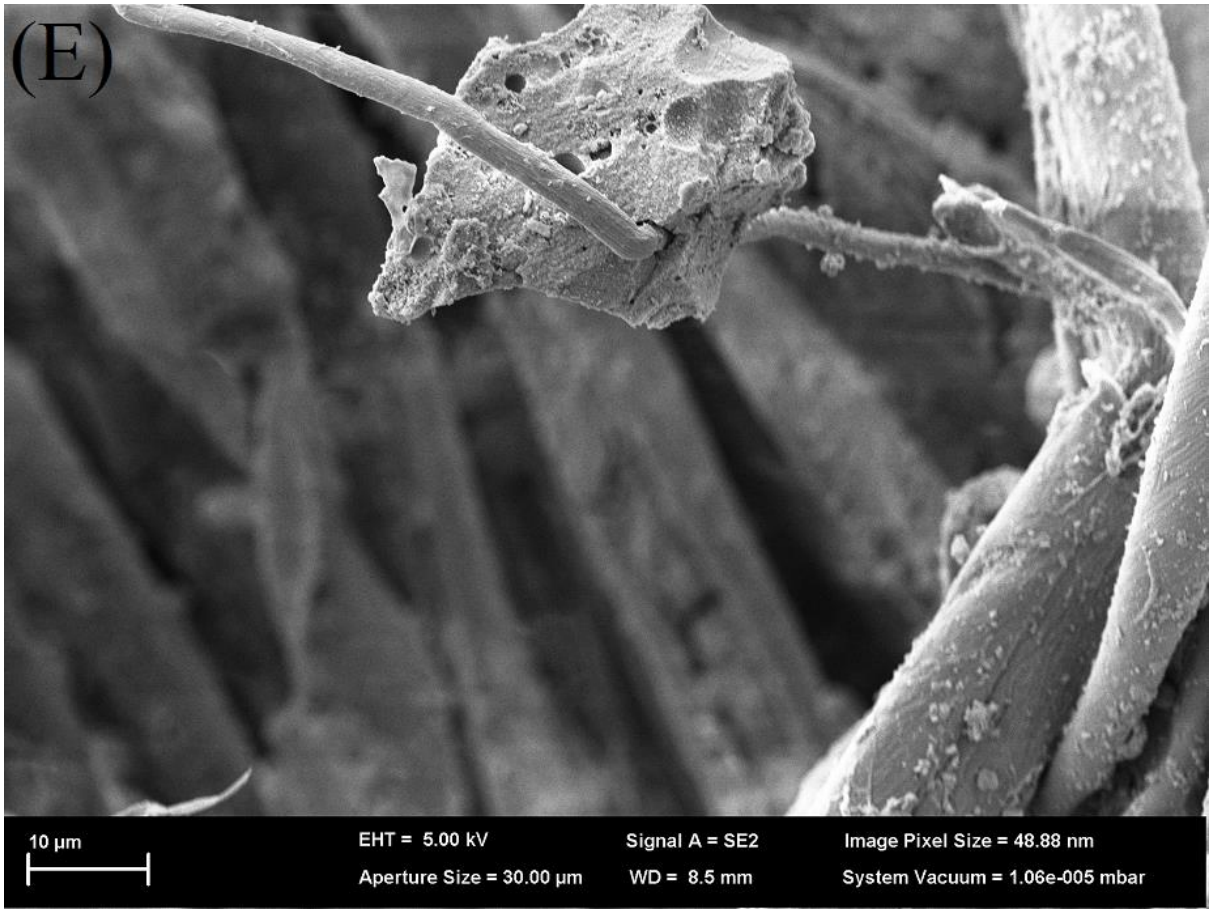

(E)
10 µm
EHT = 5.00 kV
Aperture Size = 30.00 µm
Signal A = SE2
WD = 8.5 mm
Image Pixel Size = 48.88 nm
System Vacuum = 1.06e-005 mbar

