Graphene Reinforced Natural Fiber Nanocomposites for Structural Applications

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Abstract: An attempt has been made to develop novel polymer based nano composite material reinforced with natural fibre, synthetic fillers at both micro and nano-levels with increased performance in terms of mechanical properties. This work involves development of a new nano-composite material consisting of L-12 epoxy resin, graphene at nano, hemp fibres at micro levels. The percentage composition of hemp fibres is varied from 0.3%, 0.5% and 0.7% by weight of the holding matrix, A fixed dosage of graphene nano particle of 0.3wt% of holding matrix is taken. A suitable surface modification method is employed for natural fibre treatment in order to adhere the nano-particles, this paper shall be dealing with the surface adhesion by corona method. Surfactants like NaOH and KMnO₄ were applied on the fibres. Fibers used as a reinforcement with epoxy to form composite material to enhance the adhesion between the fillers and holding matrix alkalinisation. To determine interferential adhesion and homogeneous distribution of fibres in the holding matrix Spectroscopic analysis such as Fourier transform infrared (FTIR) and scanning electron microscope (SEM) were conducted. Mechanical testing like tensile and three-point bending tests were carried out. Among the varied 0.5% of NaOH and KMnO₄ treated hemp containing graphene has given optimistic results as compared to other dosage levels.

Keywords: Nano-composites, natural fiber, Epoxy, Spectroscopic analysis.

1. Introduction

Polymer reinforced composites are gaining high interests and popularity among the material science world due to their superiority in mechanical, environmental and dielectric properties and also the combination of these properties. Along with these superiorities some downgrades are pulling the composites back such as high progressing technologies, increasing costs of limited resources and being less eco-friendly. This is paving a way for natural fibers reinforcements in the polymer matrix. These fibers are well suited for structural applications like a wood substitutes in the furniture or construction sector. Environmental issues can easily be solved by the use of natural fibers. [1] – [6]. The use of natural resources for the development of better materials is showing a rapid growth worldwide. These composites showed better strength and stiffness than glass fibers. This has been reported by several researchers [7–10]. The use of natural reinforcements like hemp, henequen, sisal,
jute, banana, coir, kenaf, kapok, and many others are getting interests as expanded reinforcement in various composite based industries, aerospace, housing etc. Cannabis Sativa (Hemp) shows outstanding mechanical properties, but the poor interfacial adhesion amongst the hydrophilic natural fibers and non-polar hydrophilic polymers is becoming the biggest limitations of natural fibers reinforced polymers. [11]. To increase the compatibility between polymer matrices and natural fibers a technique of different coupling agents has to be used. Basically, when two or more than two chemically distinct materials are combined, produces synergistic effect, with a distinct separating interface of component. Due to this the component gets aggregate properties different from the component by which it is formed. The main components cannot compete with the composites in terms of properties. The component materials can be metal, ceramic or polymer etc. The use of surface treatment for the hemp fibers to is one of the way to address the moisture absorption properties of hemp[13]. Use of alkaline treatment of the fiber also reduces the risk of moisture [12]. The properties of the composites reinforced with natural fibers can also vary with the amount of fiber content or the percentage of fiber reinforcement. However, it is generally seen that the increase in the fiber content increases the tensile strength of the composite hence we can say that effect of fiber amount in the composites is significant. To improve the properties and remove disadvantages a suitable technique of processing the fibers has to be applied. [14] - [15]. This work focuses on the use of surfactants to cure the natural fibers (hemp) to increase the mechanical properties.

2. Material Selected for the study

This section focuses on the materials used in the sample preparation and development of the composites and their characterization under study. It gives the specifications of the tests in terms of mechanical, physical, micro-structural of the modified specimens.

2.1 Matrix Material

Base Matrix for holding the reinforcement materials are diverse in nature like metals, ceramics and polymers. Epoxy resin was specifically selected because of its unique nature of excellent bonding to a vivid fiber variety, besides providing the modified samples superior mechanical and electrical properties at high temperatures. They are dimensionally stable for chemical reactions. All these advantages make L-12 as a natural candidate for the base matrix in the present research study. Figure 1 shows the L-12 epoxy resin and the corresponding hardener K-6 were obtained from Atul India Ltd. Table 1 shows the significant properties of epoxy.

