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Studies Onmechanical Properties of Nano –Hemp Fibre Composite Material

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ABSTRACT
The use of natural fibers instead of traditional reinforcement materials such as glass fibers, carbon provides several advantages including low density, low cost, good specific mechanical properties, and biodegradability. Due to low prices, eco-friendly and high tensile behaviour leads to rising commercial performance of natural fibre reinforced plastics in all around the world. The primary aim of this research work was to investigate the mechanical properties of green composites reinforced with raw hemp and nano-hemp fibre. Many research report shows that the nano fibre reinforced structures exhibit higher mechanical properties. Initially the raw hemp fibre was broken down in to sub-micron fibrillar structure and subsequently to the level of nanofibril through physico-chemical methods where in a mild chemical treatment was given to separate the foreign particles such as pectin, wax and lignin. Further to this stage mechanical defibrillation has done through commercial grinder to isolate nano fibres from raw hemp. Theaverage diameters of the generated nanofibres in the range of 20-100nm and the generated nano fibres have higher crystallinityas compare with the raw fibre bundles due to the removal of hemicellulose. Nano fibres of hemp were characterized by FE-SEM and crystallinity of raw hemp structure andnano fibril based hemp structures were studied through X Ray Diffraction. The nano fibre was reinforced with Epoxy matrix to produce the composite structure with various fibre volume fraction such as 2%,4%,6%,8% and 10% ,similarly the raw hemp fibre was used to produce the same structures through hand layup technique. The mechanical properties of composites made from raw hemp fibre as well as nano hemp fibre were studied. The nano hemp fibre reinforced composites shows higher performance properties as compare with raw fibre based composites.
Key words: Nano hempfibre,resin matrix,hand layup method.

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I. INTRODUCTION

Nano fibres possess high mechanical properties such as high young's modulus ,high tensile strength and higher thermal stability and many more properties . This was the key intension to fabricate the nanofibres for industrial application.The use of natural fibers to make low cost and eco-friendly composite structures is a subject of great importance (Bismarck et al., 2001). When Compared with synthetics fiber-based composites, natural fiber based bio-composites display several excellent advantages, e.g. low density,eco-friendly and low cost. However, these natural fibers display their own drawbacks such as higher polar groups and hydrophilic. Due to this reason natural fibers are poor compatible with polymer and result in the loss of mechanical properties dueto high moisture absorption (Belgacem and Gandini, 2005).Jute stem contains crystalline cellulose in a structured manner, which forms the micro fibrils and are interconnected together through loosely arranged hemicellulose and lignin network.This structure has to be destructed to generate cellulose microfibrils from this network. Many techniques are available to separate the microfibrils such as chemical, mechanical, physico-chemical treatments. (Suhara,et al,2013). The most commonly used physico-chemical methods are such as acid and alkali treatment to remove pectins and hemicelluloses.As reported by Li et al.(2009)alkali treatment at 130^0C and sulphuric acid treatment extract the cellulose whiskers from mulberry branch-barks.SimilarlyKaushik et al.(2010) extracted cellulose nanofibrils from wheat straw using a combined chemical treatment and steam explosion subsequently followed by high shear mechanical treatment. Chen et al(2009)used successive chemical methods to separate the cellulose microfibrils from wood and bamboo.Leyva et al. (2011) produced cellulose nanofibers fromdurumwheat strawand characterized it to study its potential as reinforcement fibers in biocomposites. Alemdar et al. (2007) extracted cellulose nanofibers from the agricultural residues

(wheat straw and soy hulls) by a chemico-mechanical technique and the structure of nanofiberswere investigated by transmission electron microscopy. Leitner et al. (2007) isolatedcellulose from sugar beet chips, a by-product of sugar production bywet chemistry. Further processing of the cellulose with a high-pressure homogenizer led to the disruption of cell walls intonanofibrils. Das et al. (2010) extracted spherical and elliptical shaped jute nanofibers from jute fibers by acid hydrolysis and were incorporated as reinforcing filler in a biocopolymer matrix by the melt mixing method.Jute nanofibers (JNFs) were prepared by treating jute fibers with alkali and dimethyl sulfoxide (DMSO) and then applying acid hydrolysis.Luduena et al (2011) prepared nanocellulosefromrice husk .The nanocellulose samples were prepared by acid hydrolysis of sub products of rice husk obtained from several chemical treatments.

II. MATERIALS AND METHODS

Hemp fibres, sodium hydroxide, Epoxy resin with hardener were supplied by GVR enterprises Madurai, India. Nano fibre preparation pressure cooker (Model:VS-2416; Koyo Engineering Corp., Saitama, Japan) with capacity of 0.5 Mpa is used from Chemical laboratory of GandhigramUniversity,India.

