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Development and mechanical characterization of composites based on unsaturated polyester reinforced with maleated high oleic sunflower oil-treated cellulose fiber

Hafiz Qasim Ali[1], Mohsin Ali Raza[1]*, Aidan Westwood[2], Faizan Ali Ghauri[1], Hassnain Asgar[1]

[1]Department of Metallurgy and Materials Engineering, CEET,

University of the Punjab, Lahore, Pakistan

[2]School of Chemical and Process Engineering,

University of Leeds, LS2 9JT, UK

*Corresponding author email: mohsin.ceet@pu.edu.pk

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Abstract

This work studies the effect of surface functionalization of chopped cellulose fibers (CF) on the mechanical properties of unsaturated polyester (UPE) matrix-based composites. Surface functionalization of CFs was carried out by treating them with maleated high oleic sunflower oil (MHOSO) solutions at concentrations of 2.5, 5 and 10 vol.%. Untreated and MHOSO-treated CFs were dispersed in UPE at 1, 2 and 3 wt. % by mechanical mixing. Fourier transform infrared spectroscopy confirmed successful functionalization of CF with MHOSO. Scanning electron microscopy revealed that MHOSO -treatment improved dispersion of the fibers in UPE. Tensile and compressive testing of the composites showed that MHOSO -treated CF improved the strength of the composites compared to untreated CF. This was attributed to better dispersion of the former cf. the latter. Increasing the extent of functionalization of CF with MHOSO resulted in increased strain to failure of the composites due to the MHOSO acting as a plasticizer. MHOSO -treated CF increased the hydrophobicity of the composites compared to untreated CF, thereby offering protection against moisture-induced degradation.

Keywords: cellulose fibers; unsaturated polyester; functionalization, mechanical properties

1. Introduction

Natural fibers have gained much interest as fillers for polymeric materials primarily due to their low-cost and renewability. Polymeric composites developed with natural fibers are considered to be green composites, and these have now been utilized in automobile, aerospace and construction applications. Thermoset polymers such as unsaturated polyester, epoxy and polyurethane are commonly employed as matrices for natural fiber-based composites as it is more convenient to mix chopped natural fibers in the liquid resins from which these matrices derive. Unlike synthetic fibers (glass, carbon and aramid), natural fibers are available only in chopped or discontinuous form, and this might be one reason why they do not outperform synthetic fiber-based composites in terms of properties. Thus, there is great interest in developing natural fiber-based composites with better properties to widen the scope of their applications [1, 2] .

Cellulose fibers (CF) are lightweight, inexpensive and biodegradable. Plants, especially cotton, are the major source of CF which has 91 % cellulose. The major uses of CFs are in the paper and textile industries. CFs are also used for insulation, ropes and mat production. CFs are promising fillers for production of polymer matrix composites. However, these composites cannot offer superior mechanical properties compared to synthetic fiber-based composites, mainly due to their hydrophilic nature which limits their bonding to organic matrices. Owing to moisture absorption by CF, mechanical properties of CF-based composites deteriorate due to poor interfacial bonding between the CFs and the polymer matrix [3]. In order to make CFs hydrophobic and thus enhance their compatibility with hydrophobic polymers, various surface modifications have been carried out [4]. Dispersion of fibers into the matrix and the interfacial bonding between the fibers and matrix are two important factors responsible for improved composite properties [5]. Dhakal et al. [6] studied the effect of water absorption on the mechanical properties of non-woven hemp fiber reinforced unsaturated polyester composites following immersion at room temperature and higher temperature. It was found that moisture uptake increased as a function of fiber volume fraction due to increased voids and cellulose content. Exposure to moisture resulted in significant drop in tensile and flexural properties due to the degradation of the fiber-matrix interface. Low et al. [7] developed and characterized polymer composites consisting of epoxy resin reinforced with recycled CF. Significant improvements in mechanical properties were achieved through the reinforcement of recycled CFs. However, the longer-term exposure of these materials to sea water had a detrimental effect on the flexural properties, although their impact properties were significantly improved.

Unsaturated Polyester (UPE) is a thermoset resin which is commonly used in composite manufacturing due to its low-cost and good mechanical properties [8]. It is widely used in sheet molding, electrical components, pipes, ducts, bath wares, etc. There are many reports published on natural fiber/UPE composites. For these composites, surface modification of fibers by coupling agents develop covalent bonds between matrix and fibers which was shown to improve the mechanical properties of composites [9].

