

# Mechanical Properties and Delamination Factor Evaluation of Cellulose (Nettle) Fiber Reinforced Polymer Composites using RSM

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## Research Article

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# Abstract

This paper discusses on fabrication, testing and evaluation of delamination factor of nettle fiber based composites for low duty applications. The randomly oriented nettle fibers were used to fabricate the biocomposite by conventional hand lap up technique. Epoxy and Nettle based composite plates were developed by varying fiber weight percentage from 5% to 25%. The flexural, tensile, impact, chemical resistance and water absorption rate of developed nettle fiber based biocomposite were examined for different fiber weight fractions in the randomly oriented patterns as a unique and innovative attempt. During the investigation, flexural strength and tensile strength were improved up to 20 wt% of fiber addition and then it was decreased. This resulted in a continuous rise in impact strength with an enhancement in fiber wt. %. The influence of fiber weight percentage on water absorption and chemical resistance of fabricated composite was examined in different environments. The result showed that the nettle fibers can be used as an essential reinforcing material to design and fabricate mechanical and structural members for low duty application. The chemical behavior of nettle based composite was studied by the FTIR spectroscopy method and the presence of chemical functional group was confirmed. The drilling behavior of developed nettle/SiC/epoxy hybrid composites was evaluated by consider cutting process parameters like feed rate (0.125, 0.212 and 0.3 mm/rev), spindle speed (400, 600, 800 rev/min) and drill diameter (4, 6, 8 mm). Analysis of variance was used in designing experiments for the current investigation. Feed rate was found to be a very impressive factor in influencing the delamination factor.

## Introduction

Natural fiber composites have evolved as a lightweight and good strength material at the starting of twentieth century and their popularity has increased in recent time for various applications [Gholampour and Ozbakkaloglu 2020]. The growth of natural fibers was seen in a very short period and various industries like automobile, sports and aerospace were in a race to utilize its potential. India is a great resource of natural fibers and many researchers are working to utilize its potential to develop structural and mechanical members of biocomposites. India holds second place in terms of bamboo resources in the world and scientists are continuously working to develop bamboo fiber composites (Dev et al. 2020). Plant based fibers are categorized into three types namely mineral fibers, animal fibers and plant fibers: amongst these, plant fibers are famous for reinforcement in polymer resins. Figure 1 shows the graphical abstract of fiber and epoxy and its fabricated samples.

Fibers from plants can be obtained from leaves, stems and stalk through chemical or mechanical extraction techniques (Paul et al. 2020). Plant fiber like nettle, jute, hemp and flax were traditionally used to make ropes for domestic applications from ancient times. In the 21st century, nettle was used as a raw material for clothes and paper making. Nettle

fiber is considered as the best reinforcement constituent in thermoplastic because of its higher relative comparative strength as compared to other plant based fibers. Many experimental and analytical studies have revealed that the inclusion of nettle fibers as filler member will improve the mechanical performance

of the neat polymers (Buyukkaya and Demirer 2020). The presence of cellulose and hemicellulose in nettle fibers are the main responsible factors for higher strength and hydrophilic nature respectively; also the presence of lignin and pectin yields thermal stability and biodegradability to the fibers (Viju and Thilagavathi 2019) The interfacial linkage between natural fiber and polymer resin plays an important role in deciding the mechanical and thermal performance of the natural fiber composites. Since the natural fibers are obtained from natural process, so they have inherent moisture quantity. Due to the moisture content natural fibers have poor wettability with polymers, which need to be improved by various chemicals techniques (Panchal et al. 2018). Tensile and flexural strength of biocomposites improves with the addition of fiber content up to a certain limit and beyond that limit it decreases. Uttarakhand state in India, possess a variable physiographic diversity which makes it fertile land for various fiber plants. Himalayan nettle grows up to a height of 3 meters and is found in various places in Uttarakhand state in India. Fibers extracted from Himalayan nettle plants looks like a silky white thread which can be used for various application like clothing and domestic purpose. There was a time when nettle fiber was considered as a discarded articles in Uttarakhand but now a days it is harvested by local communities for making crafts, textile products, to enhance their economic and financial conditions (Srivastava and Rastogi 2018).

Many researchers have developed epoxy based composite added with plant fibers and has shown that the mechanical and thermal performance of natural fiber added composite are better as compared to the unreinforced materials (Adesina et al. 2019) An enhancement in tensile and impact strength was reported with the reinforcement of woven Himalayan nettle fibers into polyester resin. The highest tensile strength of 31.39 MP was observed for 15 wt% fiber content; while the maximum impact energy of 29 joules was observed for 20 wt% fiber filling (Pokhriyal, Prasad, and Rauri 2018). The other aspect of natural fibers were observed where a decrease in young's modulus and tensile strength of single nettle fiber was observed when the fiber diameter was increased but overall outcomes were good in terms of biodegradability and better mechanical performance (Mahendrakumar et al. 2015). The mechanical properties can be improved via introducing particulate reinforcement into natural hybrid composites. The flexural strength and tensile strength and increased when a hybrid composite of kevlar and sisal was introduced with nano silica (Chowdary et al. 2020). In another study, when silicon carbide content was varied from 5–15%: a significant enhancement in tensile strength and hardness was detected for jute based composite (Patnaik and Nayak 2018). The effect of jute fiber as a filler material used in epoxy resin was studied to evaluate the chemical form and mechanical properties through Fourier-transform infrared spectroscopy. Natural fibers showed hydrophilic nature which limited their use in higher load and marine applications, but still, ample research is occurring on to reduce moisture absorption behavior of the plant fibers. The phenomena of moisture absorption was studied when sisal fibers were reinforced into polypropylene (Maurya et al. 2021). The moisture absorption was high for natural fiber based composite as compared to the synthetic fibers (Chandramohan et al. 2019). A comparative study of water absorption in composites of flax/epoxy and Jute/hemp/epoxy was done and it was found that flax fiber based composite had low water absorptions as compared to the Jute/hemp fiber based composites (Chaudhary et al. 2020). Water absorption can be decreased by chemical treatment of fibers and by

controlling composites fabrication parameters. Reduction in water absorption was reported when the Luffa cylindrical fiber based composites were processed at higher temperatures (Pires et al. 2020). The water absorption rate was high for untreated juliflora fiber and decreased when NaOH concentration increased from 5–15% (Reddy et al. 2019). It has been reported that the moisture absorption rate for a hybrid composite of kenalf and sisal was 3.2% while for kenalf fiber it was 5.3%. Thus the development of hybrid composite can reduce the water absorption rate for marine applications (Ramasubbu and Madasamy 2020). Through literature findings, it has been concluded that very little work has been presented in fabrication of nettle fiber based composites. The prediction and evaluation of mathematical modelling for drilling of SiC/nettle/ epoxy hybrid composite is not reported by any researcher yet. Therefore nettle fiber based composites were fabricated to study the mechanical performance and drilling behavior of developed composites for this this research work.

## Materials And Method

The Fabrication of natural fiber composites is as follows

### Material used

In the presented research study, epoxy LY 556 was used as a matrix and hardener HY 991 as a curing agent. The density of epoxy was  $1.3 \text{ g/cm}^3$ . Nettle fiber was used as the reinforcement into epoxy resin which was obtained from the Utrakhand bamboo development board, India. To create better bond strength, epoxy and hardener was used in the ratio of 10:1. Nettle fibers which were obtained in random oriental format from bamboo development board are shown in figure 1 (b). High duty silicon spray was used as the releasing agent.

### Composite Fabrication

Five different composites were fabricated by changing the fiber and resin weight percentage. All the sampled were fabricated through the reinforced of untreated nettle fibers. The weight proportion of matrix and epoxy resin is specified in Table 1.

Table 1

Composition Structure of different composites

Samples	Weight of resin (%)	Weight of nettle fiber (%)
Z1	95	5
Z2	90	10
Z3	85	15
Z4	80	20
Z5	75	25

The conventional hand layup practice was performed to prepare the composite specimens. Epoxy and hardener were mingled properly in the ratio of 10:1 and was slowly stirred to remove the air bubbles formed while mixing. The short randomly oriented fibers were placed inside the mixture of epoxy resin and were thoroughly mixed for better wettability. The dimension of the steel mould was 250 x 200 x 4 mm which was designed in solid works and fabricated by welding the steel plates to the desired size. Teflon sheets were placed inside the steel mould and high duty silicon spray was used to avoid sticking of the epoxy with mould. The mixture of nettle fiber and epoxy was then carefully poured inside the mould. It was then gently spread precisely inside the mould and rollers were used for removing the air bubbles. Teflon sheet sprayed with high duty silicon was then placed on the top and covered with a steel plate. A load of 150 N was placed on the whole assembly and was retained for curing up to 24 hours. After complete curing, the composite was detached from the mould and for each weight fraction ratio, three specimens were fabricated and its average values were considered. The fabrication of composite was done by varying fiber weight percentage as 5%, 10%, 15%, 20% and 25%. Composites (Z1-Z5) of different composition are prepared. Developed composites for various testing are shown in figure 1 (c). The samples for tensile, flexural, impact and water absorption testing were prepared according to ASTM standards. Composites were converted into powders for studying its chemical form through fourier transform infrared (FTIR) spectroscopy.