2.1 Preparation of nanofibers

In this research work Combined chemical and mechanical process was followed to extract the hemp nano-fibre.The fibre separation procedure was Raw hemp fibre was mercerized with 17.5% of sodium hydroxide solution for 1hour,for that the fibres are cut in to small length of 2 to 4mm. After mercerization the fibers were washed with distilled water and then the fibres were soaked in water for 12 hours and disk refined then the fibres dried well and then treated with 1M hydrochloric acid solution at 60-80 °C for 2h. The swollen were cooled at ambient temperature, and neutralized with a solution of ammonium hydroxide to pH 9.5, and soaked overnight to separate the soluble minor and extraneous components such as pectins, starch and fat from the insoluble fibres. Then the fibers are subjected to the pressure of 0.078 MPa for 30 minutes in a pressure cooker. As reported by Teixeira et al (2010) High pressure steam defibrillation created an efficient reduction in the diameter of hemp fibres up to nano scale. The average diameter of the nano hemp fibres were less than 100 nm.

2.2 Preparations of composites samples from raw hemp and nano hemp fibres

The low temperature curing epoxy resin and the corresponding hardener (HY951) were mixed thoroughly in a ratio of 10:1 by weight. The nanofiber of various wt.% (2%, 4%, 6%, 8% and 10%) was mixed with epoxy resin then stirred well uniformly then the composite slabs were produced with hand lay- up techniquewith 210mmx210mmx10mm mould specification and then cured for 24 hours. Subsequent to this process a mild compression moulding was given to get high density composite.Similar procedure was followed for raw Hemp- epoxy based composites with the same sample specification. Specimens of suitable dimension were cut out using a diamond cutter for mechanical testing. Fabricated composite samples are shown in Figure-1

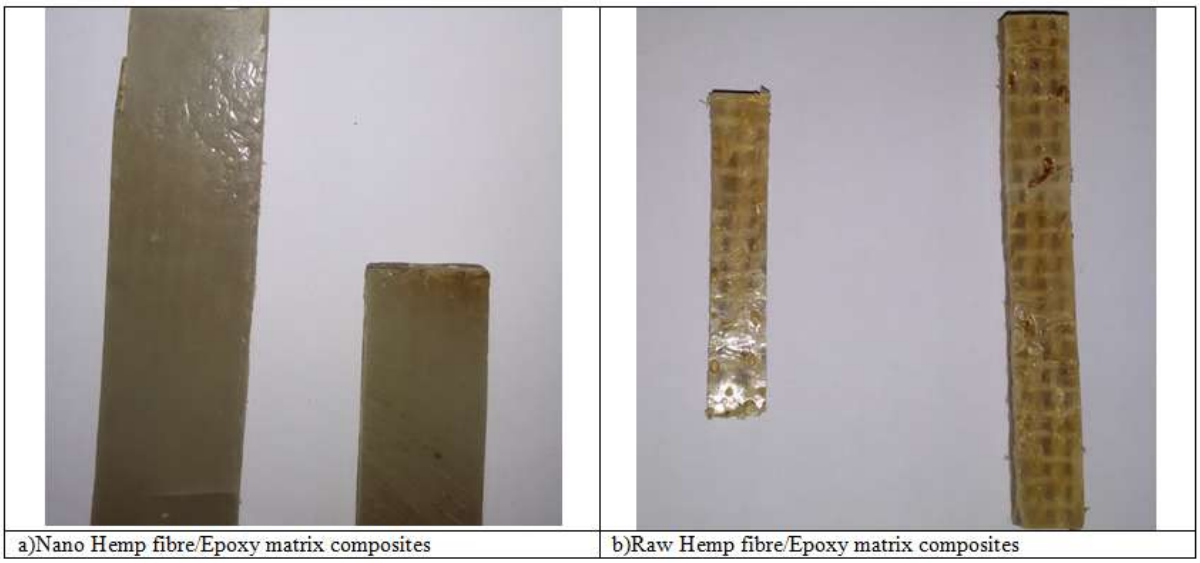

Figure-1 Samples Hemp fibre reinforced composites

2.3 Estimation of chemical composition

As reported by MariesIdicula et al(2005) the chemical composition estimation technique was followed. The major chemical component are available in the hemp fibre .All the plant cell walls consist mainly of sugar-based polymers (carbohydrates) that are combined with lignin and lesser amounts of extractives, protein, starch, and inorganics. The chemical components are distributed throughout the cell wall, which is composed of primary and secondary wall layers. Chemical composition varies from plant to plant. The chemical composition will change the physical and chemical properties of fibre . So it is essential to analysis the changes in the chemical composition of raw hemp fibre to nano hemp fibre.From the below form is used to analyse the cellulose and hemicellulose composition present in the fibre

Neutral Detergent Fiber(NDF) = Hemicellulose + Cellulose + Lignin + minerals
Acid Detergent Fiber(ADF) = Cellulose + Lignin + Minerals
Hemicellulose = NDF – ADF
Cellulose = ADF – Residue after extracted with 72% H2SO4

2.4 Microscopic Study and Diameter Distribution

After each chemical treatment, morphological studies such as SEM ,FESEM has done to verify the structure of nano hemp fibre after removal of pectin, hemicellulose, and lignin.Since the samples are bad conductors sputter coating was given to the samples to avoid electrostatic charge during examination .