Alkali treatment is used for surface modification of natural fibers. NaOH treatment effects the hydrogen bonding of the cellulose structure and replace -OH groups of the surface with Na^+ ions [10]. Rokbi et al. [11] reported that NaOH treatment of Alfa fibers improved flexural properties of UPE composites. They reported that alkali treatment changed the color of fibers to yellow, and through SEM they observed that treated fibers had smooth surface while untreated fibers had much rough surface. Benyahia et al. [12] showed that Alfa fibers treated with 7 % alkali solution improved mechanical properties of composites compared to the composites produced with fibers treated with 1, 3 and 5 % alkali solution**.** Similar findings were reported by Li et al**.** [13] who reported that treating sisal fibers with 10 % NaOH solution led to deterioration in mechanical properties of resulting composites as the high concentration of NaOH damage fibers and increased delignification. They obtained good properties when fibers were treated with 5 % sodium hydroxide solution.

Apart from the alkali treatment, natural fibers' surface modification to transform their nature from hydrophilic to hydrophobic was also carried out to improve bonding between fibers and matrix. Zadorecki et al. [14] treated CFs with coupling agent based on trichloro-s-triazine. As a result of this treatment, tensile strength and stiffness of the UPE composites increased. Sabinesh et al. [15] treated coir fiber with NaOH and then with 5% silane and 95 % ethanol solution. The tensile and flexural properties of coir fiber reinforced polyester composite were significantly improved with increasing fiber volume fractions. The maximum tensile strength, modulus and flexural strength of the chopped fiber/polyester composite was achieved at 25 % volume fraction. Liu et al. [16] modified hemp fibers with 3-isopropenyldimethylbenzyl isocyanate in the presence of catalyst dibutyltin dilaurate and studied the effect of functionalization on the mechanical properties and water absorption of hemp/UPE composites. They found improvement in the tensile strength and reduction in water absorption in the case of treated -hemp fiber/UPE composites. In another study Liu et al. [17] coupled bamboo fibers with isocyanate group using isocyanatoethyl methacrylate and reported almost similar results as reported in previous study. Further, Liu et al. [18] studied the interfacial bonding and mechanical properties of N-methylol acrylamide grafted bamboo fibers in UPE matrix. They concluded that treated bamboo fibers improved interfacial bonding with UPE matrix resulting in enhanced mechanical properties of composites compared to corresponding composites produced with untreated fibers. Recently, Liu et al. [19] modified bamboo fibers by treating with 1,6-diisocyanatohexane and 2-hydroxyethyl acrylate for better mechanical properties and water resistance. They found that the treatment resulted in better mechanical properties and less water absorption of treated bamboo fiber/UPE composites.

Maleated high oleic sunflower oil (MHOSO) is commonly used in paper manufacturing as a sizing agent, and is responsible for the brightness and hydrophobicity of the paper. The molecular representation of MHOSO is shown in Fig.1. The anhydride group of MHOSO can covalently bond with CF by substituting -OH group on the surface of fibers resulting in ester linkage between fiber and MHOSO as shown in Fig. 1. The non-polar segment of MHOSO can impart hydrophobicity to fibers and thus can improve their compatibility with polymers. Raza et al. [20] treated CFs with MHOSO to render them hydrophobic and to improve their dispersibility in styrene butadiene rubber.

Figure 1

In this work, chopped CFs were treated with MHOSO and incorporated in UPE to develop composites. The MHOSO treatment was employed to make CF hydrophobic and to develop a strong interface between fibers and UPE. CF were reinforced in UPE matrix at concentration of 1, 2 and 3 wt.%. The effects of MHOSO treatment and fiber content on the mechanical properties and water absorption ability of CF/UPE composites were investigated.

2. Experimental

2.1 Materials and reagents

Following materials were used for production of MHOSO and composites: CF (Ex. Valdivia pulp mill, Chile), UPE (procured from local market), maleic anhydride (BDH), high oleic sunflower oil (procured from local market), xylene (Merck) and aluminum chloride (BDH).

2.2 Preparation of MHOSO

MHOSO was prepared by the reaction of maleic acid anhydride with high oleic sunflower oil at a molar ratio of 2:1 in the presence of 0.5 % aluminum chloride. The reaction was carried out under continuous stirring in a three neck round bottom flask. The temperature of the solution was maintained at 215 °C for 7 h. Maleic anhydride is grafted onto hydrophobic ligands of high oleic sunflower oil by the process of maleinisation [21]. Maleic anhydride can react either with the allylic site or double bond present in the high oleic sunflower oil [22]. The grafting reaction in the case of allylic site is presented in Fig. 2.