Fig. 7F

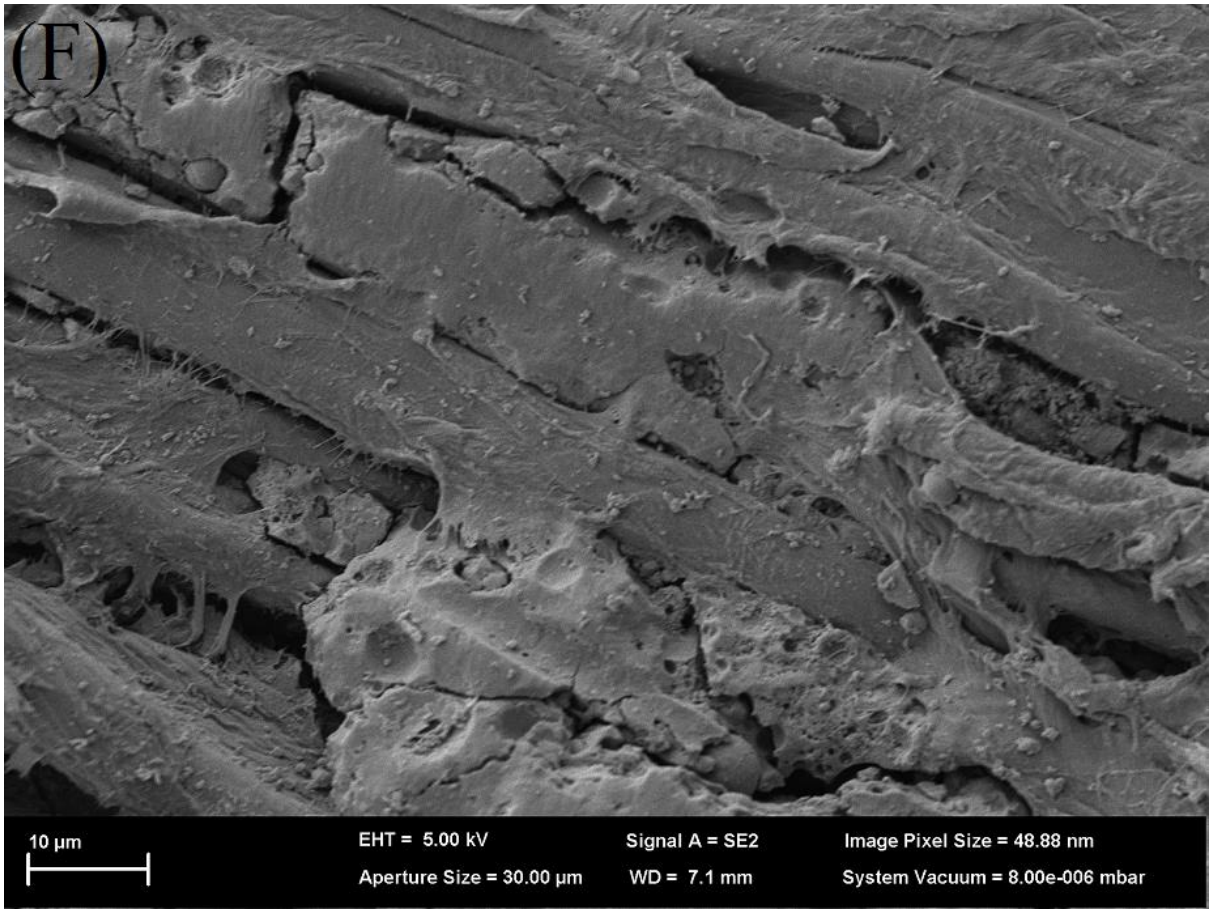
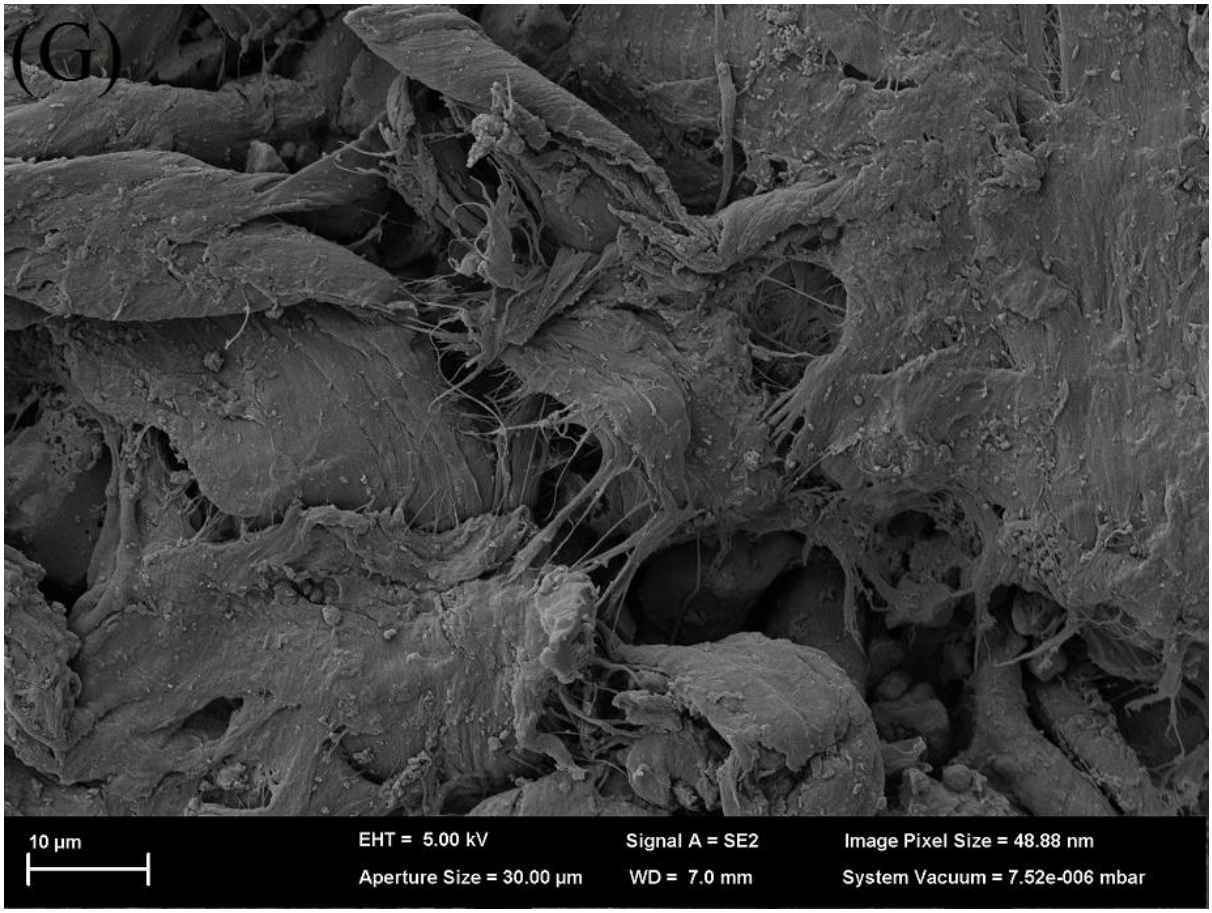