2.5 X-ray Diffraction Analysis

The crystallinity of the Hemp fibre before and after defibrillation was examined using a Bruker AXS D8 Discovery Diffraction System.Raw hemp fibers and nano hemp fibers were subjected to a powder X-ray diffraction method analysis (PXRD) respectively.Thecrystallinity index (CI) was evaluated by using Segal et al. (1959) empirical method as follow:

$$CI\% = \frac{(I0\,0\,2 - Iam)}{I0\,0\,2} X100$$

where I0 0 2 is the maximum intensity of diffraction of the (0 0 2) lattice peak at a 2θ angle of between 21◦ and 23◦, which represents both crystalline and amorphous materials. Iam is the intensity of diffraction of the amorphous material, which is taken at a 2θangle between 18◦ and 20◦ where the intensity is at a minimum. 10 replicates were used.

2.6 Mechanical studies

Mechanical properties such as tensile strength, flexural strength,and impact strength were studied as per ASTM standard for both raw hemp fibre reinforced composites as well nano hemp fibre reinforced composites.

2.6.1 Tensile strength Test

As per the ASTM-D638 procedure the tensile testing was carried out withInstron Model 20. The cross head rate of 2.5 mm/min was maintained. All mechanical testing were performed at room temperature and the tests for 5 sample specimens for each set was carried out to get the average value.
Tensile strength or tenacity is the stress at the breaking point of the test specimen (Gindl et al 2004). Tensile strength was obtained from the experimental data using equation

Tensile strength = load at break/ original cross-sectional area= L / b x D
Where, L is the load applied in N, b is the width in mm and D is thickness in mm.

2.6.2 Flexural Strength test

As per ASTM D790 procedure the testing procedure was carriedout. The Dimensions of the samples are as follows 3 mm in depth, 10 mm in width and 90 mm in length. The testing was done through Instron tester at 10 mm / minute cross-head speed.

Flexural strength (S) = PL / bd2

Where, P = load at a given point on the deflection curve (N)

L = length of the support span (mm)

d = width of the bar (mm)

b = depth of the beam (mm)

2.6.3 Impact Testing

The Izod impact strength of raw and nanohempfiber reinforced epoxy polymer composite samples were tested as per ASTM D 256-88(Model 6025, UK). The dimension of the samples as follows thickness 3.2 mm with 10 mm cross-section and 64 mm long was clamped in the base of the pendulum testing machine. The pendulum was released and the force consumed in breaking the sample was calculated from the height the pendulum reached on the follow through.

2.7 Thermal properties

The thermal properties of the raw and nano hempfiber reinforcedepoxy composites were obtained by performing thermogravimetric analysis(TGA).Thermogravimetric analysis measures the amount and rate ofchange in the weight of a material as a function of temperature or time in acontrolled atmosphere. Measurements are used primarily to determine thecomposition of materials and to predict their thermal stability at temperatureup to 1000^{0}C. The technique can

characterize materials that exhibit weightloss or gain due to decomposition, oxidation or dehydration.

III. RESULTS AND DISCUSSION

Characterization of nanoHempfiber and fabrication of Hemp nanofiber reinforced epoxy polymer composites with various percentages (2%, 4%, 6%, 8% and 10%). These composite samples were characterized by mechanical (tensile,flexural, impact and hardness), thermal (TGA) and surface morphological studies (SEM and FESEM).

3.1 Estimation of Cellulose, Lignin and Hemicellulose

Cellulose and hemicellulose contents were analyzed in raw and nanohempfibers. It was observed that cellulose content in the nanohempfiber was 85% ,where as in raw hempfiberit was 73% and the hemicellulose content from raw hemp fibre and nano hemp fibres were showing 21% and 3% respectively. The following graph (Figure 2) Indicates the comparison between the both the fibres.

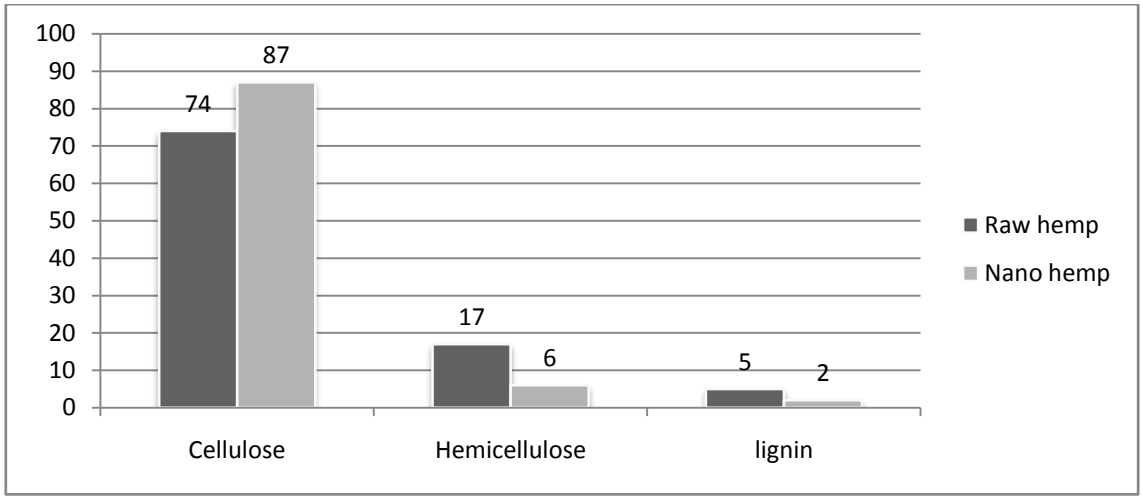

Figure-2 Comparison of chemical composition between raw hemp fibre to Nano hemp fibre

Higher the cellulose leads to a better stiffness and strength of the fibers. In the above graph,it is understood that the cellulose content is higher because of removal of hemicellulose and lignin and other compounds from the raw hemp fibre.