Figure 2

2.3 Surface modification of CFs

CFs were obtained from bleached paper pulp sheets which were chopped in rotary grinding machine. After grinding, fibers were dried in an oven at 100 °C for 2 h. The diameter of these fibers was about 20-30 µm and length was about 0.4-1 mm.

Surface modification of CF was carried out at using MHOSO solution with concentration of 2.5, 5 and 10 vol.%. For preparing MHOSO solution, a known volume of MHOSO was poured along with xylene in three neck round bottom flask fitted with a condenser. The amount of fibers was taken in such a way that fiber to mixture ratio always remained 1:20 wt./vol. The mixture was continuously stirred at 100-110 °C for 40 min. Afterwards, fibers were filtered, washed with ethanol and dried at room temperature for 1 day followed by drying in an oven at 80 °C for 3 h.

The treated and untreated CFs were dispersed in water to assess their surface treatment. The untreated fibers dispersed in water while MHOSO-treated fibers floated on water (Fig. 3) suggesting the successful attachment of MHOSO moieties to the fibers.

Figure 3

2.4 Development of composites

CF-based composites were produced with untreated and treated-CF at concentration of 1, 2 and 3 wt %. The calculated amount of CF was dispersed in UPE resin using mechanical mixer stirrer at the speed of 2000 rpm for 15 min. After mixing, curing agent (2% methyl ethyl ketone peroxide) and accelerator (1 wt.% cobalt naphthenate) were mixed in the CF/UPE dispersion. The dispersion was then poured in a custom-made die for making samples for tensile and compression testing. The dispersion was initially cured at room temperature followed by post curing at 45 °C for 1 h.

2.5 Characterization

Fourier transform infrared spectrum of untreated and MHOSO-treated CF were obtained in ATR mode in the range of 4000 cm^{-1} to 400 cm^{-1} (Shimadzu). Dispersion quality of fibers in UPE was observed on freeze-fractured surface, sputter coated with Au-Pd alloy, using scanning electron microscope (FEI, Inspect S50).

Dumbbell shaped specimens were prepared according to BS 2782-3 (overall length = 75 mm, gauge length = 20 mm, width within gauge length = 4 mm and thickness of 2 mm) by injection moulding. Tensile properties (fracture strength, UTS, percentage elongation, modulus and toughness) were averaged by testing at least three specimens of each composition using tensometer (Monsanto tensometer, UK).

Compressive strength was also determined by tensometer on cylindrical samples (diameter= 9 mm and length =18 mm) according to ASTM D695-15.

The water absorption test was carried out by immersing 10 mm long specimen of each composite in water for 15 days. Samples of different compositions of fibers were suspended in water in such a way that all sides of the samples were completely immersed in water and weight gain was measured in accordance with ASTM D2842.

3. Results and discussion

3.1 FTIR analysis

The FTIR spectra of MHOSO, untreated and MHOSO treated CF are shown in Fig. 4. In FTIR spectrum of untreated CF and MHOSO-treated fibers a peak at 1025 cm^{-1} represents C-O-C stretching of polysaccharide skeleton, which is the back bone of the CF. A peak at a wave number 1589 cm^{-1} indicates the presence of C=C bond of lignin. The peaks at 2921 cm^{-1} and 2922 cm^{-1} in both untreated CF and MHOSO-treated CF represents anti-symmetric and symmetric stretching of CH_2 group. A peak at 3330 cm^{-1} in both treated and untreated CF represents stretching of -OH group.

<u>Figure 4</u>

A peak at 1737 cm^{-1} in the spectrum of MHOSO-treated fibers is due to ester group and this peak confirms the treatment of CF by MHOSO as a result of esterification reaction as presented in Fig. 2. The same peak is also present in MHOSO spectrum due to ester linkage in high oleic acid (Fig. 2). Further, two peaks at 1780 and 1860 cm^{-1} from anhydride group of MHOSO attributed to symmetric and anti-symmetric stretching of C=O [23] disappeared in spectrum of MHOSO-treated CF (Fig. 4). Similarly, two peaks at 722 [23] and 915 cm^{-1} present in MHOSO spectrum due to anhydride CH and C-O [24], respectively, disappeared in MHOSO-treated CF because anhydride group of MHOSO developed an ester bond with CF (Fig. 1). Thus, the FTIR analysis clearly suggests that MHOSO treatment of CF was successfully carried out.

