Investigation of Mechanical Properties of Jute Fiber Epoxy Reinforced by Synthesized Hematite (Fe_2O_3) Nanoparticles

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Abstract: Jute fiber was modified by epoxy resin and hematite nanoparticle. In this study alpha Fe_2O_3 nanoparticle in size at 22~23 nm made of sol-gel method commonly known as hematite was used. Chopped and scoured jute fibre was washed, neutralized and finally dried called modified fiber. Modified fiber was grafted with admixture of polyethylene glycol and aluminum salt and grafted sheet was then cut into small pieces. The raw jute fiber was layered using epoxy and pressed preparing Raw Jute Reinforced Epoxy Composite (RJREC). Similarly, the Bleached Jute Reinforced Epoxy Composites (BJREC) and Modified Jute Reinforced Epoxy Composites (BJREC) were also prepared. The Modified Jute Fibre Nano Disperse Epoxy Composites (MJRNDEC) was prepared by sandwiching three layers of Modified jute fabric with epoxy and nano particle. Epoxy sheet with nanoparticles and without nanoparticle were also produced. Produced samples were characterized and compared Tensile in terms of their mechanical properties. Obtained results show that modified jute fiber epoxy nano Fe_2O_3 dispersed composites exert better tensile and flexural strength compared to raw and bleached jute fiber epoxy composites.

Keywords: Hematite, Nanoparticles, Epoxy, Jute Fiber, Composites.

1. INTRODUCTION

Recently, scientist and engineers working in the field of materials are deeply concerned with sustainability issues and environmental protection. Therefore, environmental friendly, natural, recycled, or biodegradable materials are of great interest. Due to environmental friendly, bio degradability and sustainability, natural fiber composites are preferred as compared to conventional synthetic fiber based composites [1-4].Natural fibers are in considerable demand in recent years and play a key role in the emerging “green” economy. They are abundantly available, less costly, biodegradable, and easily recyclable and have low environmental impact. Therefore, fibre reinforcement in polymeric composites is growing day by day [5]. The major drawback is their high moisture absorption and poor dimensional stability, which stops their successful use in long-term composite applications. Their susceptibility to moisture absorption is the main causes of their poor mechanical properties[6].Polyester resin, as a structural polymer, was chosen as a polymer matrix because the cured resins are thermosetting with a network structure possessing high resistance to the moisture and chemicals, and good mechanical properties [7]. Nano structural materials such as nanoparticles (NPs) or nanofibers have been used as fillers in both the polymeric nanocomposites to improve the mechanical, electric, electronic and optical properties, and the metallic nanocomposite to control the electrodeposition. Hematite nanoparticle is prominent of them. Several researcher studied in recent years about hematite nanoparticles [8]. Hematite nanoparticles are extensively use in biomedical but it may cause some hazard which [9]. Hematite nanoparticles show four polymorphs. These polymorphs have magnetic behavior and transitions [10]. Alpha hematite nanoparticle is a good candidate for these purposes to improve the properties of Nano composite materials. The hematite iron oxides ($\alpha-Fe_2O_3$) are of great importance in technological and industrial applications [11]. Hematite exhibits high resistance to corrosion, therefore, it has been extensively used in many fields which include photo-anode for photo assisted electrolysis of water. Various techniques have been used to fabricate hematite nanomaterials of desired morphology (*e.g.* nanorods, nanofibers, nanowires, etc.) for various applications. In this work alpha hematite were synthesized from iron nitrite and citric acid by sol-gel method. The adding of alpha hematite nanoparticles changed the mechanical properties of Nano composite materials. The major objective of this paper is to explore a novel approach on mechanical properties of nanoparticles dispersed in to the polyester nanocomposites were developed and characterized.