3.2 Morphological Studies on Nano HempFiber and composites

After each chemical treatment, scanning electron microscopy was done to verify the removal of pectin, hemicellulose, and lignin, and to visualize the separation of the fibers from the fiber bundles. The morphological development of raw Hemp fiber is shown in Figure 3. The Figure 4. shows the change in the morphology of Hemp nanofiber after physio-chemical treatment due to the removal of hemicellulose, lignin, and pectin. Hemp micro-fibrils are around 30~50 nm. High pressure steam defibrillation created an efficient reduction in the diameter of hempfibers due to hempfibers splitting along the fiber axis (wang et al 2009). The FE-SEM images of the Hemp nanofibers are (shown in Figure 5.) having less than 100 nm in diameter. The uniform distribution of pressure will influence the diameter of Hemp fibrils.

Similarly the SEM image for raw fibre and fibre after defibrillation indicated as figure 3 and 4 .This diagram shows that nano fibres produced were almost even diameter .The even diameter nano fibre distribution was possible due to sufficient pressure in the pressure cooker and efficiency of pre-treatment given to the fibre surface.

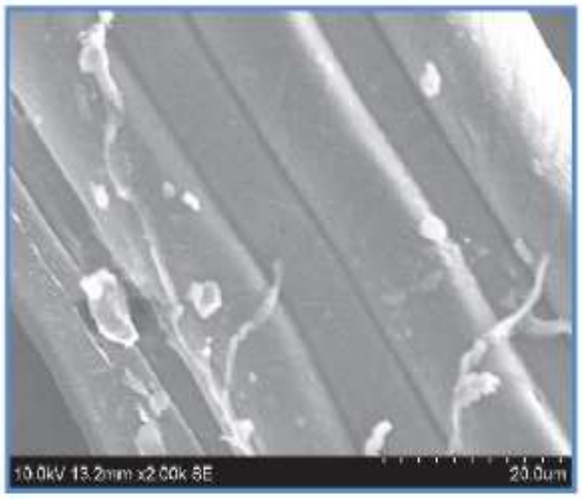

Figure-3 SEM Image of Raw Hemp Fiber

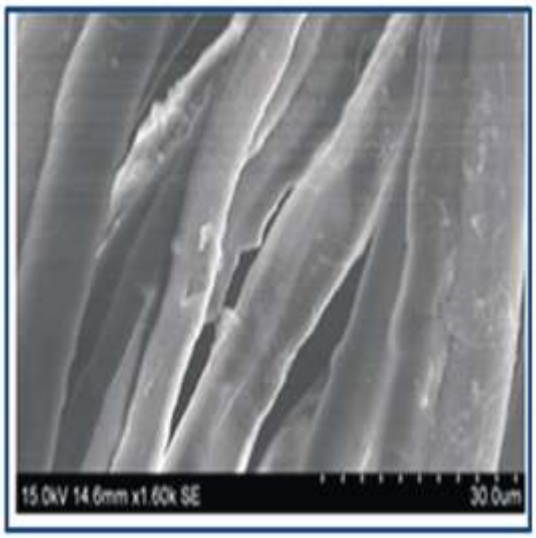

