EXPERIMENTAL INVESTIGATION OF MECHANICAL AND FTIR ANALYSIS OF FLAX FIBER/EPOXY COMPOSITES INCORPORATING SiC, Al_2O_3 AND GRAPHITE

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The research work aspires to analyze the effect of various fillers with flax fiber reinforced epoxy composites. Composites filled with varying weight percentage of different fillers such as silicon carbide, alumina and graphite were fabricated by compression moulding techniques. The effect of alkaline treatment of flax fiber was verified by FTIR analysis. The composite containing 15 wt % flax fiber and 7 wt % SiC gave the better mechanical properties. The optimum SiC for better mechanical properties of this present composite is found to be 7 wt. %. The result of this research indicates that incorporation of flax fiber and 7 wt. % SiC filler significantly enhance the mechanical properties of the composite. We concluded that ceramics filled composites will provide substitute brake pad materials respect to asbestos and synthetic based materials.

Keywords: *Flax fiber, SiC, Al_2O_3, Graphite, FTIR*

1. Introduction

Polymer composites are one of the emerging areas in polymeric science that triumph attention for application in various sectors ranging from automobile to the construction industries [1]. Over the past two decades, natural fibers have been receiving considerable attention as substitutes for synthetic fiber reinforcements like carbon, glass, aramid and boron. Unlike the synthetic fibers these natural fibers are capable of imparting certain benefits to the composites such as low cost, high strength, low density, biodegradability and high degree of flexibility during extraction of fiber and processing [2]. The natural fibers in its own quality have a positive impact to the environment. Natural fibers play a key role in the emerging "green" economy based on energy efficiency. The use of biological sources in polymer products reduces the carbon emissions due to plastic burning [3]. Natural fibers such as flax, hemp, kenaf, sisal, bamboo and jute are alternatives to the use of synthetic fibers as reinforcement in polymer composites. Flax technical fibers are obtained from the bast of a plant of the Linaceae family (Linum Usitatissimum), which is originally from Europe, but is also cropped in USA and Canada for human nutrition. Among the different natural fibers, flax fiber is certainly a promising fiber due to its excellent strength, less density, low cost, availability, production at its ease and eco friendly [4]. Furthermore, flax fiber is significantly less abrasion than glass fiber. Flax composites have attracted attention to be considered as the next generation materials for structural application for infrastructure, aerospace industry, and automotive industry [5]. The strength of natural fiber reinforced composites not only depends on the matrix but also on the number of parameters such as fiber-matrix bonding, fiber orientation, volume fraction of fibers and fiber aspect ratio. Most of the studies on natural fiber composites involve study of mechanical properties as a function of fiber content, effect of chemical treatment on fibers and the use of external coupling agents [6]. The tensile, flexural and impact strength of flax and bamboo fibers reinforced hybrid composites varies linearly with the volume fraction of the flax fiber. It is clearly observed that the mechanical properties of the flax and bamboo fibers reinforced hybrid composites is more at 40 % flax fiber volume fraction [7]. In addition, the ceramic fillers can also be used with some polymeric matrices primarily to reduce cost, improve the wear and friction resistance and improve the strength. The improved property of composites in industrial and structural applications by the addition of ceramic fillers has shown a great promise and has lately been a subject of considerable interest. Among different parameters, weight percentage of fillers is the most important factor significantly influencing the properties of composites. The flexural strength of

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jute/Al_2O_3 composite increased from 48.1 MPa to 72.94 MPa due to the addition of Al_2O_3 and ability of natural fiber to resist bending forces and good load transfer from the fiber to matrix resulting in improves strength properties [1]. The tensile and impact strength of Sisal/Polyester composite has been increased with addition of 10 wt % SiC when compared to without of SiC [2]. The flexural strength of glass/epoxy composites labefaction by 34% while the Al_2O_3 content increases from 0 to 20 wt.%. This reduction probably caused by an incompatibility of Al_2O_3 and the epoxy, leading to poor interfacial bonding between the fillers and surrounding matrix. The tensile strength of bamboo/epoxy composite increases with 10 wt % SiC as compared to without of SiC. This may be uniform dispersion of SiC and better stress transfer in interface zone [3]. The tensile and flexural strength of jute/epoxy composite are found to increase with increasing the SiC content up to 4 vol.%. Beyond this content drop in mechanical properties is found [4]. The present research work has been done on the mechanical and FTIR analysis of various ceramic fillers and how these fillers interact with flax fibers. However the study on the effect of ceramic fillers on the mechanical and FTIR behavior is limited. To this end, the objective of the present research work is to study the effect of ceramic fillers on the mechanical and FTIR analysis of flax fiber reinforced epoxy composites.