#### Mechanical characterization

The natural fiber based composite can be fabricated by various processing techniques according to the designed size and use. The most important aspect is to check its quality and mechanical properties so that it can perform efficiently without failure at the desired loading conditions. The various mechanical testing adopted for the developed composites is explained further.

#### Tensile strength testing

When a mechanical member is applied with an axial tensile load, it is expected from that member to withstand those loading conditions. This property named tensile strength can be evaluated by tensile testing of standardly prepared samples. For the present research work samples were prepared as per the ASTM D638 standard and tested on the universal testing machine.

#### Flexural strength testing

Flexural strength is one of the important properties to be evaluated for mechanical members under bending conditions. To measure flexural strength of prepared composite for this study has been evaluated using 3 point bend test and samples were prepared as per the ASTM D790 standard. The flexural strength test was done at room temperatures and the average value of three samples were taken as its final flexural strength.

#### Impact strength testing

The toughness of prepared samples were calculated by performing an impact test on the impact testing machine. Specimens were fabricated by following ASTM D256 standards and were subjected to impact

loading. Three impact test for each fiber wt. % were performed and the average was considered as the final value.

#### Fourier Transform Infrared Examination (FTIR)

FTIR is one of the popular and best technique to validate the dispersion of functional group and bond category in the composites. The chemical form of composites can be easily examined through FTIR technique. To perform this examination powder form of the prepared composites was placed under the instrument and the results have plotted a graph.

#### Water Absorption rate

Water absorption performance of nettle fiber reinforced polymer composite was studied according to ASTM D570-98 standards. Samples with different fiber loading were dipped inside the glass of water to observe the water absorption characteristics. The change in weight of samples was measured after equal intervals of 24 hours. The samples were cleaned properly with tissue paper before weighing in precise electronic measuring equipment. The water intake of the composite samples was calculated by following formulae.

$$\text{Water absorption percentage} = \frac{W1 - W2}{W2} \times 100$$

Where W2 represented the initial weight of the sample before water dipping and W1 represent the final weight of the sample after water dipping.

#### Chemical Resistance

The natural fiber composites should have the capability to work under severe conditions and impart long duration functional properties for practical applications; therefore, it was very important to study the effect of distinct environmental conditions on biocomposites and many researchers are examining composites under various environmental conditions. In this respect, to design and fabricate a commercial storage tank of composite material for chemicals, the evaluation of chemical resistance is obligatory. In this study chemical resistance test of untreated nettle plant fiber reinforced polymer composites was done according to ASTM D543-87 standard. In each investigation, the samples were weighed and dipped in an aqueous solution of KOH (10%), NaHCO<sub>3</sub> (10%) and NaOH (10%). The specimens were removed from the aqueous solution and cleaned properly to remove any water droplet adhered to the composite surface. Then the composites were measured precisely to calculate the weight loss/gain (Ashok et al. 2010). All the experimentation was done at room temperature and three specimens of each weight% were studied. The chemical resistance behavior of the prepared composite specimens was calculated by the following formulae.

$$\text{Chemical resistance percentage} = \frac{C1 - C2}{C1} \times 100$$

Where C1 denoted the initial weight of the specimen before chemical dipping and C2 denoted the final weight of the specimen after chemical dipping.

#### Evaluation of delamination factor

All drilling operations were performed on drilling machine to analyze the delamination factor during drilling operation of SiC/nettle hybrid polymer composites. The hybrid composite was fabrication by reinforcing 3% SiC with nettle fibers in the epoxy resin. The dimensions for drilling operation were selected as 180 mm x 90 mm x 6 mm. High speed steel drill with variable diameters (4mm, 6mm and 8mm) was used to perform the drilling operation. The three factors namely, drill diameter, spindle speed and feed were selected at three levels each during this investigation as shown in Table 2. Drilling was accomplished dry, at spindle speeds of 400, 600 and 800 mm/rev and three feed rates of .125, .212 and .3 mm/rev.

Table 2

Design of Experiments

Sr. No.	Factors	Notation	Units	Levels		
				-1	0	1
1.	Drill diameter	D	mm	4	6	8
2.	Spindle speed	N	rev/min	400	600	800
3.	Feed	F	mm/rev	0.125	0.212	0.3

The samples with drilled holes were scanned with digital microscope and obtained images were imported to imageJ software. The threshold feature was adjusted to measure the delamination around the hole. Two concentric circles were drawn to divulge delamination around the hole. While drilling plant fiber based polymer composites, the delamination is one of the predominant defect. The delamination factor for the evaluation of damage was evaluated via the given equation:

$$F_d = D_{max}/D$$

Where  $D_{max}$  is the enlarged diameter due to drilling and  $D$  is the nominal diameter.

## Results And Discussions

The results and discussion on the conducted experimental study is discussed further.

## Tensile strength testing

The practical applications of different natural fiber composites is possible when they exhibit the desired tensile strength and tensile modulus properties. The optimum filler content is very important to find out the lower or higher filler contents and their deterioration in respect to the load carrying performance of composites. At lower filler content, the mechanical performance of biocomposites is low because of its lower loading capacity and at larger filler content failure occurs because of the occurrence of higher voids and low fiber/matrix wettability. The tensile strength of the composites was calculated from the tensile

test and the result values are shown in Figure 2. It was concluded from the results that specimen number four (20% nettle fiber and 80% epoxy) had superior tensile strength (23.26 MP) as compared to other samples. The lowest value of tensile strength was recorded for the first sample (5% nettle fiber and 95% epoxy). The tensile strength of composite with 20% nettle fiber was 86.08% greater than the composite with 5% nettle fiber. The maximum tensile strength of the developed composite of this study was 159.12% greater than the pineapple fiber based composite (Sivasubramanian et al. 2020). It is perceived from the tensile strength results that the tensile strength continued to increase with a rise in the fiber wt%. This improvement in tensile strength retained up to 20 wt% and after that reduced, which specified that the superior stress transfer takes place between fiber and epoxy for optimum fiber reinforcements (Rashid et al. 2016). The decrease in tensile strength above 20% was because of the poor wettability between polymer and fibers as the inadequate flow of polymers occurs around the fibers (Pankaj et al. 2021)

## Flexural strength testing

The flexural strength of nettle fiber added epoxy composites specimens were evaluated using 3 points bending and the trend are displayed in figure 3. Flexural strength is combined properties of shear and compression. The same pattern was followed in tensile strength as it was observed in flexural strength for variations in weight percentage of fibers. After 20 wt % of fiber addition, a drop in flexural strength was seen. During the fabrication of composites, various defects were observed. These defects formed stress concentration sites at fibers- ends and higher concentrations at these sites was the main cause of crack propagation due to its failure. As discussed earlier in the tensile strength segment, at higher content of fiber (20 wt. %) the resin was unable to cover the entire surface area of fiber and hence caused a reduction in the flexural strength. The presence of voids, faults in composite processing and lack of interfacial strength were also the common reasons for the drop in its flexural strength (Suriani et al. 2021). As the fiber addition (up to 20 wt.%) increased the flexural strength and after that it decreased; similar results were also reported for different fiber content when jute, sisal and elephant grass based composites were fabricated (Gunti et al. 2018). The increase in fiber concentration also made the composites brittle, because of the irregular load distributions between fiber and polymers. The flexural strength primarily affected by the binding efficacy between the matrix and its reinforcement. Poor binding efficacy induces lack of flexural strength between the polymers and its filler content (Harish et al. 2009).

## Impact strength testing

The impact strength is the material capability to endure high speed stress or load, the experimental results showed that with a rise in the reinforcement of nettle fibers in the epoxy resin, it improved the impact strength. When plastic deformation of composites took place then energy storage capacity increased in higher fiber contents because larger energy was required to break the fiber bundles (Devireddy and Biswas 2017). The results of the impact strength of nettle fiber based biocomposites for the current investigation are displayed in figure 4. It was perceived from the results that the impact strength of biocomposite with 25 wt % was held the highest toughness as compared to other samples. The impact strength of sample Z5 is 7.21 KJ/m<sup>2</sup> which was 71.66% higher than sample Z1. The same

pattern of increase in impact strength has been observed by the researchers when untreated luffa fiber based composites were fabricated and the composites in the current investigations had higher impact strengths (Mohanta and Acharya 2016). This impact strength can be increased by different modification technique like the chemical modification of natural fibers which enhanced the binding strength between reinforcement and polymer, a developing area in which many researchers are working (Debeli et al. 2017) (Kansal et al. 2020).