Figure -4 SEM Image of nano Hemp Fiber after defibrillation

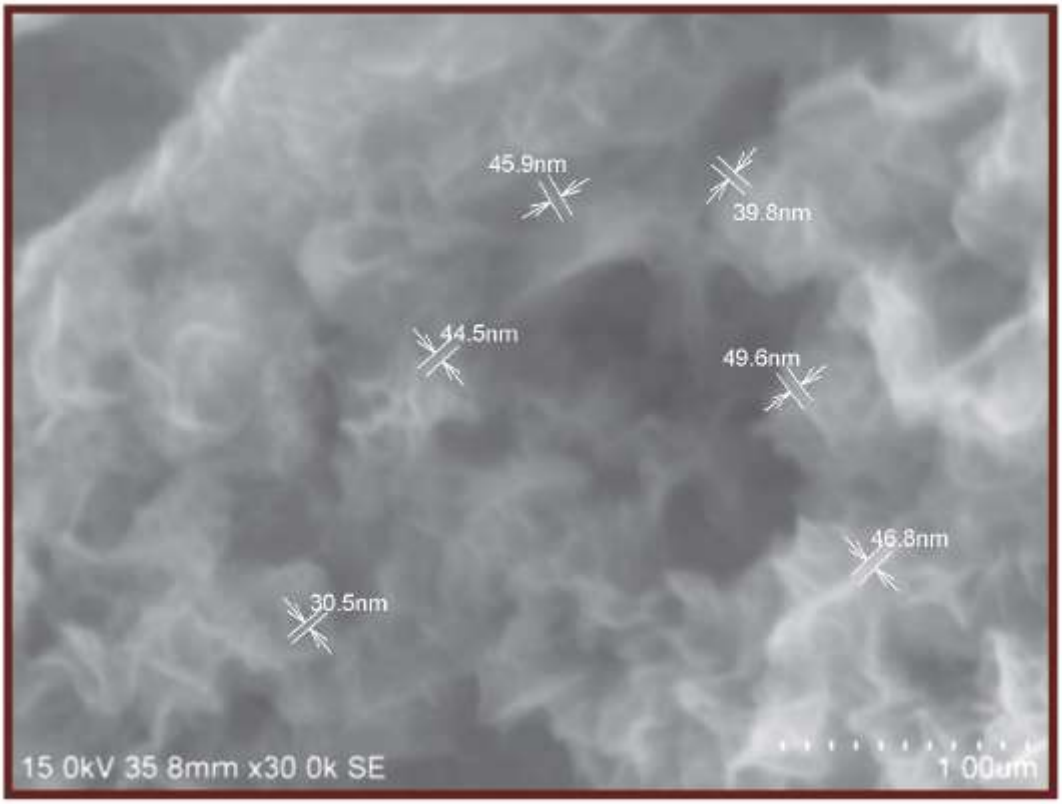

Figure -5FE-SEM Image of Nano HempFiber

3.3 X-Ray Diffraction

XRD pattern of raw hemp and nano hemp fibre after defibrillation is shown in following figure. X-ray diffractogram was used to investigate the crystallinity of raw hemp structure to nano fibril based hemp structures. An example of X-ray powder diffraction spectra from these hemp fibers is given in Fig. 67Crystallinity index analysis was made and summarized in Table no .1. It can be seen from Table 3 that the major crystalline peak of the hemp fibers with various treatments occurs from 19^0 to 22.5 ^{0} peak above the base line for both the cases.In this paper the method of minima and

maxima for the calculation of crystallinityindex, with following expression
Crystallinity index (C.I.) = Imax-Imin/ Imax
The crystallinity index for raw Hemp fiber and Nano Hemp fibre is 64.9% and 69.3% respectively.

It is apparent that after nano fibre generation crystallinity of hemp fibers increased significantly. This may be due to the removal of impurity on the fibers or attributed directly to attaching nanocellulose.

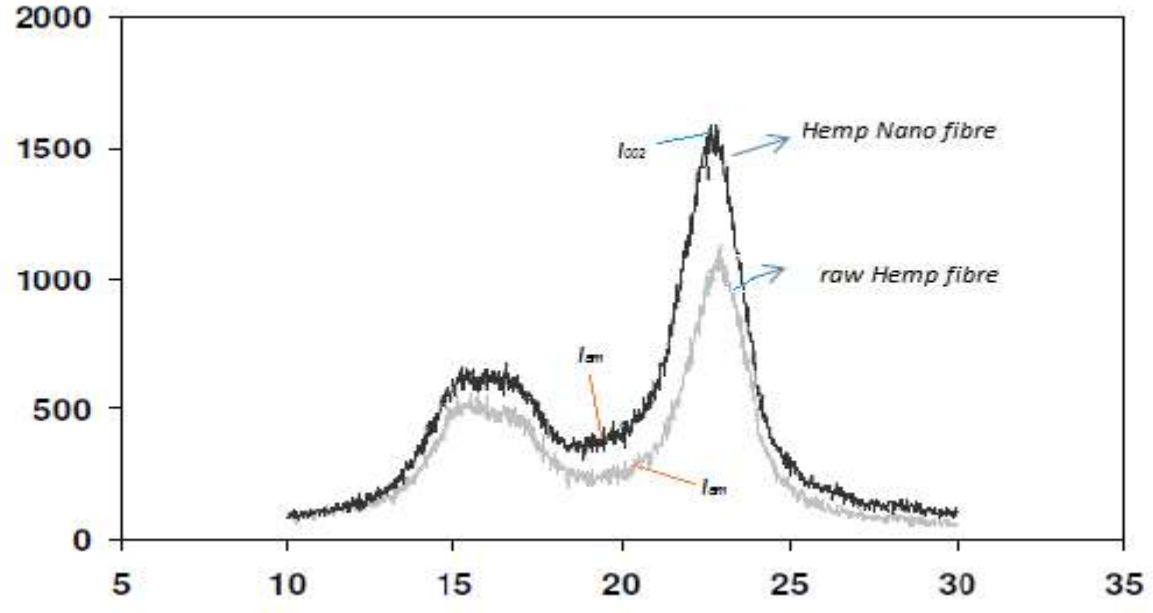

Figure -6X-ray diffractogram of Raw hemp fibre to Nano hemp fibre

Table No-1Crystallinity Index of Raw hemp to Nano hemp fibers.