2. Materials and experimental procedures

2.1.Materials

In the present research work, flax fiber is used for fabricating the composite materials. Flax fiber is extracted by mechanical decortications process. The epoxy resin (LY556) and hardener (HY951) in the ratio of 10:1 by weight is mixed uniformly by using mechanical stirrer, supplied by M/s Covai seenu Ltd., Coimbatore, India. The benefits of epoxy resin are their low shrinkage volume, excellent adhesion, better heat resistance and good mechanical properties [8,9]. SiC, Al_2O_3 and Graphite are provided by M/s Covai Metal Mart Ltd., Coimbatore, India.

2.2.Chemical treatment

The major problem of natural fiber composites originate from the hydrophilic nature of the fiber and hydrophobic nature of the matrix. The inherent incompatibility between fiber and resin results is weakening bonding at the interface [10]. Chemical treatment on natural fibers is an essential processing parameter to reduce hydrophilic nature of the fibers and thus plays a vital role in improving adhesion with the matrix. In order to avoid the wettability of the fiber and to increase the bonding between reinforcement and matrix, chemical treatment on fiber is necessary [11]. The fiber is washed with distilled water to remove impurities and dirt present on the fiber. The washed fiber is dried under sun rise for 24 hrs and the fibers are treated with 5% NaOH solution for 3 hrs at room temperature, then they are washed with running water. Subsequently the fibers are dried in hot air oven at 80 °C for 3hrs [12,10]. Hydrophilic hydroxyl groups are removed from the fiber by the action of alkaline solutions.

2.3.Preparation of composites

The materials used for the research are prepared by compression moulding techniques.

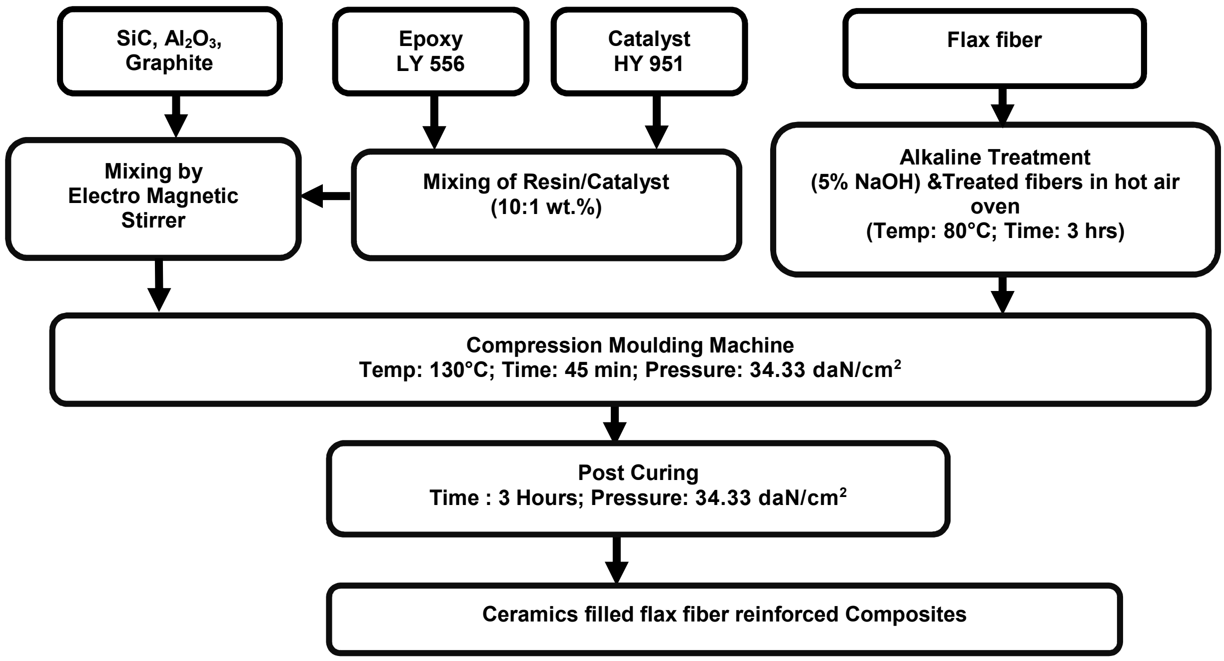


Fig 1- Fabrication route of the ceramics filled composites.