## Water Absorption

The water absorption test of biocomposites is an essential parameter to be evaluated when the composite is exposed to marine environments. In the current study, water absorption behavior was studied for the same environmental conditions in the transient mode. The main parameters which affect the water resistance properties were the amount of fiber contents, presence of hydroxyl groups and voids. The influence of dipping time on water absorption rate in terms of moisture gain

is illustrated in figure 5. An enhancement in water absorption rate was noticed while increasing the nettle fiber filling, which represented the presence of a higher hydrogen bond between nettle fibers with the retention of water molecules. The cause for the increase in water absorption seen in this study could be because of the occurrence of micro cracks and fiber's nature to absorb water and voids. The same pattern of results was observed by many researchers during their previous studies (Gupta 2020) (Ramesh et al. 2020) (Prakash et al. 2018) (Panchal et al. 2018)). The composite samples fabricated with epoxy and nettle fiber proportion was 75:25 and they showed higher water absorption rates than 85:15, 90:10 and 95:5. The matrix material chosen was Epoxy which showed water resistant properties and nettle fibers depicted water intake properties. The maximum of 10.96% water intake rate was reported for biocomposite made up of 25% nettle fiber and minimum of 2.01% water intake rate was reported for biocomposite made of 5% nettle fiber. When we require low water absorption the sample Z1 was best but in such cases, a compromise with the mechanical properties needs to be done.

## Chemical Resistance

Chemical resistance is a scientific technique used to find out the capability of composites to survive the when exposed to alkalis and chemicals. The chemical resistance of fabricated composites was accomplished to find out suitability for making chemical

resistant articles. Chemical resistance of different composites for KOH (10%), NaHCO<sub>3</sub> (10%) and NaOH (10%) in the graphical form is illustrated in figure 6. From this figure, it is clear that the weight gain was detected for all samples with different weight contents. It indicated that the prepared composites did not suffer any erosion as there was no weight loss. From another perspective view of point, the fibers did not show any chemical resistance and this chemical resistance reduced with a rise in the fiber content as a large number of chemicals came in contact with the chemicals. A higher absorption rate corresponds to the rich interaction between the fibers and aqueous solutions. Conversely lower absorption rate corresponds to poor interactions between the fibers and aqueous solution which is desirable for

designing composite materials. Amongst all samples, sample Z5 showed maximum absorption rate and this absorption rate was absorbed maximum for the aqueous solution because fibers showed hydrophilic behavior for aqueous solutions. Such findings of an increase in absorption was stated in the case of bamboo fiber filled epoxy composites (Gupta 2016).

## FTIR analysis

The mechanical properties of biocomposites decreases because of the moisture absorption characteristics of natural fiber composite due to the hydrogen bond formation between the water molecule and natural fibers. The nettle fibers were hydrophilic in nature and held hydroxyl groups in fiber structures. When the percentage of O-H group was higher in fibers, then fibers showed a very attractive nature for water molecules and the overall moisture resistance decreased. This lead to the decrease in overall mechanical and thermal performance of the biocomposites (Hestiawan 2020). FTIR is a vital tool to examine the functional group of natural fiber and biocomposites. The FTIR graph was divided into two groups: function group and fingerprint group. FTIR spectra of nettle fiber reinforced epoxy composite in powder form was taken with FTIR spectrometer in the wavelength range  $500\text{-}4000\text{ cm}^{-1}$  and the same is revealed in figure 7. High intensity and broader peaks near  $3500\text{ cm}^{-1}$  represent stretching vibrations of hydroxyl groups for the tested powder sample of composites and this had been also represented by the FTIR analysis of the composite where the peaks formed between  $3600$  and  $3000\text{ cm}^{-1}$  was consigned to O-H stretching (Celino et al. 2013). This peak confirmed that the natural fibers had higher contents of O-H group and could easily absorb moistures. This peak can be decreased or we can decrease the percentage of O-H group by chemical treatment of natural fibers (Jayamani et al. 2020). The band near  $1600\text{ cm}^{-1}$  shown in figure 7 are because of the presence of water content in the fabricated nettle fiber based composites which was specified by the peak formation between  $1693$  and  $1607\text{ cm}^{-1}$  for the representation of water in natural fibers (Ray and Sarkar 2001). The peaks near the  $2800$  to  $3000\text{ cm}^{-1}$  were because of C-H stretching vibration in cellulose and hemicellulose which were also reported when jute fibers were examined (Saha et al. 2010). The peak formed between  $1100$  and  $1200\text{ cm}^{-1}$  denoted the bending present in the cellulosic and bonding structure of fibers (De Rosa et al. 2010).

## Effect of machining parameters on delamination factor

The delamination factor was evaluated after performing experiments that were designed according to box-behnken design in the design expert software. The responses obtained during the investigation are shown in Table 3. A quadratic model was statistically suggested by the design expert software when experimental responses were analyzed in the design expert software. During the investigation, it was observed that the quality and appearance of drilled hole was significantly affected by the drilling conditions. The delamination phenomenon was predominantly intensive at the exit of the hole as compared to the entrance observed for various natural fiber based composite plates (Belaadi et al. 2020) (Belaadi et al. 2019). Therefore, the current study was focused on the prediction of delamination behavior at the exit only. The characteristic curves revealing the influence of the spindle speed, feed rate and drill diameter on delamination factors were plotted to evaluate the delamination damage that occurred during

the drilling operation. Delamination was evaluated for the developed hybrid composites reinforced with SiC particulates. The state of the hole after at drilling conditions of spindle speed 800 rev/min, drill diameter 6 mm and feed rate 0.3 mm/rev is shown in figure 8. The maximum delamination factor during this investigation was observed as 1.301 (for the drilling conditions of spindle speed 600 rev/mm, feed rate 0.3 mm/rev and drill diameter 6 mm). The minimum delamination factor during drilling operation in the current study was observed as 1.096 (for the drilling conditions of spindle speed 800 rev/mm, feed rate 0.125 mm/rev and drill diameter 6 mm). The delamination factor at the exit of the hole creates various research opportunities for engineers, scientists and researchers; therefore, it is very difficult to compare the research work of this investigation with the literature because many experimental studies showed that the delamination factor gets affected by the composite manufacturing process (Rezghi et al. 2019), tool type and geometry (Suriani et al. 2021) and selection of machining parameters (Malik et al. 2021). For comparison, the delamination factor during the drilling of jute/polypropylene was found to be varied from 1.23-2.09 (Pailoor et al. 2021), while for the current investigation delamination factor was varied from 1.096- 1.301. The output response as a delamination factor of our experimental study was higher than the delamination factor of drilling of the flax/polylactic acid (Lotfi et al. 2018).

## Response surface methodology

Response surface methodology (RSM) is a useful technique that is used to predict and model the problems in which response is affected by the input parameters and influencing parameters are optimized to get the desired response. The major mechanism of RSM consists of: (1) performing experiments and collecting data according to the experimental design (2) approximating the correlation between factors and responses by empirical modelling. (3) Empirical model based optimization to obtain the best response. These models were used by many researchers in the estimation and optimization of response and process variables in the drilling of fiber reinforced polymer composites. Response surface methodology using Box-behnken design was employed during the experimentation work to get a quadratic model consisting of 17 experiments. The experimental response in the terms of delamination factor was taken as an average value of two responses. The three independent process parameters and levels, drill diameter (D), feed rate (F) and spindle speed (N) were selected as displayed in Table 2. The empirical second-order polynomial equation was established for the delamination factor on the basis of experimental results shown in Table 3. The mathematical equation developed during experimentation by RSM is given below.

$$F_d = 1.37544 - 9.30357 \times 10^{-3} \times D - 4.61964 \times 10^{-4} \times N - 0.86531 \times F - 1.87500 \times 10^{-5} \times D \times N + 0.031429 \times D \times F - 5.14286 \times 10^{-4} \times N \times F + 2.00000 \times 10^{-3} \times D^2 + 3.62500 \times 10^{-7} \times N^2 + 3.91837 \times F^2$$

## Analysis of variance for delamination factor

The significance of each process variable and their interactions are analyzed through analysis of variance (ANOVA) details derived from the factorial investigational results. The sum of the square, probability, F-value, degree of freedom and mean square is shown in ANOVA Table 4. The F-value is helpful in predicting the qualitative influence of each process variable and its effects. When any process

parameter exhibits a larger F value then it means, that factor has a larger effect as compared to the error variance. On another aspect, the smaller p value specifies the significant effect of the process parameter on response.