![Epoxy resin and hardener](image)

Fig. 1 Epoxy resin and hardener

Table 1. Epoxy properties

| Characteristic Property | Inferences                  |
|-------------------------|-----------------------------|
| SNS Part Number         | 31200000LPX0002             |
| Type of Product         | Epoxy Resin with Hardener   |
| Brand                   | Lapox                      |
| Characteristic Property                  | Inferences                                      |
|----------------------------------------|------------------------------------------------|
| Formula Weight: 12.01 g/mol            | Appearance (Form) Powder                       |
| Appearance (Color) Black              | Carbon nanofiber- graphitized (iron free)      |
| Iron (Fe) < 100 ppm                   | Composed of conical platelets                  |
| Iron(Fe) ≤ 100ppm                     | D X L 100nm X 20-200μm                        |
|                                        | Diameter                                        |

### 2.3 Filler Material -1: Hemp

For the present study untreated hemp was used. The pictorial view of the same is show in Fig 3. Hemp fibres have a high tensile strength and they elongate easily during breaking. The moisture content is around 12%. The characteristics of the fibres is heavily influenced by the chemical composition and likewise affect the properties of the composites thus developed. Fibres were chopped to the length of 5mm using a fibre chopping machine.
2.4 Chemical Treatment

2.4.1 Alkaline Treatment
Treatment using alkaline sodium hydroxide is one of the most sought out treatment for natural fibers for reinforcing in composites. Alkaline treatment hampers in disruption of hydrogen bonding on the surface network structure which leads to higher surface roughness. The amount of lignin, wax that covers most of the external surface of hemp is removed to a certain extent. It also helps in depolymerizing cellulose and exposing of short length crystallites. The ionization of the hydroxyl group in aqueous sodium hydroxide to alkoxide is achieved \[\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber} - \text{O}^{-} \text{Na} + \text{H}_{2}\text{O}\].

2.4.2 Permanganate Treatment
Potassium permanganate is an inorganic chemical that has permanganate group $\text{MnO}_4^-$ ions. Treatment with permanganate leads to the formation of cellulose radical through negative ions formations. The highly reactive $\text{Mn}^{3+}$ ions then work for copolymerization \([11]-[12]\). But permanganate treatment has be done after the alkaline treatment. Kuruvilla et al \([16]\) treated fibers with alkali and then soaked the alkali treated fibers using 1l of $\text{KMnO}_4$ solution in the presence of acetone having different concentrations for a minute. Fibers were then dried in air. Figure 4 shows the potassium permanganate used for the study.
3. Methodology

The Graphene employed in this work were of industrial grade with a purity of 95 wt.%. To produce modified composites varying concentration of 0.1-0.5% in the incremental steps of 0.1% by the weight % of the epoxy matrix were used. Resin and Hardener were selected in the stochiometric ratio of 90:10 to produce specimen under study. Dispersion of graphene and hemp in epoxy was done using sonication method. For further dispersion of Graphene and Hemp 3-roll shear mixing process was used and the mixture was mixed mechanically. This mechanical mixing is done for certain amount of time till the mixture becomes homogeneous. During mechanical mixing gas bubbles gets trapped, and are removed using degasification process. The mixture is cured at 60°C for 2 hours followed by 100°C for 4 hours. The blend of resin and hardener was then poured into an aluminum mould of size 260 mm x 225 mm x 5 mm to produce laminates. For tensile testing alone six replicas were casted as per ASTM norms.

For alkaline treatment raw hemp was taken and thoroughly cleaned. The cleaned hemp was then cut in to 5 to 8 mm length manually. As per the literature the Concentration of 5% of NaOH which gives maximum strength was prepared and the hemp fibers were made to soak in it for 2 hours. The then treated hemp was neutralized with acetic acid until pH was 7 which was tested with the help of a universal indicator. The neutral solution was washed 3 times with distilled water so as to remove any excess chemical. This would conclude the alkaline treatment.

For permanganate treatment hemp was treated with KMnO4 for half an hour and washed again. The fibers were wrung and dried in the hot air oven at 333K for 24 hours. The moulds were prepared and the samples of 3%, 5%, and 7% by weight was prepared of both KMnO4 and NaOH treated Hemp by hand lay-up method. The hemp was taken in a beaker with graphene and epoxy and sonicated for 50 minutes. It was then poured into the mould and remained to settle for 24 hours, this would end the permanganate treatment of the hemp.