Table 1

Formulation of composite materials

Sample code	Flax fiber (% wt)	Epoxy (% wt)	SiC (% wt)	Al_2O_3 (% wt)	Graphite (% wt)
A	15	70	5	5	5
B	15	70	6	5	4
C	15	70	7	5	3
D	15	70	4	6	5
E	15	70	3	7	5

Flax fibers are cut into 30 cm length to prepare samples. The lamina consists of six layers of flax with homogeneous ceramics filled epoxy resin. The required composition of fillers are mixed with epoxy resin and hardener and then stirred gently by electromagnetic stirrer to form homogeneous mixture. Initially, the epoxy resin is filled over the aluminum foil sheet. The first layer is the flax fiber, homogeneous mixture of epoxy resin and hardener with ceramics filler is filled over the flax fiber and then subsequent layers are filled after resin gets dried. Finally, these laminas are kept in frame, for over 45 minutes at 130°C and pressure of 35 Kg/cm^2 is applied for 45 minutes before it is removed from the mould. The laminate size is limited to 30x30x0.5cm [7]. Figure 1 shows the fabrication route of the ceramics filled composites. By varying the content of ceramic fillers of different weight percentage, a series of composites were prepared. Table 1 shows formulation of composite materials.

3.Mechanical and FTIR studies

3.1.Tensile and flexural test

The tensile test is conducted using computerized universal testing machine (UTM) and results are analyzed to calculate the tensile strength of ceramics filled composites. The samples with dimensions of length 250 mm and width 25 mm are used. Experiments are conducted at a cross head speed of 20 cm/min. The three point bend test is performed to understand the flexural behavior of composites according to ASTM D 790 standards on computerized universal testing machine. The samples with dimensions of length 125 mm and width 12.7 mm are used. The test is conducted at a cross head speed of 10 mm/min [7].

3.2.Inter-laminar shear strength and impact test

The data recorded during the 3 point bend test can be used to determine the inter-laminar shear strength by using below Eq.

$$\mathrm{ILSS} = \frac{3\mathrm{F}}{4\mathrm{bt}}$$

Where F is the braking load (N), b and t are the width and thickness of the samples.

The impact test samples are made according to the ASTM D 256 standard dimension 60mm x 12mm x 5mm [7].

3.3.FTIR analysis

A Fourier transform infrared spectrometer is used for the structural determination of functional groups and compounds [13]. KBr disk sample preparation method is followed in taking infra spectrum. Fibers are mixed with KBr at the defined ratio then the mixer is pressed under vacuum to form pellets. FTIR spectra are recorded between 4000 cm^{-1} and 400 cm^{-1} with a resolution of 4 cm^{-1} and 20 scans are carried for each specimen in transmittance mode.

3.4.SEM analysis

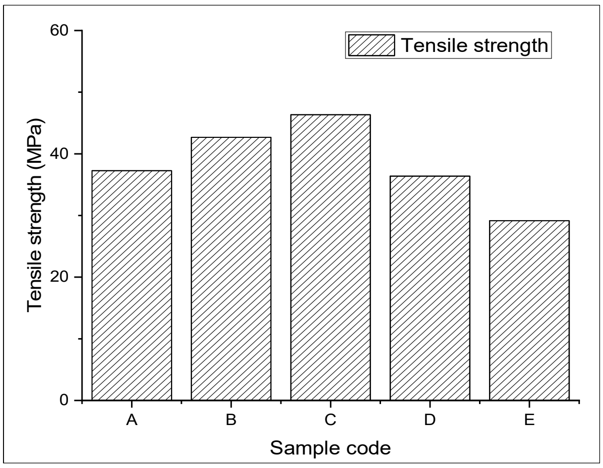
The bonding properties like fiber-resin interaction, fracture surface and fiber pull out of alkali treated flax fiber reinforced composites is analyzed by scanning electron microscope (SEM). A fractured thin section of the sample was mounted on an aluminum stub and was sputter coated with gold prior to morphological analysis.