Table 3  
Results of delamination factor

Experimental number	Input variables			Output variable
	D (mm)	N (rev/min)	F (mm/rev)	F <sub>d</sub>
1	6	400	.300	1.301
2	8	800	.212	1.119
3	6	600	.212	1.149
4	4	800	.212	1.099
5	6	800	.300	1.201
6	6	600	.212	1.112
7	6	800	.125	1.096
8	6	600	.212	1.151
9	6	600	.212	1.152
10	8	600	.300	1.269
11	8	400	.212	1.251
12	8	600	.125	1.143
13	4	600	.300	1.212
14	4	600	.125	1.108
15	6	600	.212	1.161
16	6	400	.125	1.160
17	4	400	.212	1.201

Table 4 showing the linear coefficient process parameters (D, N and F) and quadratic term coefficients (F<sup>2</sup>) were significant as they showed fewer P values (Prob. 0<.005). The other process parameter and their interactions were not significant because they had a larger p value (Prob. >.005). From Table 4, it could be wrapped up that feed rate (F) was the most influencing parameter for the delamination factor while drilling developed composites during this investigation. The next effective independent process parameter is the spindle speed (N). The diameter of drill (D) was found to be a very less effective influencing parameter. The coefficient, R<sup>2</sup>, also known as the coefficient of determination is the measure

of the fit degree in the analysis of variance (ANOVA) tables. The  $R^2$  provides a good correlation between predicted and experimental results when it is nearing unity.

Table 4  
ANOVA for delamination factor

SOURCE	SS	DF	MS	F-VALUE	P-VALUE	
Model	0.057	9	6.380E-003	20.76	0.0003	Significant
D	3.281E-003	1	3.281E-003	10.68	0.0137	
N	.020	1	.020	64.44	< 0.0001	
F	0.028	1	.028	92.17	< 0.0001	
D x N	2.250E-004	1	2.250E-004	.73	0.4205	
D x F	1.210E-004	1	1.210E-004	.39	0.5502	
N x F	3.240E-004	1	3.240E-004	1.05	0.3387	
D <sup>2</sup>	2.695E-004	1	2.695E-004	.88	0.3802	
N <sup>2</sup>	8.853E-004	1	8.853E-004	2.88	0.1334	
F <sup>2</sup>	3.789E-003	1	3.789E-003	12.33	0.0098	
Residual	2.151E-003	7	3.073E-004			
Lack of Fit	7.050E-004	3	2.350E-004	0.65	0.6232	Not significant
Pure Error	1.446E-003	4	3.615E-004			
Cor Total	0.060	16				
R <sup>2</sup> = 0.9639						
R <sup>2</sup> adjusted = 0.9175						
R <sup>2</sup> predicted = 0.7727						
Adequate precision = 16.252						

It is very essential to inspect the viability of the modal of the ANOVA table and results of delamination damage for various process factors that are displayed in Table 4. The modal F-value of the ANOVA table was predicted as 20.76 indicating that the modal was significant. The  $R^2$  adjusted value was 0.9175 and it was in good agreement with  $R^2$  predicted of 0.07727. SEM images of cut section and top view of drilled nettle fiber based hybrid composites is shown in Figure 9 and 10 respectively.

### Surface plots for delamination factor

The relative influence of process factors in 3D surface plots is displayed in figure 8. Since the modal is adequate, therefore the estimation of delamination factor for input parameters namely drill diameter, feed rate and spindle speed can be done by using these 3D surface plots. The modal predicted in this experimental investigation had three process parameters, each 3D plot was created by holding one variable constant at the central level. Figure 3 (a) shows the evolutionary relationship between spindle speed and drill diameter, while the feed rate was kept constant. It is clear from the figure that drill diameter has a substantial influence on the delamination factor as compared to the spindle speed. Figure 3 (b) illustrates the impact of drill diameter and feed rate on the delamination factor. The figure depicts that feed rate and drill diameter had a substantial influence on the delamination factor. The delamination factor was witnessed to be increased with the rise in feed rate and drill diameter. It has been noticed that, feed rate was more significant as compared to drill diameter. The larger feed rate escalates the thrust force in the machining area and the delamination factor increases which is not a desired effect among biocomposites while drilling. Therefore it is necessary to drill biocomposites at a lower feed rate. The 3D surface plot for the delamination factor at constant drill diameter and variable feed rate and spindle speed is presented in figure 3 (c). It is clearly illustrated by the figure that, feed rate increases the delamination factor and spindle speed decreases the delamination factor. This happens because larger forces are required for chip removal as a larger feed rate increases the region of the sheared chip.

## Conclusions

Investigations of the influence of filler loading on mechanical properties and chemical resistance of nettle fiber based composites have been done successfully and stated in this paper. During the experimental investigation, it was noticed that

- Tensile and flexural strength of developed biocomposites enhanced up to 20 weight% of filler loading and above that, it reduced.
- In the case of impact strength, a continuous surge in strength was observed.
- It was observed that tensile and flexural strength enhanced by 1.86 times and 1.84 times respectively for 20% fiber content as compared to 5% fiber content.
- The impact strength increased by 71.66% for 20% fiber content as compared to 5%.
- Because of the hydrophilic nature of nettle fibers a gradual increase in water absorption rate in composites was observed and an approximate constant pattern was observed for water immersion from 192 to 240 hr.
- When composites were placed in different environments least and maximum absorption was observed for  $\text{NaHCO}_3$  and  $\text{NaOH}$  respectively.
- No composite erosion was found and the trend for weight gain was observed.
- The presence of a chemical functional group was confirmed when composite was observed by FTIR analysis.

- Delamination factor in the drilling behavior in the SiC/nettle fiber hybrid composites was found in the range of 1.096 – 1.301.
- Feed rate was observed as the most effect parameter in delamination factor of SiC/nettle fiber hybrid composites.

The finding of this the conducted experimental study was that natural fibers were good reinforcing elements in polymers and can emerge as environmentally friendly materials for various engineering fields like sports, automotive and aerospace for designing interiors as well as exterior components.

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**\*Authors' contributions:** All authors have done equal contribution.

**\*Compliance with ethical standards:** Yes

**\*Consent to participate:** Yes. All equally participated.

**\*Consent for Publication:** Yes granted

**\*Availability of data and material (data transparency):** Not applicable

**\*Code availability (software application or custom code):** Not applicable

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## Figures



**Figure 1**

(a) Epoxy and Hardener (b) Nettle Fiber (c) Fabricated composites for various testing