Sample	2□ [0]		Fibre chemical composition			Crystallinity index %
	Iam	*I002*	*Cellulose*	*Hemicellulose*	*Lignin*	
Raw Hemp fibre	19.8	24.2	74	17	5	74.1%
Nano fibril Hemp	19.1	24.5	87	6	2	78.2%

3.4 Mechanical properties

Mechanical properties such as like tensile strength, flexural strength and impact strength of raw hemp and nanoHempfiber reinforced epoxy polymer composites are evaluated as per ASTM standards . Five samples were tested on each fibre volume fraction and the average value is tabulated as 2.The trend of the mechanical behaviours are also observed from the below graphs (Figure 7,8 and 9) indicated that the mechanical properties of composite material withnano fibre reinforcement showing 15 to 30% higher performance as compared with raw hemp fibre reinforced composites .This is trends is because of the high surface to volume ratio of nano fibre , crystallinity percentage and uniform fibre distribution throughout the matrix.

Table No -2 Comparison of Mechanical Properties of Raw Hemp Vs Nano Hemp fibre

Fibre volume fraction	Raw hemp fibre based composites						Nano hemp fibre based composites					
	Average Tensile strength (Mpa)	S.D	Average Flexural Strength (Mpa)	S.D	Average Impact Strength (Kg/m2)	S.D	Average Tensile strength(Mpa)	S.D	Average Flexural Strength (Mpa)	S.D	Average Impact Strength (Kg/m2)	S.D

2%	2473	33.04	28.2	1.11	2.35	0.15	3020	25.55	31	1.87	3.1	0.18
4%	2546	26.57	28.75	0.76	2.86	0.1	3450	26.43	32.13	0.15	3.25	0.08
6%	2856	27.3	29.5	0.15	3.12	0.14	3800	4.53	34.5	0.27	4.21	0.1
8%	3010	17.51	29.81	0.52	3.84	0.06	3900	26.95	34.5	0.27	4.64	0.02
10%	3085	6.07	29.2	0.25	3.5	0.24	3681	37.63	34.7	0.25	4.54	0.07