4.Results and discussions

4.1.Tensile and flexural strength

The tensile strength of different ceramics filled flax composites are depicted in Figure 2.Tensile strength increases linearly with SiC up to 7 wt. %. The addition of SiC will increase strength which is an indication of better adhesion between fillers and flax fiber with epoxy resin. The lowest SiC (3 wt. %) addition clearly shows that negative effect for composites, both stress and stiffness are strongly decreased. From the results, the tensile strength is suddenly reduced from 46.34 MPa to 29.16 MPa when the percentage of alumina increased from 3% to 5% by weight. It is inferred that the load and stress transfer between the fiber and resin with fillers is highly reduced due to more filler material in the composite damages matrix continuity and more void formation in the composite which is the main reason for the reduction of tensile strength.

The flexural strength is one of the important properties for composites mainly used in quantifying structural applications [14]. Figure 3 shows the variations in the flexural strength over different ceramics filled composites. It is observed that the highest flexural strength is obtained for sample C. The increased flexural strength signifies that SiC particles are homogenously dispersed in the epoxy resin. The flexural strength gets



. Fig 2 - Tensile strength of ceramics filled flax fiber reinforced composites.

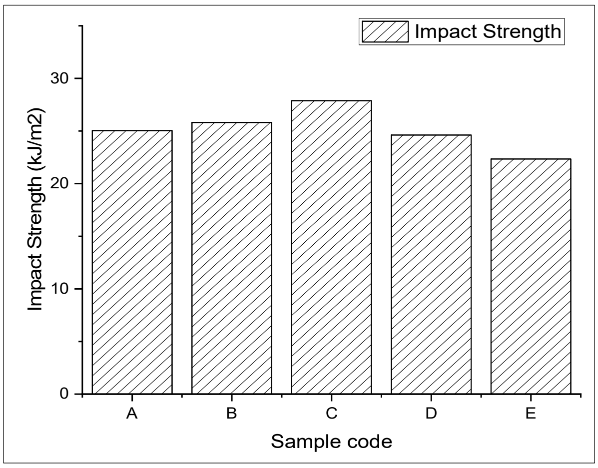


Fig 4 - Impact strength of ceramics filled flax fiber reinforced composites.

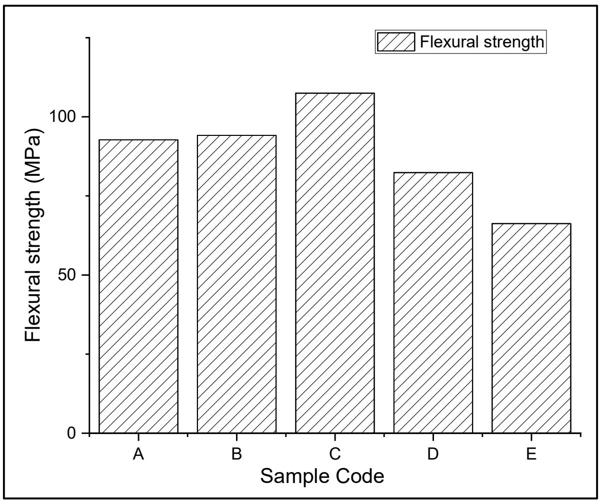


Fig 3 - Flexural strength of ceramics filled flax fiber reinforced composites.

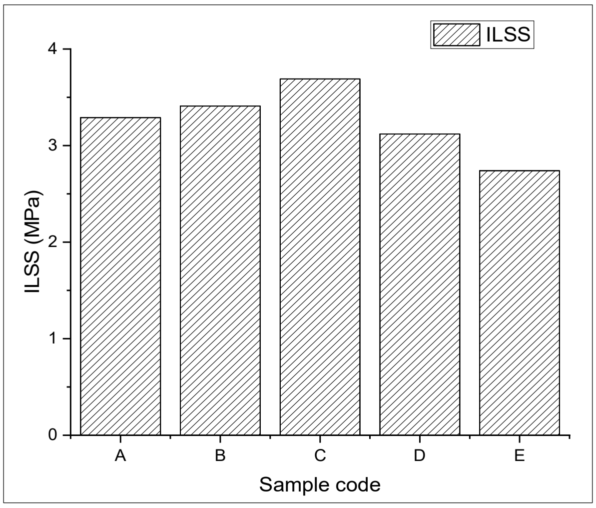


Fig 5 - Interlaminar Shear Strength (ILSS) of ceramics filled flax fiber reinforced composites.

drastically reduced when the weight percentage of SiC reduced up to 3 wt. %. During the composite preparation, if the %weight of the SiC is less than 7%, it leads to insufficient compacting of SiC with epoxy resin which is one of the main reasons for the incomplete composite.