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2. EXPERIMENTAL

2.1. Materials

For Hematite Nanoparticle, Fe_2O_3 nanoparticles were synthesized chemically by a modified sol-gel method. In this method, Iron Nitrate $Fe(NO_3)_3.9H_2O$ (Aldrich 98%), producer Loba Chemical Ltd, India, purity 98% and Mono Hydrated Citric Acid (Aldrich 98%) , producer Merck Chemical ltd, India, Purity 99.5% solution is used. For modification of Jute fiber and composite- detailed chemical used for the experiment are Jute fabric collected from Rajshahi, Bangladesh, Surf Excel (detergent) from Unilever Bangladesh, Aluminium Sulphate ($Al_2(SO_4)_3$, $7H_2O$, 99.5%), Polyethylene glycol (M.W.6000), Sodium Silicate, Hydrogen Peroxide (30%), Sodium triphosphate, Sodium Carbonate from Merck Co. Ltd, India, Epoxy resin and Amine from Dhaka, Bangladesh, origin Singapore.

2.2. Methods

2.2.1. Synthesis of Iron Oxide (α-Fe_2O_3) Nanoparticles

Firstly, Fe_2O_3gel are prepared by using sol-gel method. Secondly, Fe_2O_3 Nanoparticles powder prepared. To achieve this 200 ml (0.1 M) of Fe $(NO_3)_3.9H_2O$ drop wise added with vigorous stirring and gelated by 800 ml (0.1 M) of $C_6H_8O_7.H_2O$ [12]. Continuously heated with 70 ºCwith vigorous stirring Fe_2O_3 gel formed. This gel is then drying until the contained water was evaporated. The gel then annealed at 300 ºC with 180 minutes. Using Ball milling and Planetary milling Fe_2O_3 Nanoparticles are formed from dried gel powder. This work provides 1.61g of Fe_2O_3 ranging in size from 22-23 nm.

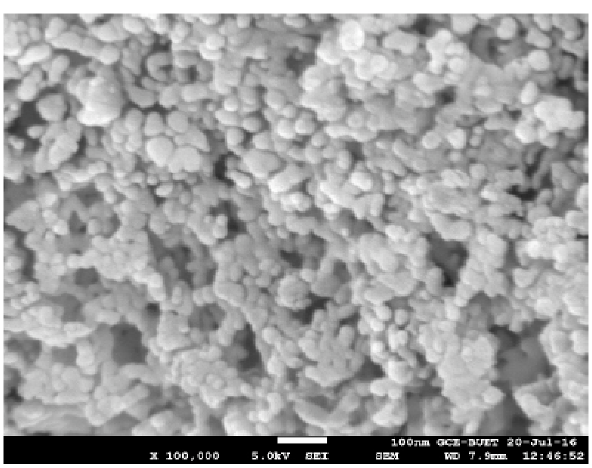

Figure 1: SEM image of iron oxide (Fe_2O_3) nanoparticles.

2.2.2. Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) Studies

The morphology of α−Fe_2O_3 nanoparticles studied by SEM is shown in Figure **1**. It is seen that, the produced iron nanoparticles are spherical shaped. Although some particles are agglomerated, most of the particles can be identified by nanometer scale. The average particle size of α-Fe_2O_3 was found as 27.65 nm.