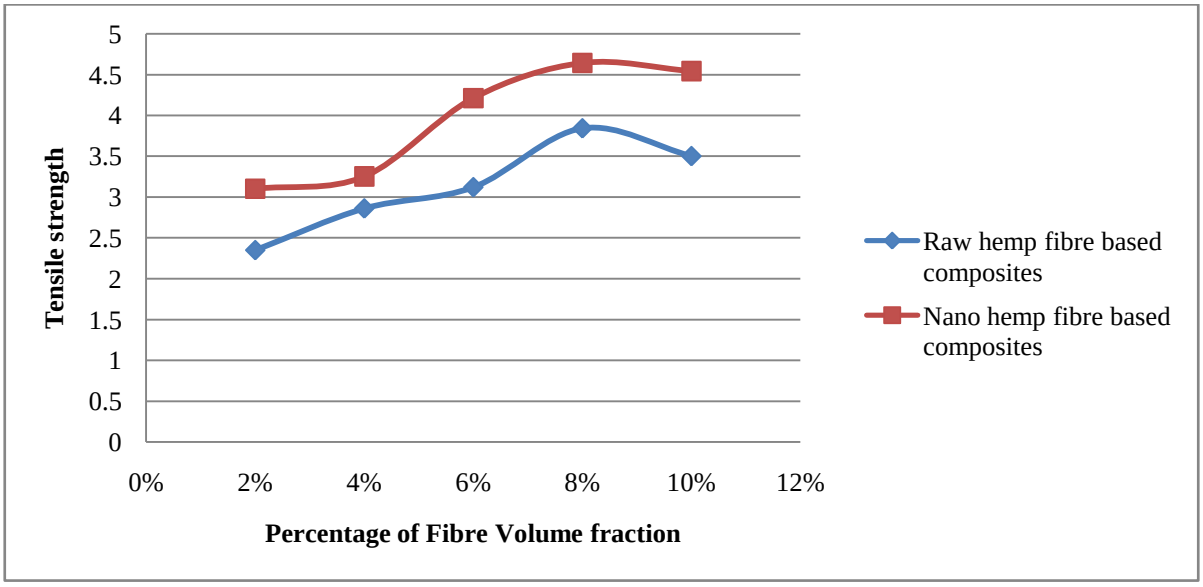

Figure-7 Comparison of Tensile properties

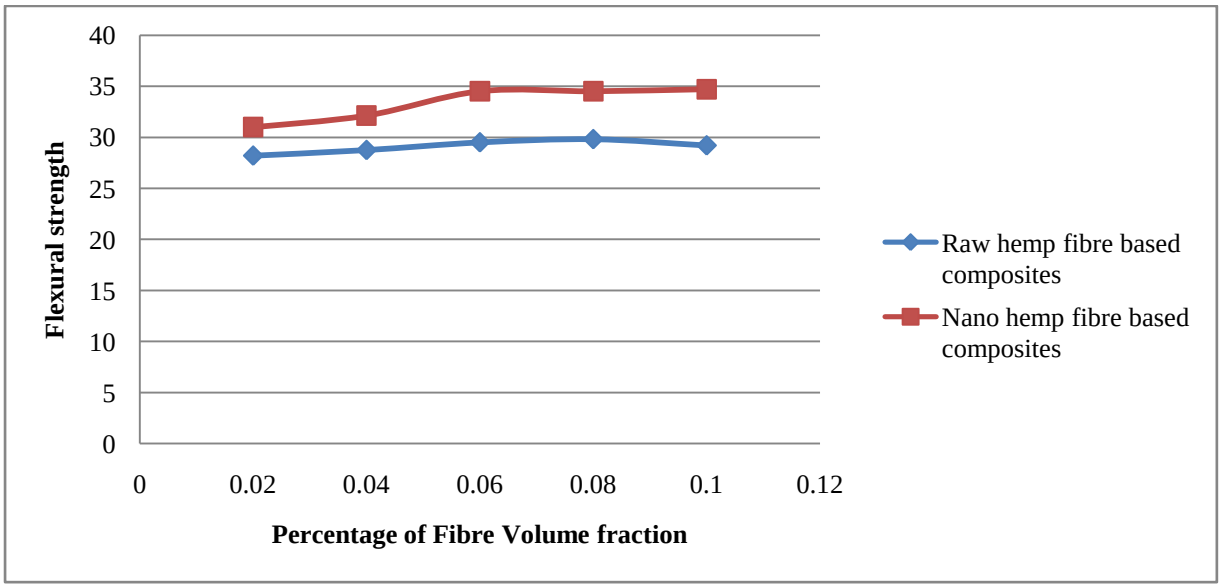

Figure-8 Comparison of Flexural properties

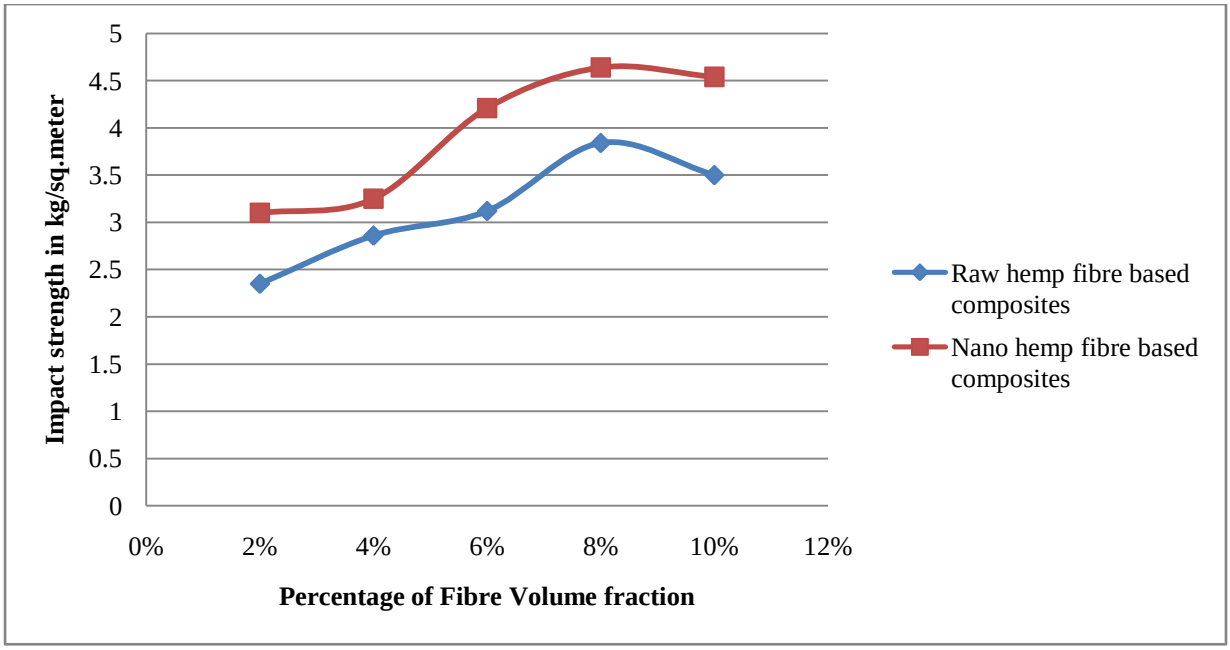

Figure-9 Comparison of Impact properties

3.5 Effect of Weight % of Raw HempFiber Reinforced EpoxyPolymer Composites on Mechanical Properties

It has been found that the tensile strength, flexural strength, and impact strength of the composite samples increase with an increase in the fibervolume fraction, for both raw hempand nano hempfiber reinforced epoxy polymer composites up to 8 % and thereafter decrease. The increase in mechanical properties composite materials are due tofiber-matrix interfacial bonding (Herrera-Francoet al 2005) and fibre strength component. The decrease in the mechanical properties attributed to the poor fiber matrix adhesion due to insufficient matrix component. Since more the fibre volume percentage results minimizing the resin component, which might have resulted in the micro-crack formation at the interface, and non-uniform stress transfer due to fiber agglomeration within the matrix (Weiming et al 2008 and Xia et al 2009).Similar observations were made by (Mohanty et al 2000). On comparing the raw hempfiber/epoxy composite with the nanohemp fibre reinforced epoxy composite, the latter was found to be superior in the mechanical properties. This is because; the raw hempfibers possess waxy substances and amorphous cellulose (Paakko et al 2007 and Orts et al 2005)not providing any strength component for the composites. In a nano hemp fiber reinforced composite, the waxy substances wereremoved and the cellulose associated with the matrix through hydrogen bonding. This cellulose provides higher strength component for the composites. This bond is flexible and compact, and so increases the mechanical properties.