4.2.Impact strength and Inter Laminar Shear Strength (ILSS)

An impact test is carried out for analyzing the fracture toughness of the composites. The impact properties of composite materials are highly influenced by the nature of the constituent materials which is directly related to its overall toughness [15]. The samples were fractured in a charpy impact testing machine. Figure 5 shows the impact strength of ceramics filled composites. From the result, it can be observed that the impact strength is improved for sample C compared to other samples with the addition of 7 wt. %SiC with flax and epoxy resin due to addition of SiC the matrix increases the ability of these composites to absorb impact energy. Addition of more Al_2O_3 with flax fiber reduced the impact strength of the composite. From the formation of voids during compression moulding method, these void content increases the crack propagation, typically a resin with high loading fillers has less ability to carry the impact energy. Higher loading of Al_2O_3 fillers disturb the matrix continuity leading to stress concentration, which can act as crack initiator and reduce the interfacial bonding between fiber and resin and energy absorption capacity of composites.

Inter laminar shear stress happens due to the stresses acting on the interfaces between two adjacent laminae in a layered composite [16]. Figure 4 shows that ILSS of sample C is higher than that of the other samples. This confirms better bond existence between the fillers and flax fiber with epoxy resin. Further, with the incorporation of SiC with flax fiber from 3 wt. % to 7 wt. % increases the ILSS, but in contrast a decrease in ILSS has been observed with increase in Al_2O_3.

4.3.FTIR analysis

The effect of pure and alkaline treatment on different ceramics filled composites are evaluated

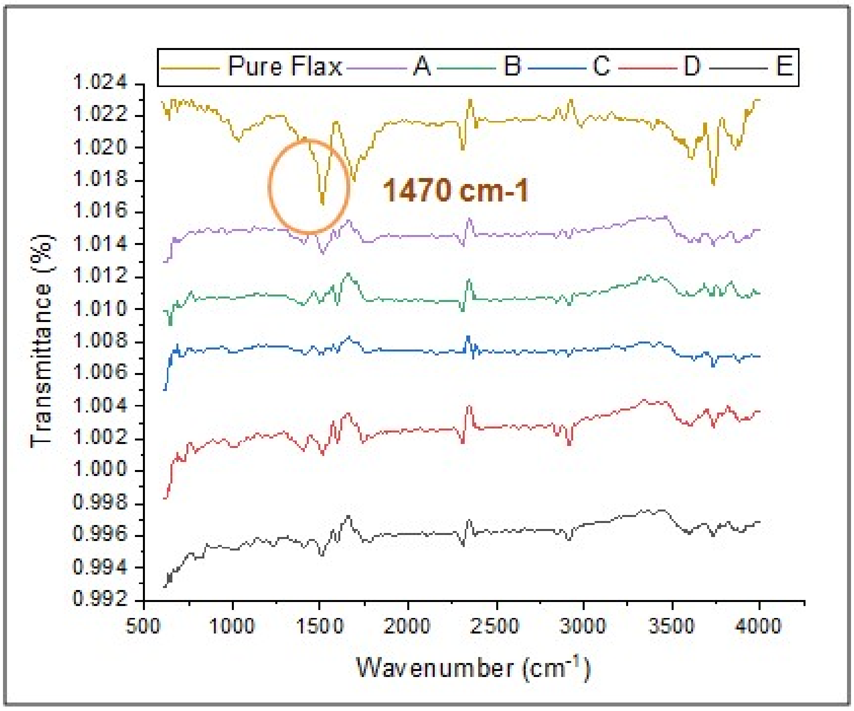


Fig 6 - FTIR analysis curves of ceramics filled flax fiber reinforced composites.