2.2.3. Preparation of Jute Fabric for Reinforcement

Jute Fabric collected and sized into desired shape for firstly named after raw Jute Fabric. The sample cut into small pieces of 15 cm length and 12 cm width .Scoured Jute Fabric obtained from Raw Jute Fabric after scouring process. Scouring containing 2 g/l of sodium carbonate and 1 g/l of a nonionic detergent at a fabric-to-liquor ratio of 1:20 (W/V) at 60 °C for 30 min [13]. The samples were dried in an oven at 105 °C in order to remove moisture and then cool at ambient condition. The weights of dried samples were taken by analytical electric balance. These samples are referred to Scoured Jute Fabric. The next step is bleaching. Bleached Jute Fabric get frombleaching of scoured Jute Fabric. Bleaching occurred with a mix solution of several reagents containing 0.5% hydrogen peroxide (H_2O_2), 1.66% sodium silicate, 0.42% trisodium phosphate (Na_3PO_4), 0.08% sodium hydroxide and 0.08% detergent(surf excel) in a laboratory in a beaker at a fabric-to-liquor ratio of 1:6 (W/V) for 1 hour at 80 °C [14]. The Jute Fabric is then washed with distilled water mixed with dilute acetic acid for neutralization. The fibers again wash with distilled water. The samples were dried in an oven at 105 °C . These samples are referred to Bleached Jute fabric. Modified Jute Fabric is originated after modification of Bleached Jute Fibre. In this modification process the Polyethylene Glycol (PEG) is use for grafting *i.e*. modification occur. In optimum conditions of 100 per cent Polyethylene glycol (PEG) and 6 per cent $Al_2(SO_4)_3$ for 90 min at 70 °C to graft PEG onto the jute surface [15]. Now it was dried in oven at 105 °C. After dried, the treated jute were cut into small pieces of 15 cm long and 12 cm wide.

2.2.4. Preparation of Jute Reinforced Epoxy Composite

The Raw Jute Reinforced Epoxy Composites were prepared by sandwiching three layers of raw jute fabric [16]. In the sandwich, the weight ratios of raw Jute Fabric contents of 33.85 wt% were maintained by changing the weight of the Epoxy resin. Composites were made by pressing this sandwich at 105 °C for 1 hour under a pressure of 50 KN using a Carver Laboratory Press (model 2518). Then composites were cooled to room temperature using another press, cut into the desired size and kept in the desiccators. This sample is called Raw Jute Reinforced Epoxy Composite (RJREC). The Bleached Jute Reinforced Epoxy Composites (BJREC) was prepared by sandwiching three layers of bleached Jute Fabric. In the sandwich, the weight ratios of bleached Jute Fabric contents of 37.70 wt% were maintained by changing the weight of the Epoxy resin. This sample is called Bleached Jute Reinforced Epoxy Composite. The Modified Jute Reinforced Epoxy Composites (MJREC) were prepared by sandwiching three layers of Modified Jute Fabric. In the sandwich, the weight ratios of Modified Jute Fabric contents of 40.1 wt% were maintained by changing the weight of the Epoxy resin. The Modified Jute Reinforced Nano Disperse Epoxy Composites (MJRNDEC) was prepared by sandwiching three layers of Modified Jute Fabric. In the sandwich, the weight ratios of Modified Jute Fabric contents of 34.29 wt% and Nanoparticles contents 1 wt% were maintained by changing the weight of the epoxy resin. The Nano Disperse Epoxy Sheet (NDES) were prepared by mixing of Nanoparticles and Epoxy resin homogenously. In the Sheet, the weight ratios of Nanoparticles contents of 0.97wt% and Epoxy resin contents 99.03 wt% were maintained by changing the weight of the Epoxy resin. Epoxy Sheets (ES) prepared by using Epoxy resin with hardener .The resin with hardener were placed inside the dice and then it was placed in the hot press.