(F)
10 µm
EHT = 5.00 kV
Aperture Size = 30.00 µm
Signal A = SE2
WD = 7.1 mm
Image Pixel Size = 48.88 nm
System Vacuum = 8.00e-006 mbar

Fig. 7G


(G)
10 µm
EHT = 5.00 kV
Aperture Size = 30.00 µm
Signal A = SE2
WD = 7.0 mm
Image Pixel Size = 48.88 nm
System Vacuum = 7.52e-006 mbar

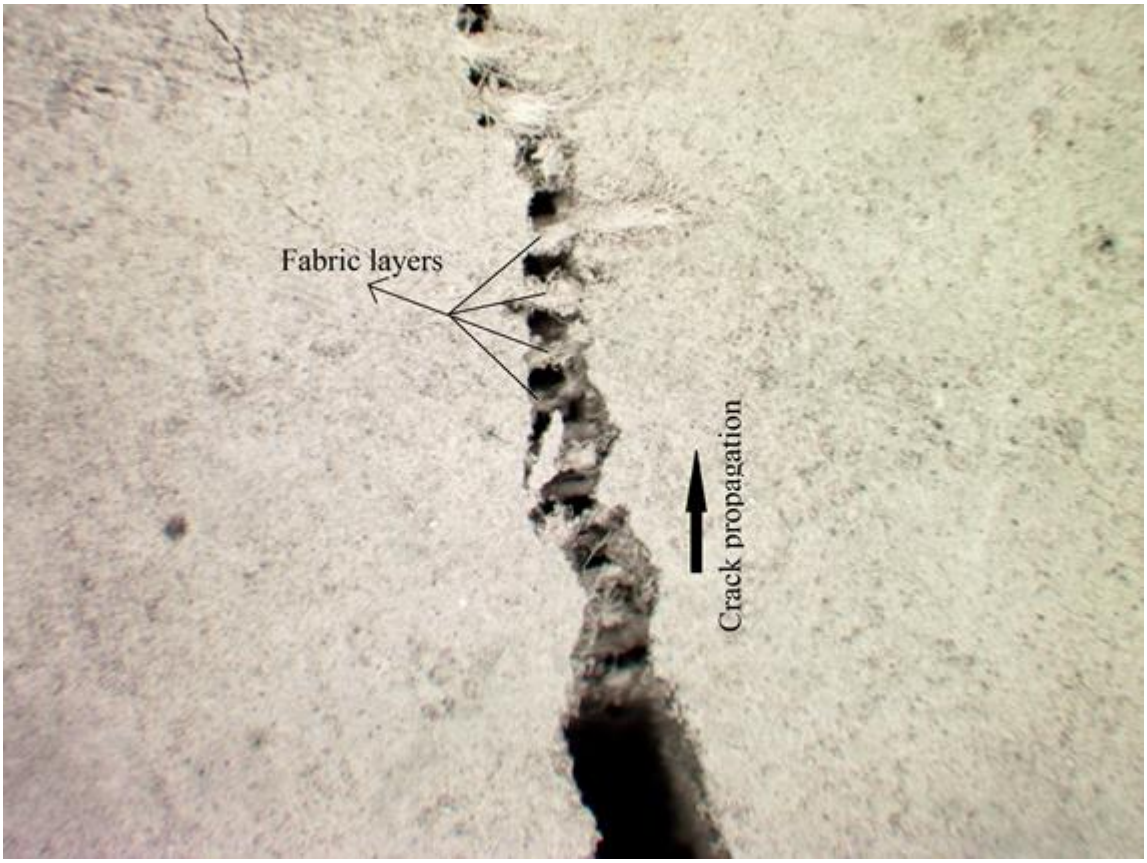



Fig. 8