by using FTIR spectroscopy. Figure 6 shows FTIR results for pure flax and alkaline treated flax fiber reinforced composites. A broad absorption band at the 3690 to 3210 cm^{-1} region is characteristic of polymeric association of the -OH groups and hydrogen bonded –OH stretching vibration in carbohydrates and lignin. It is observed that the broadness of the –OH band decreased more for NaOH solution. These results reflect the decrease of hydroxyl groups in the flax fiber after alkaline treatment. From alkaline flax fiber, the intensity of the bands at 1387 cm^{-1} from –OH in plane bending was reduced because of the formation of glycoside bonding. The decrease of the intensity of a band at 1646 cm^{-1} can be explained by the removal of OH bending in the absorbed water molecules by alkaline treatment. The intermolecular hydrogen bond in a phenolic group in lignin is observed at around 3547 to 3564 cm^{-1}. The sharp peaks at 2930 to 2880 cm^{-1} are characteristic bands of the C-H stretching vibration present in the hemi-cellulose and cellulose. In the FTIR analysis of the flax fiber, these bands are more accentuated and treated as an indicative of a larger exposition of cellulose and water on the fiber surface. It is known that more than 60 wt. (%) of the flax fiber is composed of cellulose and more than 20 wt. (%) of the flax fiber is made up of hemi-cellulose and 8-12 wt. (%) is moisture content. From the FTIR spectra, the band at 1470 cm^{-1} exhibits the presence of in-plane CH bending in pure flax fiber whereas this wavelength was not present in alkaline treated flax fiber. This indicates that the removal of hemi-cellulose present in the fiber surface could be carried out by alkaline treatment. The FTIR analysis confirms the chemical effects of alkaline surface modification on the flax.

4.4.SEM analysis

The study of interfacial adhesion, which is a well known problem for natural fibers and synthetic polymers, also shows that adhesion needs to be improved to optimize the mechanical properties of polymer composites [17]. Figure 7 & 8 shows SEM images of tensile test samples. The microstructure reveals uniform distribution of fillers and flax fiber in the epoxy resin. For the composite with ceramics fillers, the compatibility between ceramic particles and flax fiber was expected. However, to obtain the natural fiber reinforced composite with better mechanical properties, the interaction between fiber and resin needs to be improved. The bonding between the ceramics fillers in epoxy resin with flax fiber seemed to be good for sample C. Good adhesion of flax fiber and fillers with epoxy resin certainly results in very high mechanical properties. For sample E, it is clear that the interfacial adhesion between the fibers and resin is poor. Indeed, the fibers are pulled out from the surface and by absence of any physical contact between those components. The morphological changes in the fiber surface are analyzed in removing the hemi-cellulose and lignin by alkaline treatment.

4.5.Prospective applications

The potential application of ceramics filler filled flax fiber composites will acts as the alternate asbestos brake pad materials to offers avoiding of carcinogenic nature and reduction in energy requirement. The produced materials relatively lesser polluting the air, land and during processing, due to their renewable and biological origin. The inclusion of this material in brake pad shows great potential due to their worldwide availability, lower