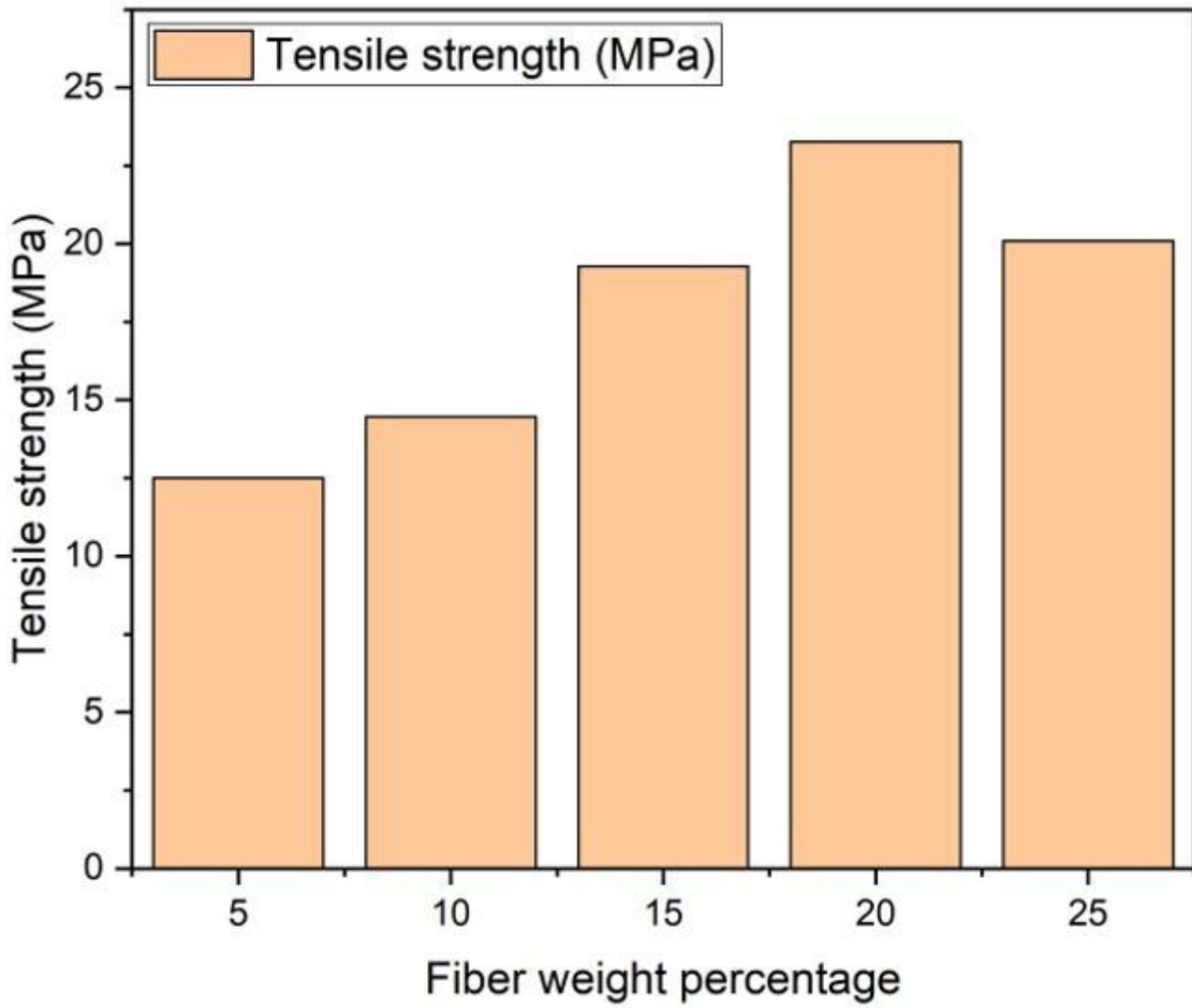


Figure 2

Tensile strength of nettle fiber based composites for various fiber loading

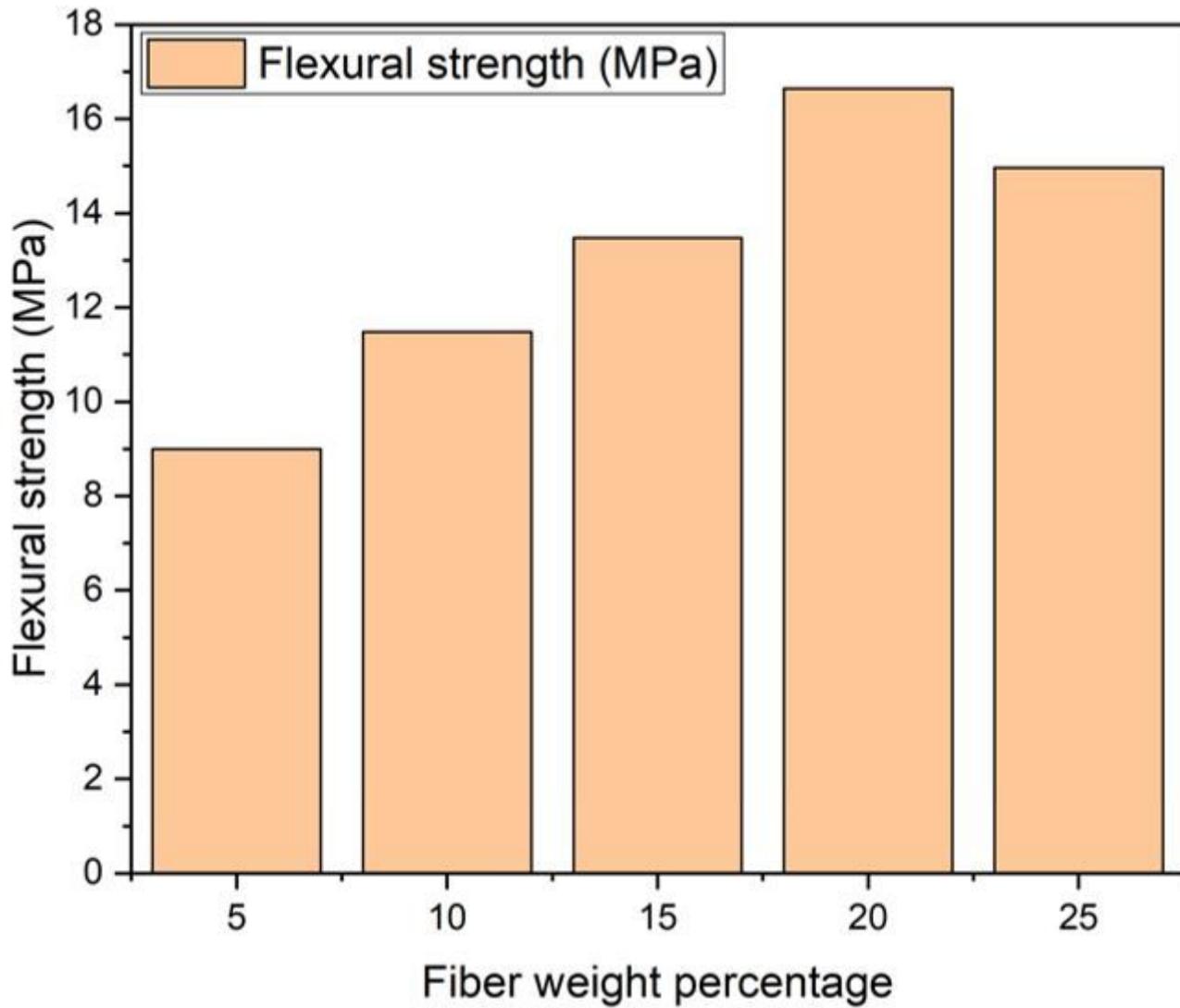


Figure 3

Flexural strength of nettle fiber reinforced composite for different fiber loading