3.2 SEM analysis

SEM images of untreated CF and MHOSO-treated CF are shown in Fig. 5. Untreated CF and MHOSO-treated CF fibers length is in the range of 0.4-1 mm and diameter ca. 20-30 µm as can be seen (Fig. 5 (a & b)). It was also be observed from these images that some of the CFs have twisted ribbon like morphology. Layers of impurities (pectin and lignin) on the surface of untreated fibers can be seen (Fig. 5(a & c)), while such impurities are not present in MHOSO-treated fibers (Fig. 5 (b & d)) implying that MHOSO treatment clears impurities from fiber surface resulting in much smoother surface of fibers than untreated fibers. Similar findings were reported by Robki et al. [11]. The MHOSO moieties were attached with the fibers by the treatment which was carried out at temperature of 110 °C for 40 min (Section 2.3). It might be the high temperature and longer time which favored the clearing of impurities from the fibers' surface resulting in more uniform attachment of MHOSO and smoother surface.

The SEM image of composites produced with untreated CF and MHOSO-treated CF are shown in Fig. 5. The untreated CF are not well dispersed in UPE matrix and agglomerated fibers can be clearly seen in Fig. 5(e). On the other hand, dispersion of MHOSO-treated fibers in UPE is much uniform with no agglomerated fibers observed from SEM analysis of these composites (Fig. 5(g & h)). The fracture surface of untreated CF/UPE composites showed clear holes resulted from fiber pullout (Fig. 5 (f)). This indicates poor interfacial adhesion of untreated CF with UPE due to presence of impurities (mainly lignin). This also implies that UPE resin was unable to wet untreated CFs' surface for mechanical interlocking between CFs and UPE matrix. In contrast, MHOSO-treated CF/UPE composites have much rougher surface, no holes and some fibers were also defibrillated and torn apart at the surface instead of pulling out (Fig. 5(g & h). Further, due to pull out of MHOSO-treated fibers matrix deformation occurred by absorption of strain energy rendering the composites more ductile. The tensile testing results discussed in following section also confirmed this behavior.

Figure 5

3.3 Tensile testing

The ultimate tensile strength (UTS) and percentage elongation of composites as a function of wt.% of fibers are shown in Fig. 6. The influence of MHOSO-treatment on the fibers is also presented in these figures.

Figure 6

The UTS of composites produced with MHOSO-treated fibers was found higher than the corresponding composites produced with untreated fibers. The highest UTS was obtained at 2 wt.% loading of fibers and when the fibers were treated with 5 % MHOSO. The UTS of this composite was found to be 42 % higher than the corresponding composite produced with untreated fibers. The UTS of UPE was increased from 28 to 41 MPa at 2 wt.% loading of 5 % MHOSO-treated CF, corresponding to increase of 146 % over pristine UPE. Unlike UTS of composites, elongation of composites developed with MHOSO-treated fibers increased compared to pristine UPE and composites produced with untreated fibers. However, it increased almost the same at 1 and 2 wt. % loading, corresponding to increase of ca. 150 % over pristine UPE. However, elongation of composites produced with 3 wt. % loading for all types of MHOSO-treated fibers was found significantly higher. The more functionalization of fibers result in more attachment of MHOSO to the fiber surface which act as a plasticizing agent by reducing the intermolecular forces between the UPE molecules resulting in increased elongation of the composites. The 5 % MHOSO treatment of fibers seems to be an ideal as it not only increase strength of composites but also imparts significant ductility to the composites, perhaps the balancing of plasticization effect and good interfacial bonding between fiber and matrix playing their role. Thus incorporation of 5 % MHOSO-treated fibers resulted in a good combination of strength and ductility resulting in more tough composite material. The results imply that MHOSO-treated fibers can reduce brittleness of UPE and make it more tough material for composites applications. Benhaya et al. [12] dispersed NaOH-treated Alfa fibers at 10 wt. % loading in UPE and found improvement in tensile strength. Sabinesh et al. [15] prepared cotton based UPE composites and achieved maximum UTS (39 MPa) at concentration of 25 wt. % fiber content.

3.4 Compression testing

The compressive strengths of UPE and composites as a function of fiber content are presented in Fig. 8. Similar to UTS of composites, MHOSO-treated fibers displayed higher compressive strength than untreated fiber composites. The pristine UPE had a compressive strength of 1644 GPa which was increased to 2053 GPa at 2 wt.% loading of 5% MHOSO-treated fibers, corresponding to an increase of ca. 25 %. It can also be observed from Fig. 8 that fibers treated with 10 % MHOSO reduced the compressive strength of composites similar to UTS of composite due to plasticization effect as discussed before.