3.6 Thermo Gravimetric Analysis (TGA)

This analysis is conducted to understand the behaviour of raw hemp fibre and nano hemp fibre composites towards various thermal condition.In the raw hempfiberreinforced composite, initially a sharp weightloss starts around 220 °C and extends upto 340 °C (Fig 10 A). This weightloss may be due to the loss of the absorbed water. After 360 °C there is adecrease, perhaps due to the elimination of hemicellulose and lignin (Plackettand Va'zquez 2004).Figure 10B shows the nano hempfiber reinforced epoxy composite;the steep decrease in the curve from 280 °C to 310 °C reveals the loss ofabsorbed water. After 310 °C, there is a shallow curve, indicating that lesswater was absorbed due to the non-availability of hemicellulose and lignin,and also due to a cellulose chain split (Fukuzumi et al 2009 andPark et al 2003).The bonds of C-C and C-O in the cellulose chain were broken(Capadona et al 2009). This can also be attributed to the decrease incrystalline cellulose chains caused by the thermally unstableanhydroglucuronate units present in the nanohempfiber (Fukuzumi et al 2010).

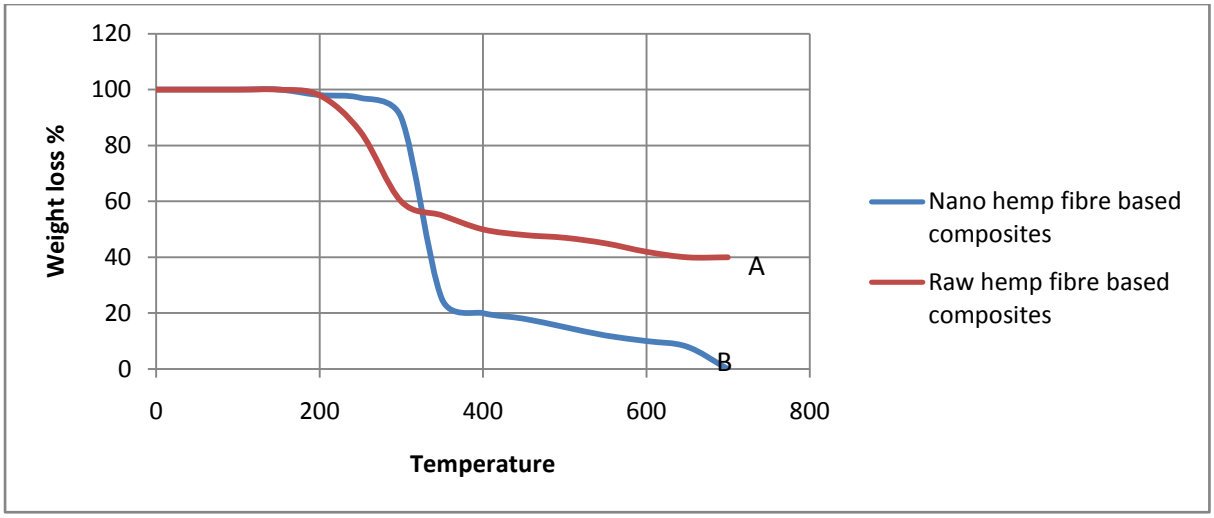

Figure-10 TGA analysis between raw hemp fibre composite to nano hemp fibre composite

IV. CONCLUSIONS

Nano hemp fibre and raw hemp fibre reinforced epoxy polymer composites prepared with different fibre volume fraction. Mechanical properties such as tensile strength, flexural strength, impact of the composites were determined according to ASTM standards.

The Surface morphology and crystallinity of the raw hemp fibre, nano fibre and their composite structures were studied by the SEM, FE-SEM and XRD. It revealed that high pressure steam defibrillation created an efficient reduction in the diameter of hemp fiber ,which is around 30~50 nm reveals .The XRD shows that crystallinity of the nano hemp fibre has improved by around 2.5% as compared with raw fibre due to reduction in hemicellulose composition ,which in turn increases the final composite mechanical properties. SEM image of composite structures revealed that fibres were scattered eventually thorough out the matrix surface due to even mixture of fibre with resin where as raw hemp fibre composite shows even distribution but fibre clusters were present in some places .Due to fibre cluster formation in raw hemp fibre structures the strength component of its composite became weaker as compared with nano hemp fibre. The tensile test results proved that over all mechanical properties (Tensile, flexural and impact) show 15-30% higher performance than raw fibre reinforced composite structures. The reason for this improvement is attributed that due to even distribution of fibre in the matrix, increased fibre crystallinity, absence of waxes and pectic maters and higher surface volume ratio of the nano fibre.

It has been found that the tensile strength, flexural strength, and impact strength of the composite samples increase with an increase in the fiber volume fraction, for both raw hemp and nano hemp fiber reinforced epoxy polymer composites up to 8 % and thereafter decrease. The increase in mechanical properties composite materials are due to fiber-matrix interfacial bonding. The decrease in the mechanical properties attributed to the poor fiber matrix adhesion due to insufficient matrix component.It is further understood that the composites prepared from raw hemp fibre and nano hemp fibres shows different TGA performance due to the elimination of fibre composition.

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