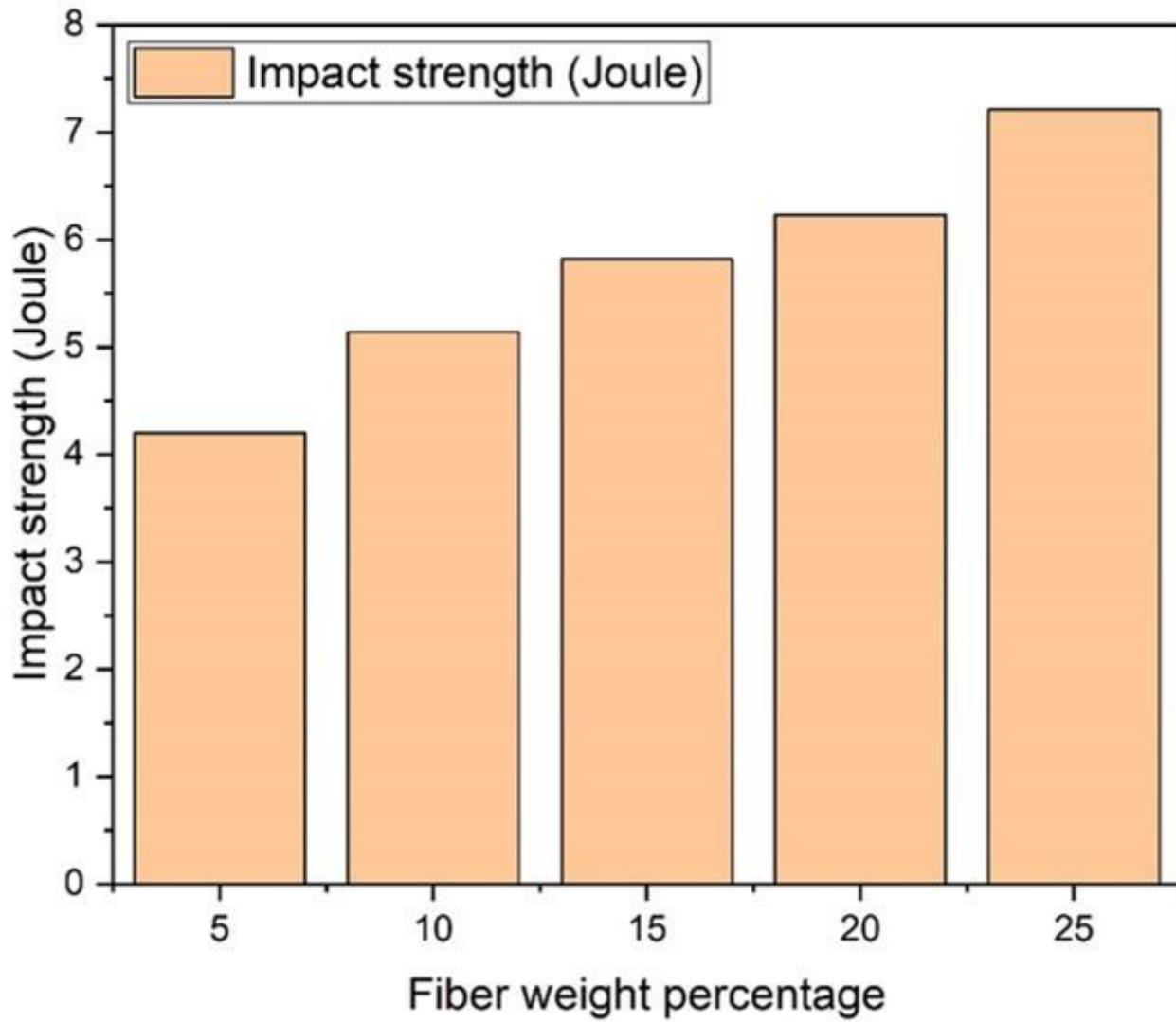


Figure 4

Impact strength of nettle fiber based biocomposite for various fiber loading

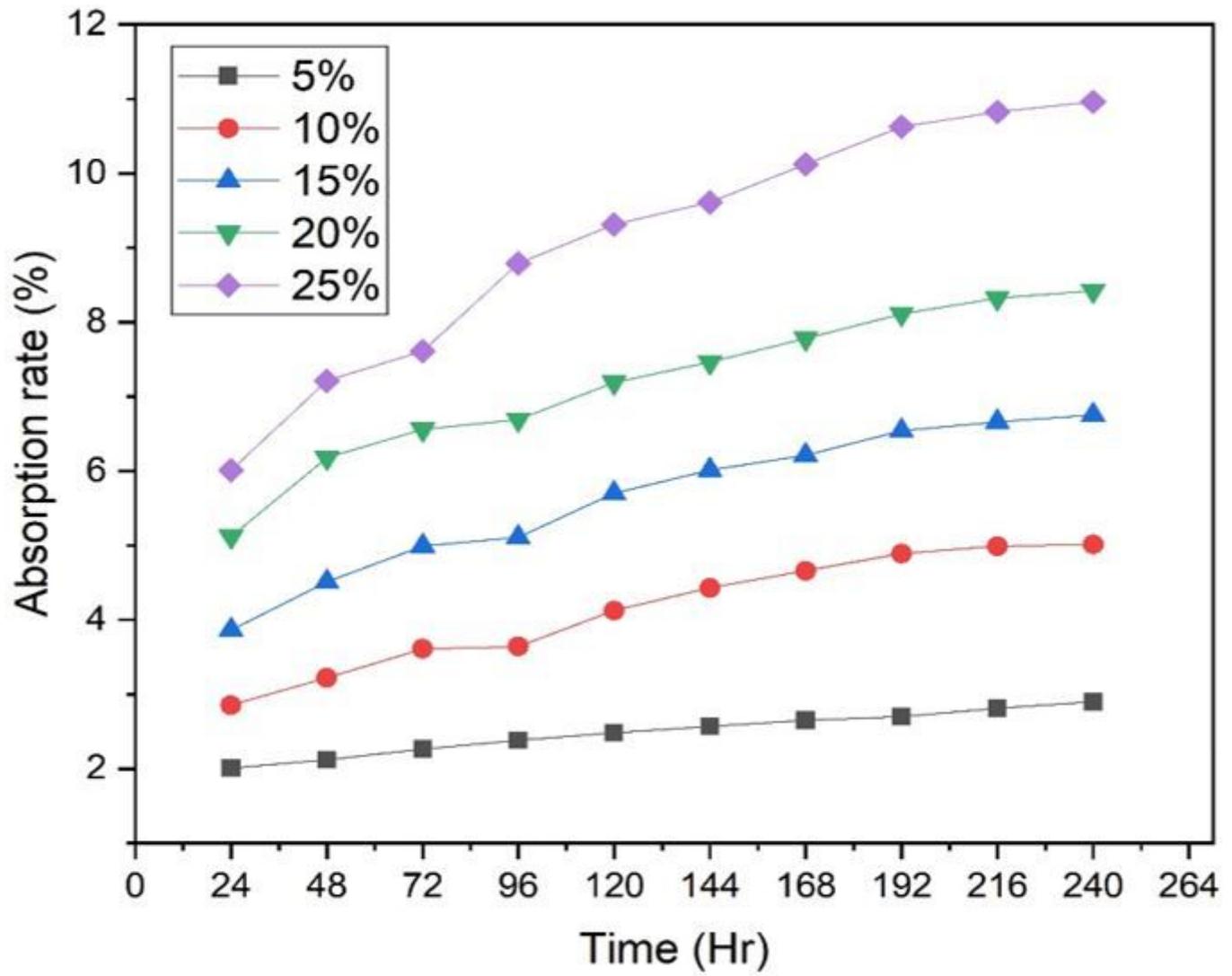


Figure 5

Water absorption rates in nettle and epoxy based composites

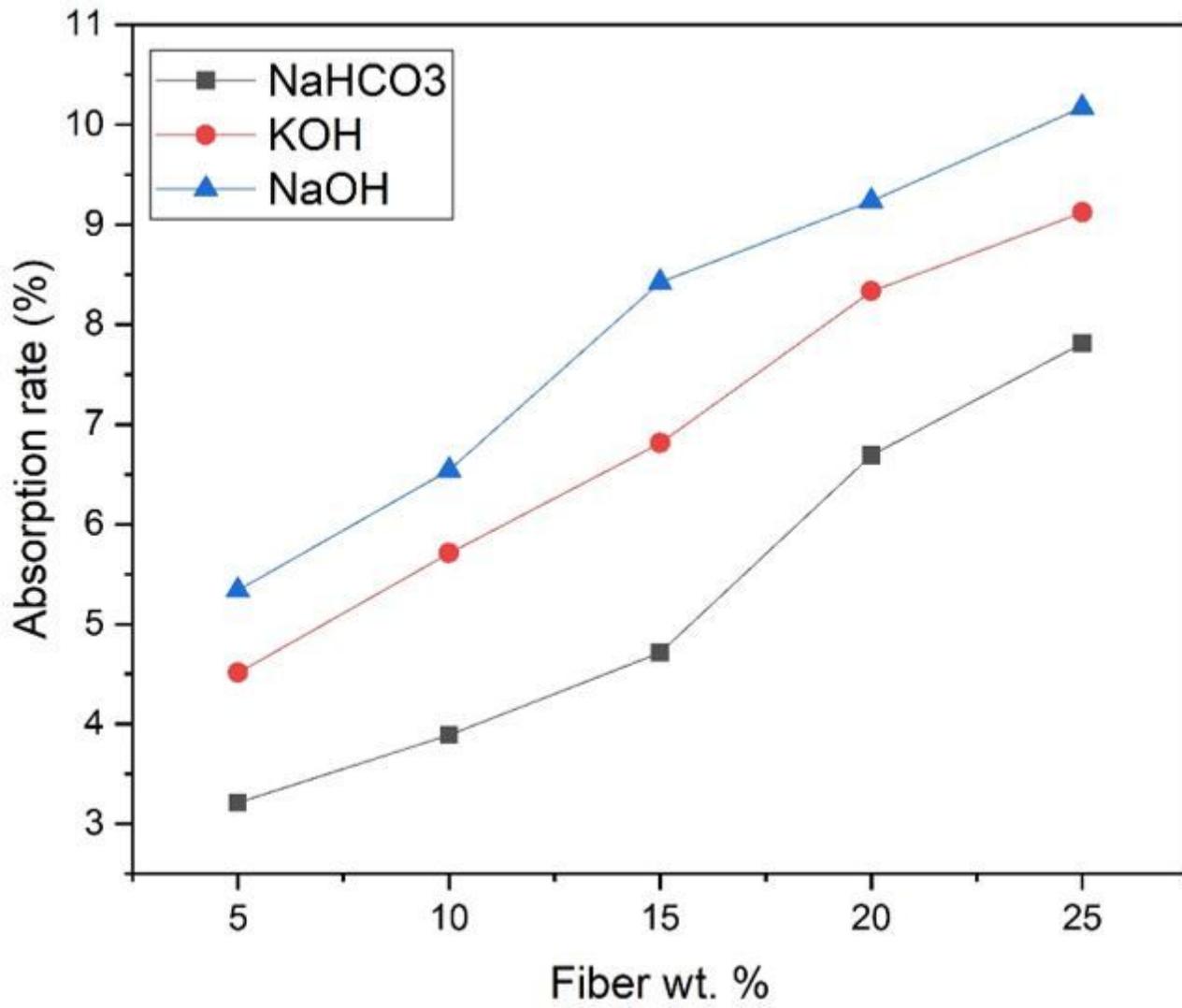


Figure 6

Chemical absorption rate of nettle and epoxy based composites