Figure 7

3.5 Water absorption

Since fibers are hydrophilic in nature, they have more ability to absorb moisture. The MHOSO treatment made CF hydrophobic to some extent and this would be responsible in reducing moisture absorption of composites. Fig. 8 shows that water absorption of composites increases with increasing fiber content, but also reduced due to the more attachment of MHOSO to the fibers.

Compared to CF, MHOSO-treated fibers become hydrophobic due to attachment of long hydrocarbon chain of oleic acid. This has also be shown in Fig. 3. Thus the hydrophobic nature of fibers as well as their better dispersion in UPE would lower the water uptake of UPE. The result suggests that MHOSO being hydrophobic in nature reduces the water intake of fibers in the composites.

<u>Figure 8</u>

4. Conclusions

Following conclusions can be drawn from this study:

- Functionalization of CF with MHOSO is an effective way of improving the dispersion of CF fibers in UPE matrix as it makes CF fibers hydrophobic and more compatible with non-polar polymer matrices.
- Composites produced with MHOSO-treated CF improved UTS, compressive strength and % elongation compared to composites produced with untreated CF attributed to better dispersion of fibers in the UPE matrix.
- The extent of MHOSO functionalization of CF also have a significant role on the mechanical properties of composites. The maximum UTS and compressive strength was achieved when fibers were functionalized with 5 % MHOSO solution. The higher functionalization makes composites more ductile as MHOSO moieties attached with fibers act as plasticizing agent by reducing intermolecular forces of UPE.
- The increased extent of CF functionalization with MHOSO decreased the moisture uptake ability of resulting composites due to more hydrophobicity offered by MHOSO to CF.

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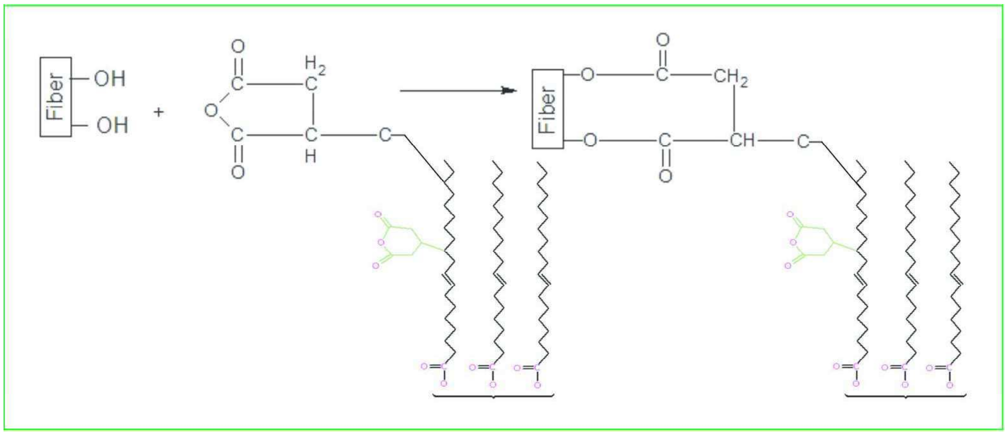

Fig. 1. Schematic representation of interaction of MHOSO with CFs.

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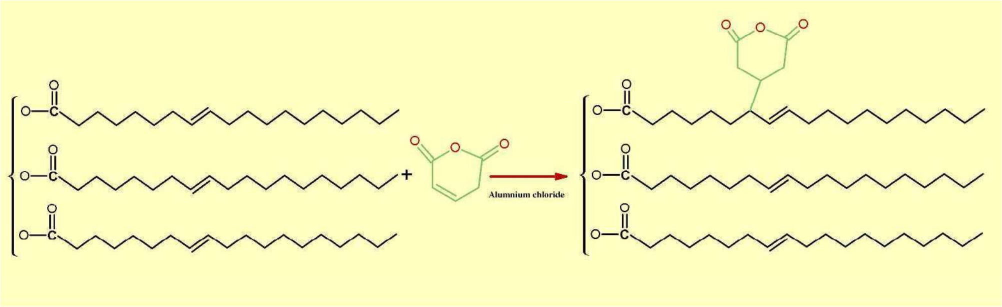

Fig. 2. Reaction between maleic anhydride and high oleic sunflower oil for the preparation of MHOSO