Fig 4 Potassium Permanganate
3.1 Tensile test specimen

Tensile test is one of the fundamental material science test, some important properties measured via tensile test are ultimate tensile strength, modulus of elasticity, maximum elongation. Fig 6 shows the dimensional view of the specimen prepared according to ASTM D2344 standards [17].

![Tensile Test Specimen Diagram](image)

L=250mm, B=25mm, T=2.5mm

*Fig 6* dimensions of tensile test specimen

3.2 Three-point test specimen

Three-point test also known as flexural test helps in understanding the maximum load carrying capacity and the maximum deformation that a composite specimen can undergo. Fig 7 shows the dimensional view of the specimen for the flexural test according to ASTM D3039 standards [18].
4. Experimental work

The properties of the graphene used in the study are presented in Table 3. Specimen characteristics used for experimental work on tensile test is presented in Table 4. Specimen characteristics utilized for experimental work of three-point bending test is presented in Table 5. The details of the test specimens and the experimental program is shown in Table 6.

Table 3 Properties of Graphene used for study

| Specifications       | Dimensions               |
|----------------------|--------------------------|
| Diameter             | 10–20 (micron)           |
| Purity               | 96-99%                   |
| Surface area         | 323-600m²/g              |
| Bulk density         | 0.231g/cc                |
| Fibre thickness      | 3-6nm                    |
| Oxygen content       | <4%                      |

Table 4 Specimen characteristics of tensile test

| Characteristics of specimen | Particulars                                   |
|-----------------------------|-----------------------------------------------|
| Size                        | 250(mm) x 25(mm) x 2.5(mm)                   |
| Epoxy resin                 | L-12                                          |
| Hardener                    | K-6                                           |
| Hemp                        | Treated hemp by NaOH and KMnO₄                |
| Amount of graphene          | 0.3 by Weight of epoxy                        |

Table 5. Specimen characteristics of three-point bending test

| Characteristics of specimen | Particulars                                   |
|-----------------------------|-----------------------------------------------|
| Size                        | 40(mm) x 12(mm) x 6(mm)                      |
| Epoxy resin                 | L-12                                          |
| Hardener                    | K-6                                           |
| Hemp                        | Treated hemp by NaOH and KMnO₄                |
| Amount of graphene          | 0.3 by Weight of epoxy                        |
Table 6. Details of the test specimen.

| Sample | Specimen Constitution | Treatment                        |
|--------|------------------------|----------------------------------|
| S1     | Pure Epoxy             | NA                               |
| S2     | Epoxy + Graphene       | NA                               |
| S3     | Epoxy + Hemp           | Untreated                        |
| S4     | Epoxy + 0.3% graphene+ HEMP | 3 % of NaOH treated hemp         |
| S5     | Epoxy + 0.3% graphene + HEMP | 5 % of NaOH treated hemp         |
| S6     | Epoxy + 0.3% graphene+ HEMP | Pre-alkaline treated + 0.3 % of KMnO4 treated hemp |
| S7     | Epoxy + 0.3% graphene+ HEMP | Pre-alkaline treated + 0.5 % KMnO4 treated hemp |

4.1 Tensile Test
Tensile test was performed on the composites developed using UTM as shown in Fig.8 with specimen of the size 250 mm x 25 mm x 2.5 mm used. To achieve experimental accuracy, ASTM D2344M was followed to obtain the statistical accuracy of the tensile test of the polymer beams tested. The specimen size and type of the test conducted is shown in Table 7. Specimens were almost rectangular shaped based on the ASTM norms for random dispersed and disoriented fillers in the holding matrix condition as shown in Figure 9.
4.2 Three Point Load Test on Beams
The mechanical performance of the hybrid nanocomposite material reinforced with individual fillers and graphene and their combination in polymer based matrix were evaluated by fracture mechanic test. Specimen beam of size 40mm x12mmx6 mm were tested by three-point loading test. A hydraulic closed loop testing machine was used. To achieve experimental accuracy, ASTM D2344M was followed to obtain the statistical accuracy of the flexural strength of polymer beams. The equipment used for the three-point load test is shown in Fig.10. The specimen size and type of test conducted is shown in Table 8. Specimens were rectangular based on the ASTM norms for random dispersed and disoriented fillers in the holding matrix condition as shown in Figure 9.
5. Results and Discussion

This section focuses on the mechanical and spectroscopic results of the specimen.