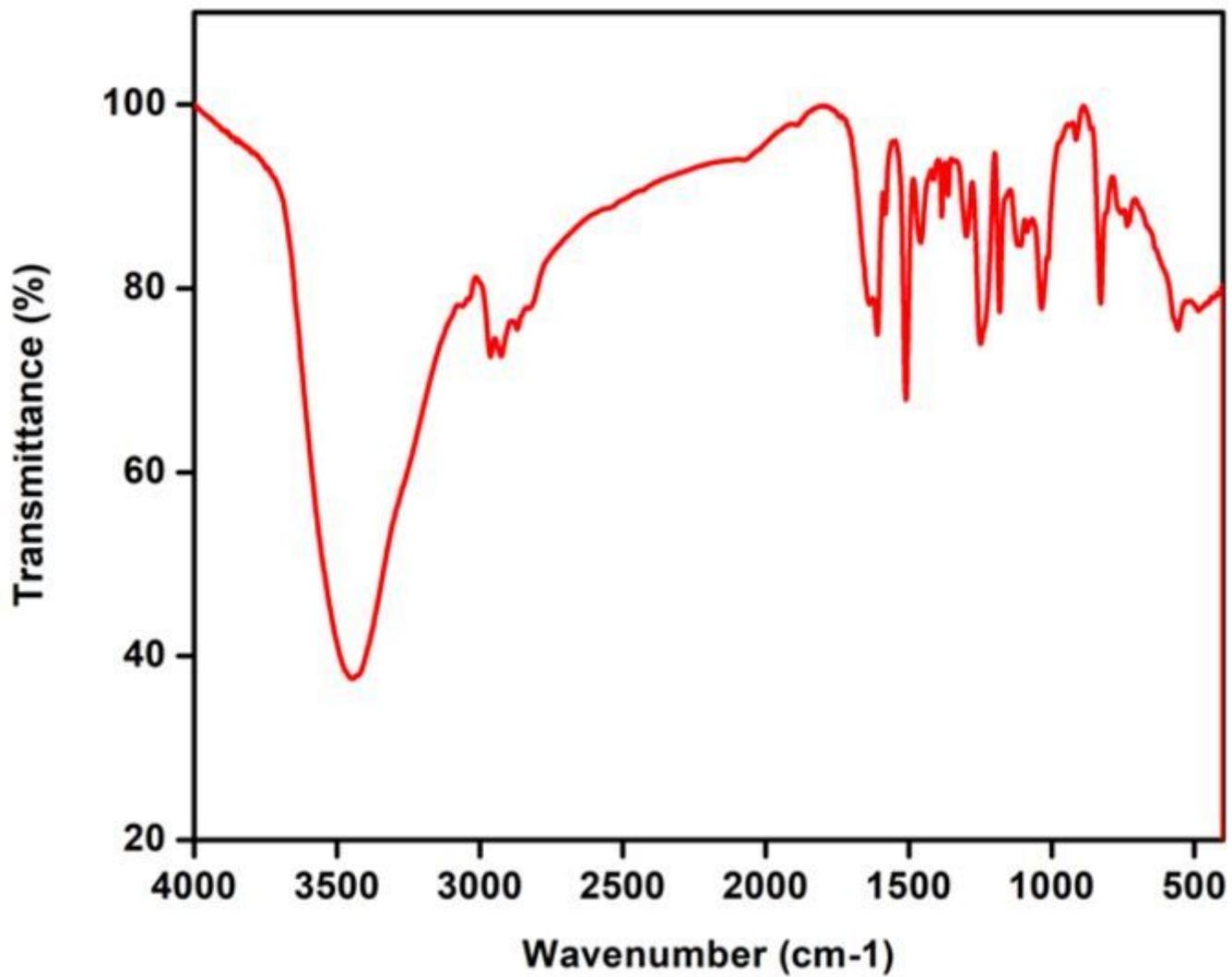
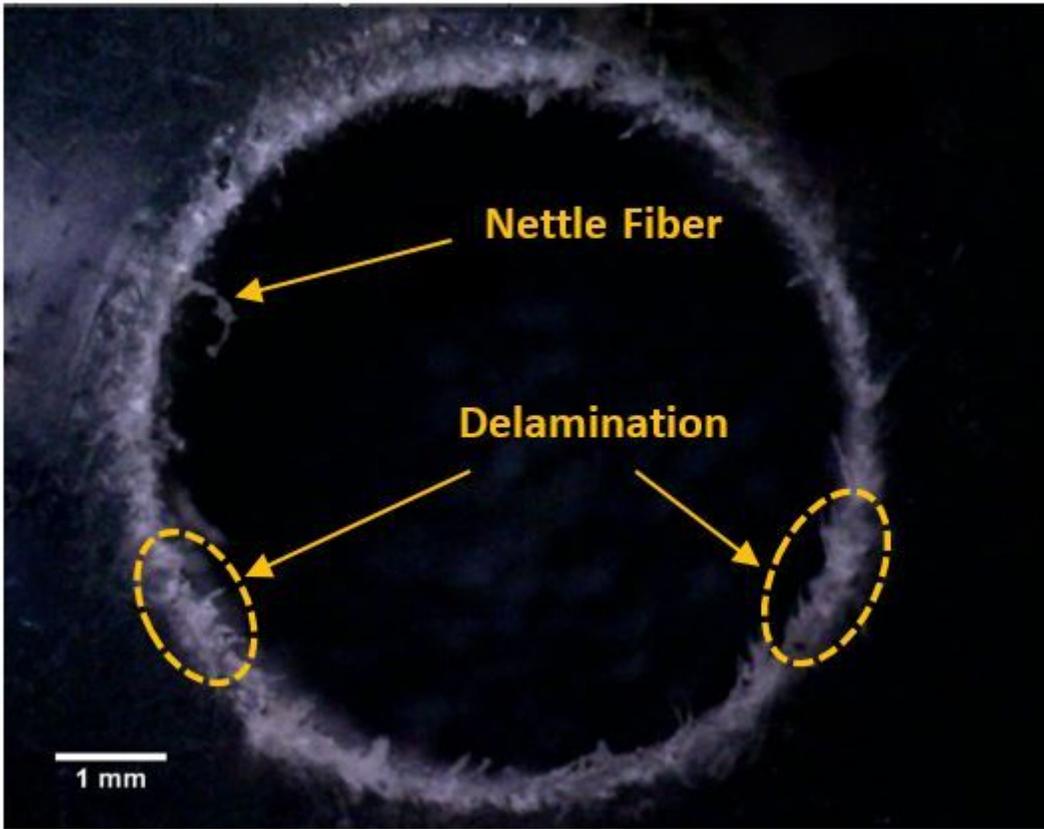


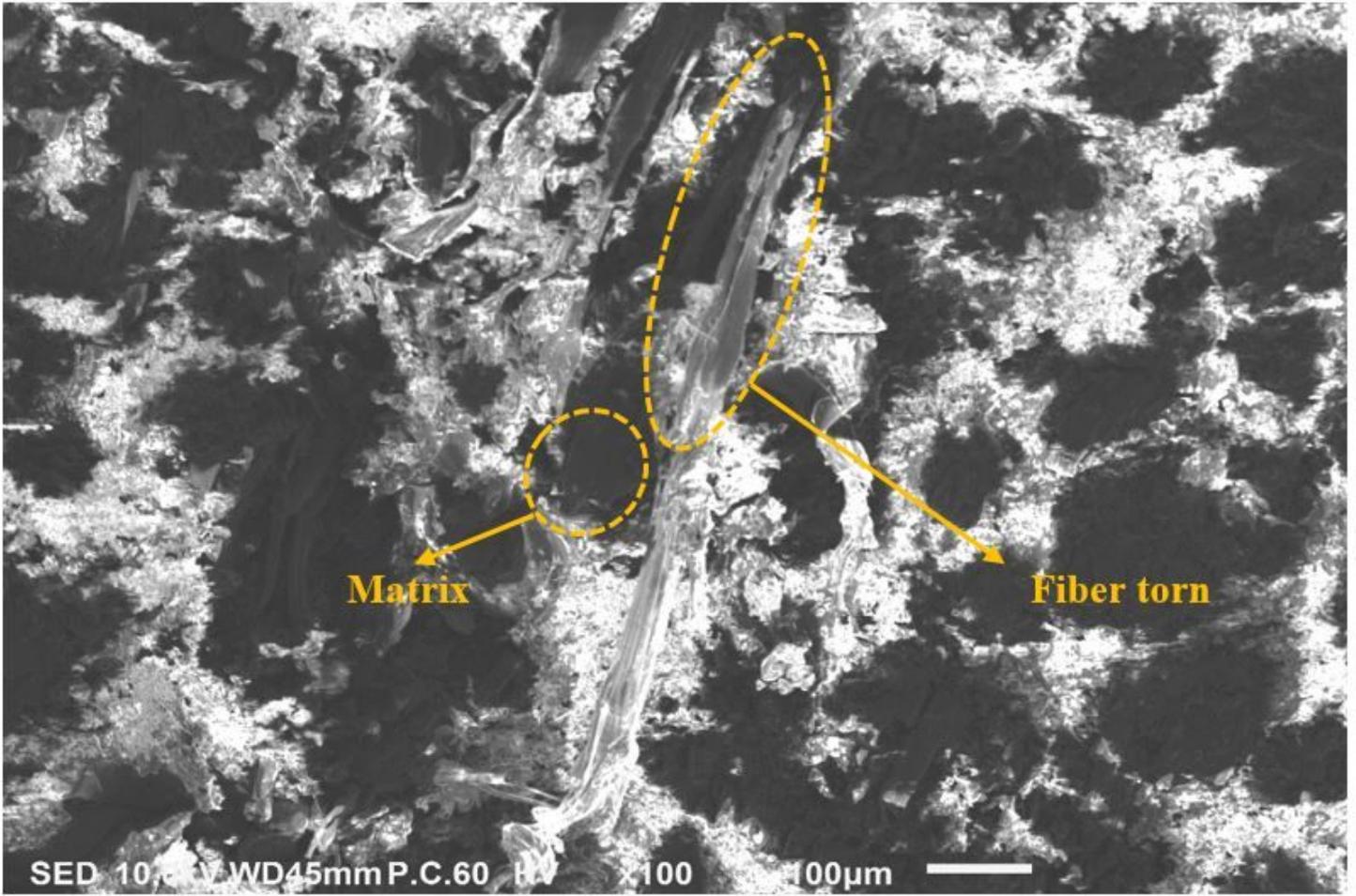
Figure 7

FTIR spectra of nettle and epoxy based composite



**Figure 8**

Typical damage in drilling of nettle/SiC/epoxy composites at exit side



**Figure 9**

SEM images of cut section of drilled samples of nettle/SiC composites.