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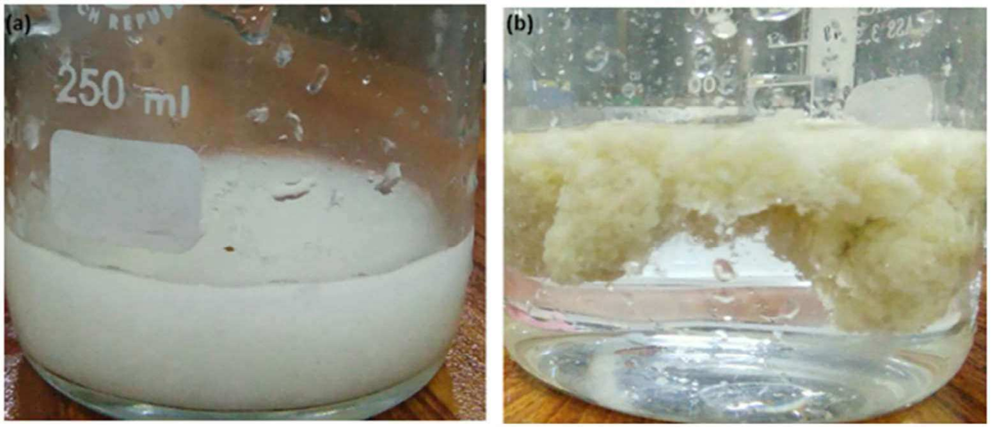

Fig. 3. Behavior of untreated CF (a) and MHOSO-treated CF (b) in water.

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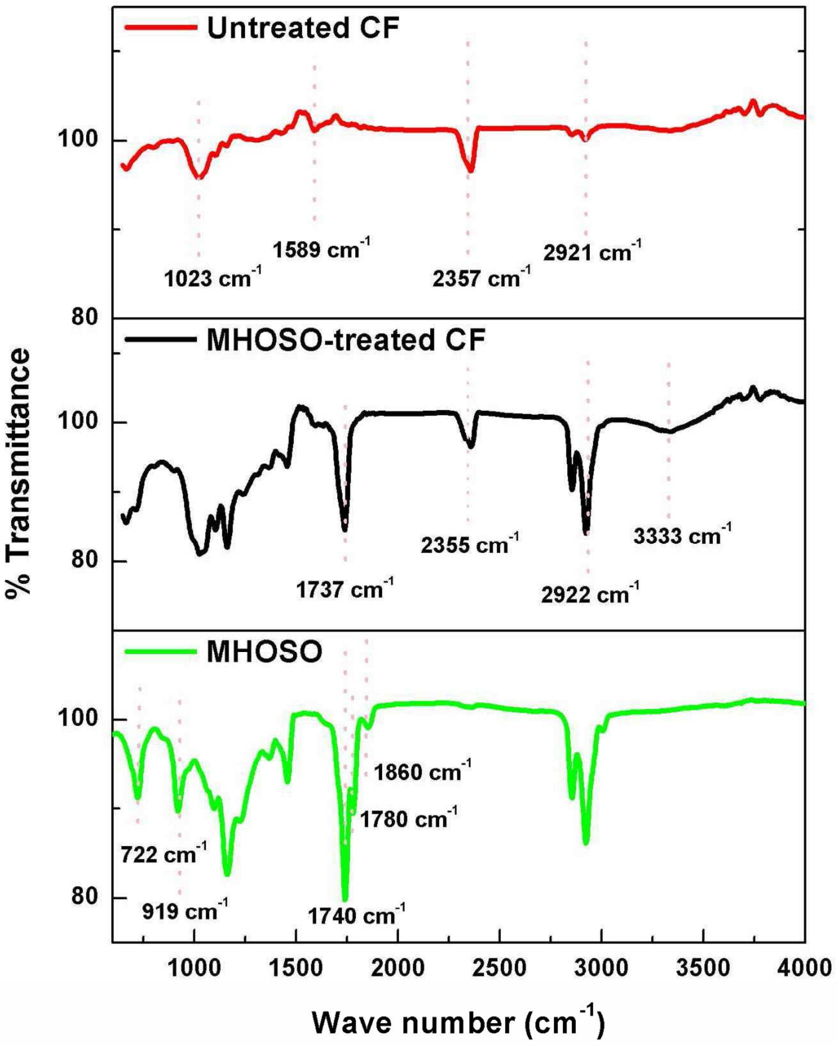