5.1 Mechanical Characterization

The results of the tensile test are shown in Table 7. The graphical representation of the same is shown in figure 12. The result of the three-point bending test is shown in Table 11. The bar graph representation and load vs deflection of the three-point bending test is shown in Figures 13.

| Sample | Ultimate Load(kN) | Ultimate Strength (N/mm²) | Deflection(mm) |
|--------|-------------------|---------------------------|----------------|
| S1     | 1.6               | 1.5                       | 1.6            |
| S2     | 3.65              | 0.66                      | 3.65           |
| S3     | 2                 | 0.1048                    | 0.45           |
| S4     | 10.88             | 0.68                      | 0.5            |
| S5     | 6.4               | 0.4                       | 0.8            |
| S6     | 5.81              | 0.36                      | 0.4            |
| S7     | 5.17              | 0.32                      | 0.78           |

Table 7. Tensile Test Results.
Based on the chart presented in Figure 11, it can be seen that treatment using NaOH showed a higher strength than the epoxy beams containing untreated hemp. Beams reinforced with untreated hemp fibers tend to show a lower load carrying capacity because of the polymer chain linkage which leads to voids, pores in the material making it more brittle. In case of alkaline treatment, moisture is removed by disrupting hydrogen molecules leading to higher surface roughness. Higher roughness leads to better bonding of molecules making it more capable of carrying load. We see a decline in strength in 5% alkaline treated hemp due to increase of viscosity compared to 3% alkaline treated hemp. In permanganate treatment, the tensile strength increases at 0.3% and then reduces at higher concentrations. The reason may be the degradation of fibers at elevated permanganate percentages. The concentration being an important aspect in realizing the tensile characteristics in permanganate treatment.

| Sample | Bending Deflection (mm) | Bending Load (N) | Flexural Strength (MPa) |
|--------|-------------------------|------------------|------------------------|
| S1     | 3.9                     | 0.5              | 69.4                   |
| S2     | 5.69                    | 0.77             | 106.94                 |
| S3     | 0.57                    | 0.25             | 34.7                   |
| S4     | 1.01                    | 0.34             | 47.22                  |
| S5     | 1.05                    | 0.32             | 44.44                  |
Fig. 12 Three-point bending test for Various Specimen

Fig 12 represents the Load versus deflection for various composites. It can be seen that permanganate treated hemp shows the highest flexural strength, after pure epoxy. With the increase in concentrations decreases the strength. In alkaline treated the strength has surely increased, but with increased concentrations the strength has slightly reduced with slightly increased deflection showing the viscosity being increased in the matrix.

5.2 Physical Characterization
The TGA abbreviation for Thermo-Gravimetric Analysis is a process of measuring the mass of each sample with the increasing temperature, by doing so we get the thermal stability, composition and purity of the material under the test. This test can also be used to know about the reaction of the material with the certain atmosphere. The test is normally conducted in air or nitrogen atmosphere. The material, which is kept in a crucible and covered with small holes, is slowly heated from the set temperature with the predefined heating rate that constantly increases the temperature of the cabin. This method essentially can be used for the solid and liquid materials since weight of gasses cannot be measured and the reaction cannot be known. A configured thermocouple one part of which is in the standard environment and the other part is in the chamber, measures the temperature of the chamber. At the start of the test which is at comparatively lower temperature the volatile gases escape first and then after all the volatile gases have escaped the material using the excess energy start reacting to the atmosphere which in most cases happens to be air, the triggered reaction can be easily known to be endothermic or exothermic reaction with the help of DSC curve. The DSC curve which is a plot between the temperature and the temperature difference can be utilized to recognize the nature of the
reaction, if the temperature difference is positive this means that the material is undergoing an exothermic reaction thus fueling its own reaction where as if the temperature difference is negative that means that the material that is reacting cannot be used for heat generation under that circumstance. This along with the weight difference curve given by the TGA is used for the thermal characterization of material. In the study a constant heating rate of 10 degree per minute was used so that ample of time was given for the material to conduct the heat uniformly in the whole volume. FTIR an abbreviation of Fourier Transform Infrared spectrum is the test made for the qualitative analysis of the material .This can be used for the recognition of certain organic bonds that have a signature energy associated with them, when light is passed over this bond is there a match in the energy carried by the photons the bond absorbs and emits the energy which essentially makes the molecule to move away from the other bonded atom. This can be seen as stretching or bending of the bond, depending upon the situation. A broad spectrum is made to shine over the material and its transmittance is measured, and the spectrum is then reduced to a single band which increases its accuracy. This essentially is the way to get the finger print of each material since the chance in any composition will give a different curve. Since every bond has its own enthalpy of formation the signature of its presence can be confirmed by recording the light that is transmitted through the substance , if the comparative energy bond is present it does not allow the transmittance of that particular band, this causes a drop in the transmittance of the band and hence the graph drops.