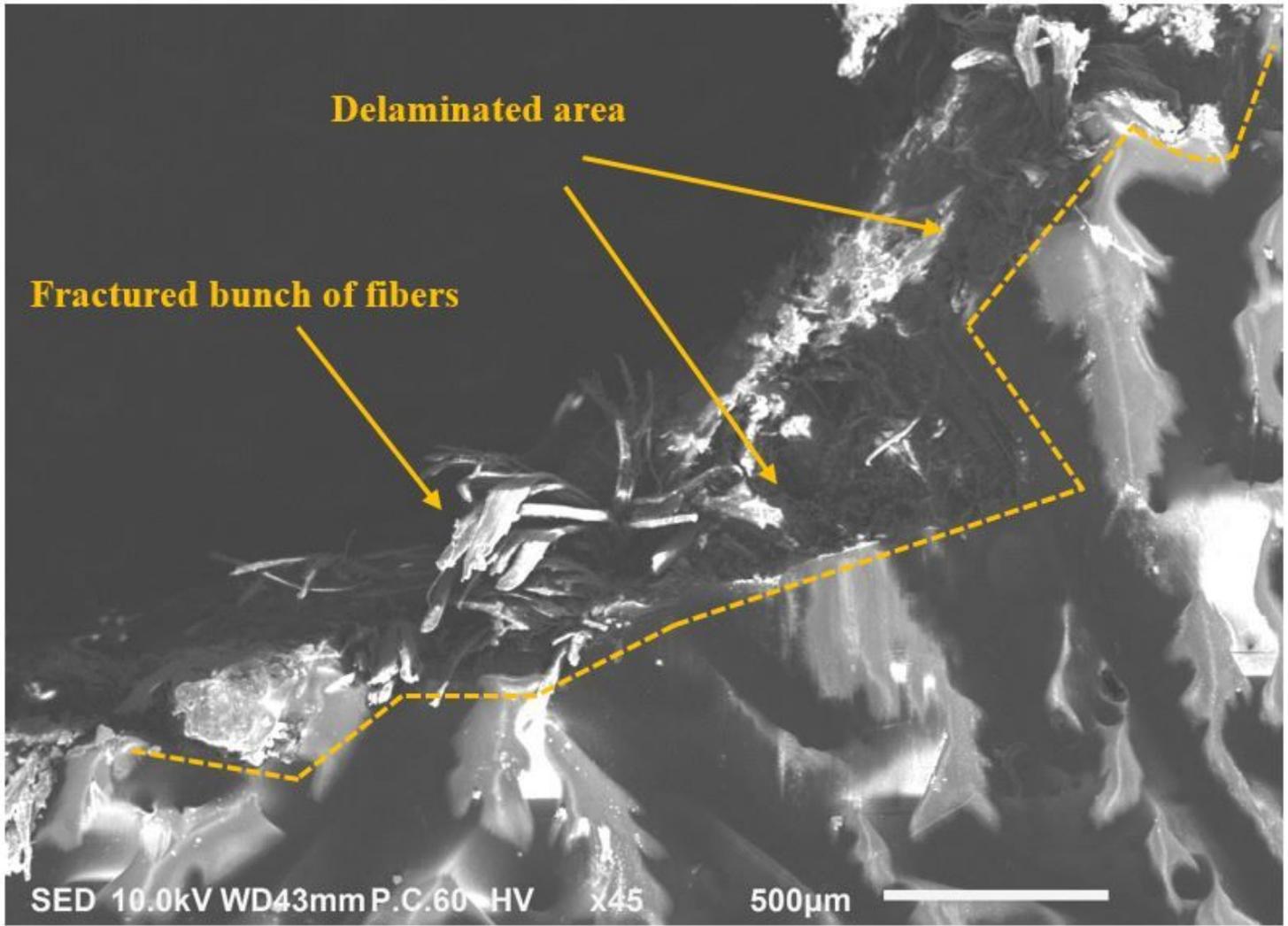
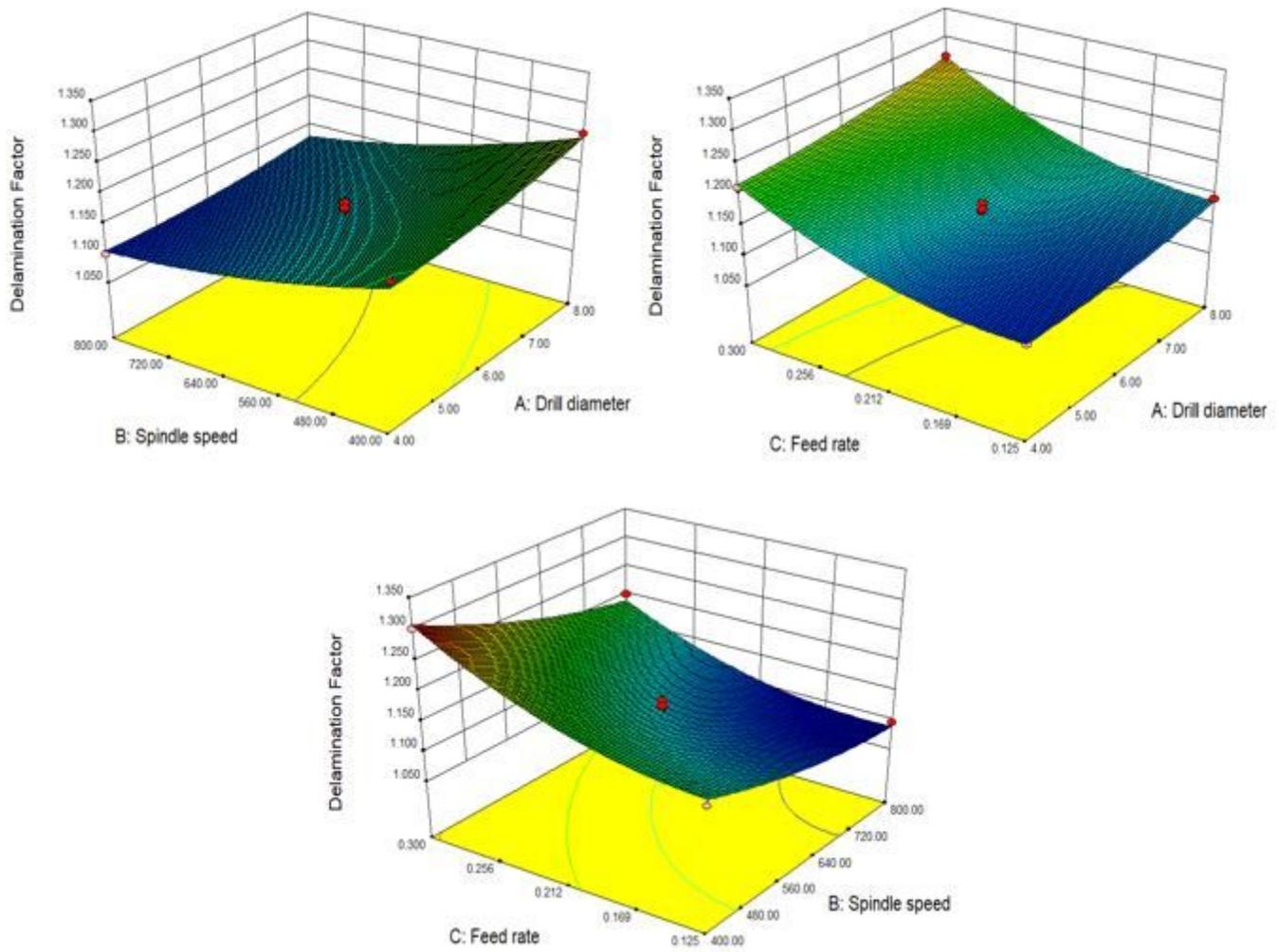


Figure 10

SEM images of top view of drilled samples of nettle/SiC composites



**Figure 11**

3D surface plot of delamination factor for SiC/nettle hybrid composites