Fig. 4. FTIR analysis of MHOSO, MHOSO-treated and untreated CF

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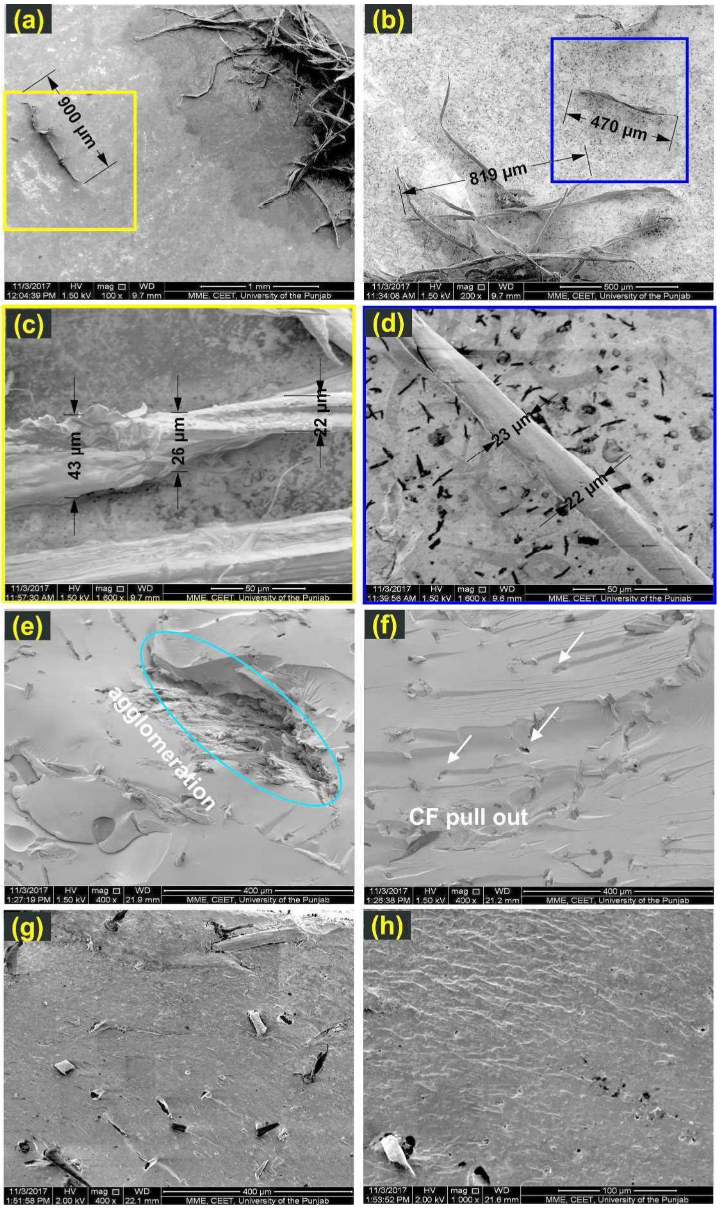

Fig. 5. Fig. 5 SEM image (a & c) untreated fibers (b & d) treated fibers (e & f) 3 wt.% untreated-CF/UPE composite (g & h) 3 wt.% MHOHO-treated CF/UPE

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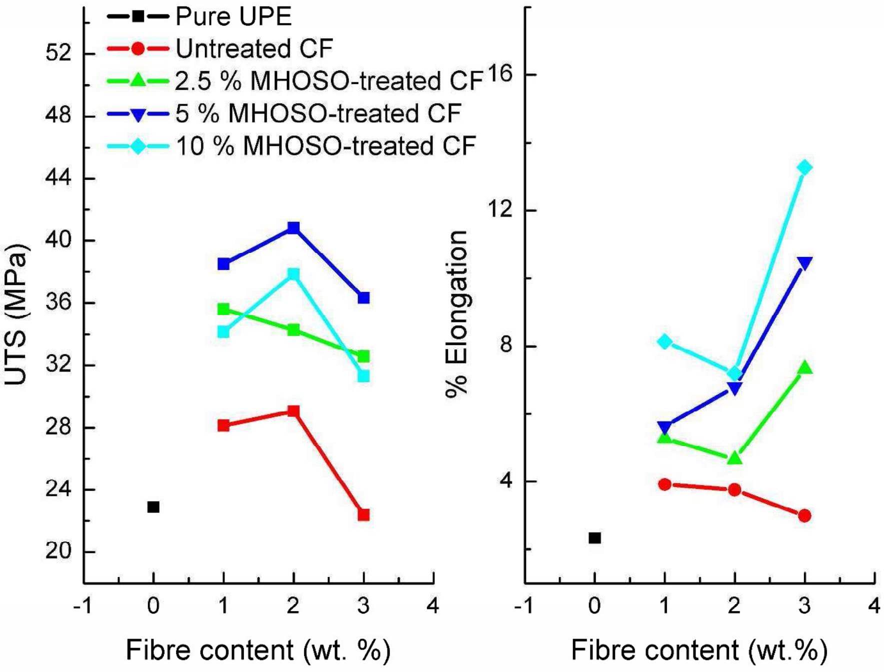