![DSC-TGA](image)

Fig 13 DSC TGA of 3% NaOH treated Hemp
Fig 14 FTIR of 3% NaOH treated Hemp

Fig 15 DSC TGA of 5% pre-alkaline with post potassium permanganate treated hemp
TGA-DSC was conducted with the maximum temperature of 1000 degree Celsius with the heat increase rate of 10 degree centigrade per minute. Graph of sample which is shown in figure 14 with 3% hemp that is NaOH treated, which shows us that the material has around 1.804% of volatile material and decomposes at 380 degree centigrade the exothermic reaction also reaches the peak at the similar temperature showing that the mass loss is mainly due to combustion. The Carbon black which is the residue is about 1.864 %, with the plastic substance being 88.16 %. This shows us that the material is stable up to the temperature of 344 degree. The TGA – DSC of sample with 3% hemp which is potassium permanganate treated is shown in figure 15 shows a similar trend with 88.16 %
as polymer component. And combusts at the temperature of 344 degree Celsius which the reaction that is exothermic in nature. FTIR spectrum shown in figure 14 shows us the fingerprint of the material with 3% hemp that is NaOH treated and the most distinguished peak is the 3431.73 wave number which shows the presence of an OH bond as per the spectroscopy which is present in both epoxy and the natural fiber which has cellulose. 1608.24 wave number shows the stretching of conjugate alkene bond which is also present in the epoxy and graphene. The next significant peak is observed in 1583.32 which is correspondent to cyclic alkene bond which is present in graphene FTIR spectrum shown in figure 16 shows the same data as in the figure 11 that explains the 3% concentration this occurs due to the fact that the coordinate bonds are not effected in the FTIR spectroscopy which renders the potassium permanganate invisible.

5.3 Microscopic images of the specimen

3% Hemp(NaOH Treated) 5% Hemp (NaOH treated)

3%( NaOH + KMnO4 treated) 5%( NaOH + KMnO4 treated)

Fig 18 Microscopic view of various treated hemp specimens

Figure 18 shows the uniform distribution of the reinforcement in the matrix and the dispersion and effects of treatment on the property of the matrix.
6. Conclusion
From the above study, we can see the more effective utilization of natural fibers in polymer composites. NaOH treatment: the 5% NaOH treatment gave the increment of strength by 34 times and load carrying capacity by 9 times in tensile test with respect to pure epoxy sample. 3% Permanganate on further treatment has increased the load carrying capacity by 1.3 times and the deflection has decreased by 2.9 times with respect to pure epoxy sample. The composite is stable until the temperature of 344 degree and then burns with exothermic reaction. Treatment of natural fibers help increase the strengths of the matrix. The results from tensile test of the material we found that 3% alkaline treated hemp yielded more than five times that of untreated hemp. Whereas with the increase in reinforcement the strength is reduced. In case of permanganate treatment the higher strength was found at 5% potassium permanganate treatment. The reason for decreased strength of the untreated hemp may be improper dispersion of hemp and the bonding capability of hemp with the epoxy. With the specialized chemical treatment fibers become rougher and bonding becomes easier. Cellulose depolymerize exposing short length crystallites. Thus we can conclude that treatment of hemp fibers help achieving higher strengths.

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