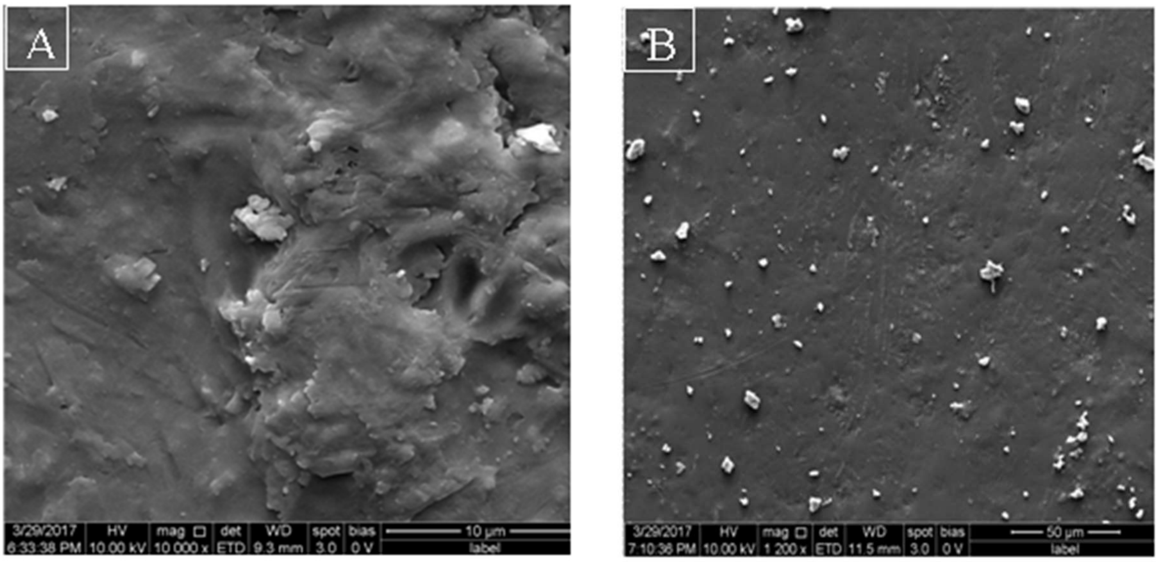


Fig 7 - SEM images of fractured composites. (A) SiC 3 wt. (%) & (B) SiC 7 wt. (%).

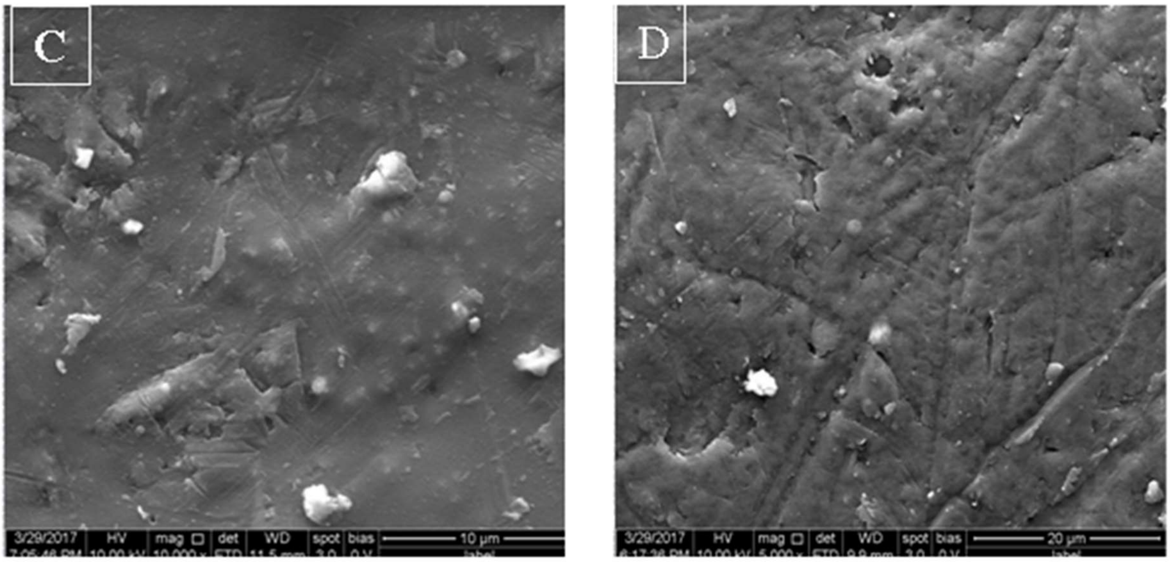


Fig 8 - SEM images of fractured composites. (C) SiC 5 wt. (%) & (B) SiC 6 wt. (%).

raw material cost, fabrication cost, minimal equipment wear and biodegradability in some cases, over synthetic fibers such carbon, boron, aramid and glass. The enhanced mechanical properties of ceramics filled flax fiber composites indicates a high possibility in the brake pad applications where renewable resource and high performance are required, with the inclusion of relatively minimum amount of additives.

5.Conclusion

The mechanical and FTIR characteristics are discussed in details and the results are reported in this paper. From the above investigation, the following conclusions were drawn:

The mechanical properties of composites increase with increase in SiC up to 7 wt. %. Hence the optimum SiC for better mechanical properties of this present composite is found to be 7 wt. %.

Likewise, optimally 5% NaOH treatment of flax fiber highly enhanced the interfacial bonding between fiber and resin. This phenomenon was examined by FTIR analysis and it leads to better mechanical properties related to the flax fiber. The fractured surface of sample C revealed better interfacial bond between flax fiber and epoxy resin, less voids and absence of fiber pull out when compared to other samples which was confirmed by SEM analysis. Thus it can be concluded that the obtained composites will acts as a low cost, lightweight and eco friendly composites to be used for a brake pad, on account of their better mechanical properties.

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