Fig. 6. UTS (a) and % elongation (b) of pure UPE and UPE composites produced with untreated CF and MHOSO -treated CF as function of fiber wt.%

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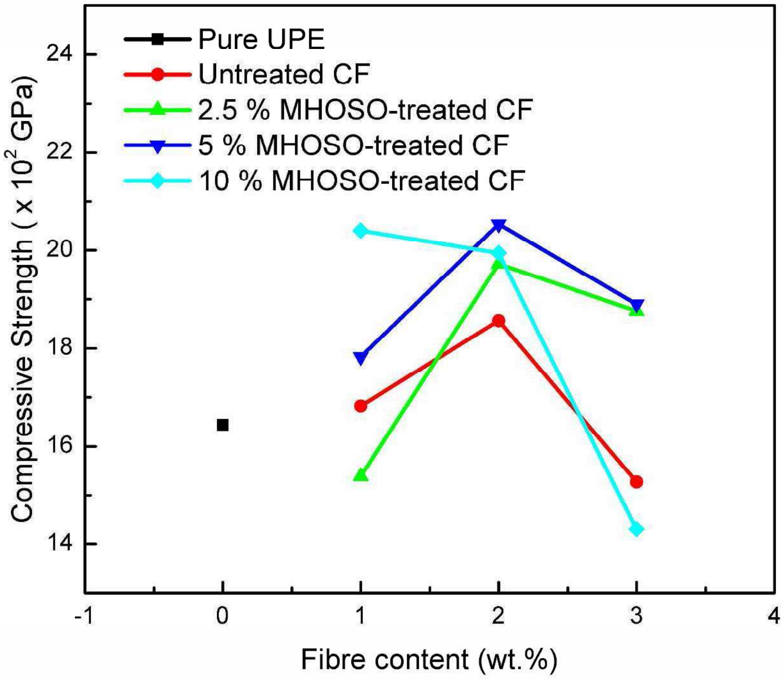

Fig.7. Compressive strength of UPE and composites produced with untreated and MHOSO -treated CF

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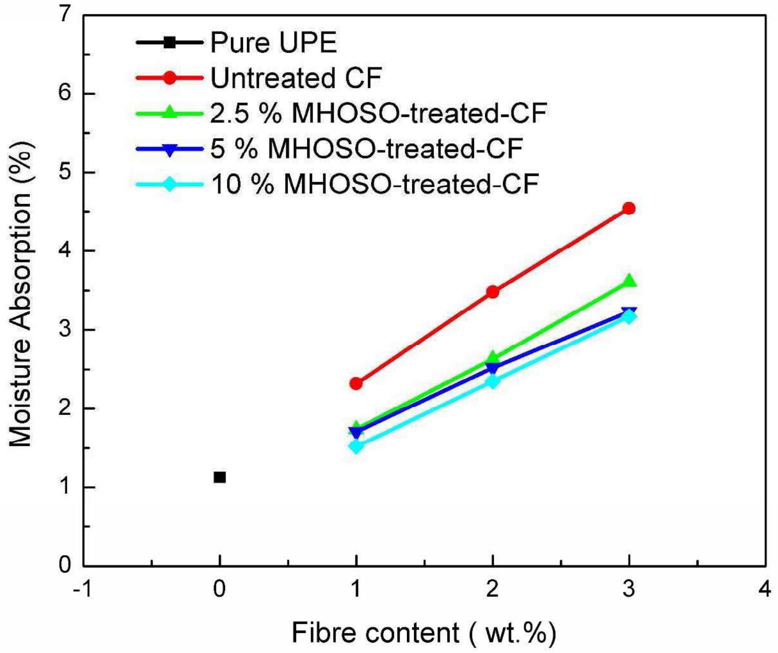

Fig. 8. Moisture absorption of composites produced with untreated and treated CF as a function of fiber content

271x209mm (300 x 300 DPI)