2.2.5. FTIR Spectroscopic Measurements of Bleached and Modified Jute Fibre

Chemical bonding test of bleached and modified jute fibre were accomplished by FTIR Spectroscopic Measurements. Study of FTIR spectroscopic measurements was carried out inCentral Science Laboratory, University of Rajshahi, Bangladesh. Model: Spectrum-100, FTIRSpectrum, Perkin Elmer. All the samples such as bleached jute fabric and Modified jute fabric were dried at 105 °C for 24 hours and the samples were cut into very small pieces. Then powdered by a mortar. The measurements were performed by mixing and grinding a small amount of the small pieces of samples (1 mg) with dry and pure KBr (200 mg). Mixing and grinding were accomplished in a mortar by a pestle. The powdered mixture was then compressed in a metal holder under a pressure of 8-10 tones to produce a pellet. The pellet was then placed in the path of the infrared beam of wave number in the range of 500-4000 cm^{-1} [18]. The infrared spectra of bleached and modifiedjute fibers are shown in Figure **2**. The bleached jute and modified jute fibre are showed by A and B respectively. From Figure **2** it is observed that the broad absorption band at (3200-3600 cm^{-1}) represents the hydroxyl (-OH) absorption peaks which are present in the bleached and modified jute fiber. In the bleaching process some of the hydroxyl (-OH) group of jute fiber oxidize to carboxylic group (-COOH). During the modification of jute fiber with PEG, the one hydroxyl (-OH) group of PEG react with the carboxylic group (-COOH) of bleached cellulose and form a glucoside bond (C-O-C) and a new -OH group created. The absorption of this newly formed -OH group enhances the intensity of hydroxyl absorption in 3200-3600 cm^{-1} region. Because of this enhance intensity of hydroxyl absorption makes more broad the peak of hydroxyl (-OH) group after modification. This broadens hydroxyl (-OH) peak shows that PEG chemically bonded with the jute macromolecule.

3. RESULTS AND DISCUSSION

3.1. Mechanical Properties

Mechanical properties such as Tensile strength (TS), Tensile E modulus (TEM), Flexural strength (FS),

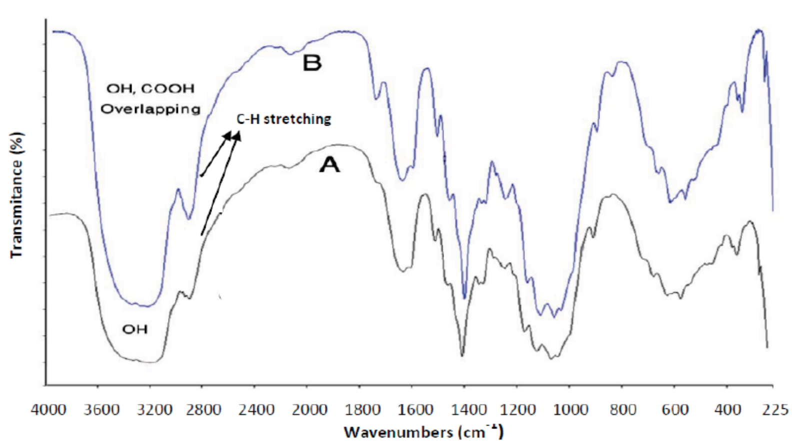

Figure 2: IR Spectra of bleached (**A**) and modified (**B**) jute fiber.

Flexural modulus (FEM) of ES, NDES, RJREC, BJREC, MJREC, and MJRNDEC have been measured. For samples, ES=Epoxy sheet, NDES=Nano disperse epoxy sheet, RJREC=Raw jute reinforced epoxy composite, BJREC=Bleached jute reinforced epoxy composite, MJREC=Modified jute reinforced epoxy composite, MJRNDEC=Modified jute reinforced nano disperse epoxy composite.

3.1.1. Tensile Strength and Flexural Strength

Tensile and flexural properties of samples are measured by universal tensile testing machine. The measured values are compared by the Figure **3**. Tensile Modulus and Flexural Modulus values are also compared by the Figure **4**. From the figure, the highest tensile value of composites is 46 MPa. This is contained by the RJREC. Because the RJREC contains much more structural ingredients 'Lignin' than other composites. On the other hand, the other composites contain the bleached and modified jute fibre as reinforcement. The bleached jute fibre contain less amount of Lignin and MJREC contain lowest value of Lignin because further chemical treatments *e.g.* scouring, bleaching, modification etc. The MJRNDEC possesses higher tensile strength and flexural strength value than MJREC because it contains Nanoparticles that act as a binder and made strong mechanical bond with enhancing surface properties. Between the two sheets, the EScontain poor tensile and flexural strength value than NDES because ES contain no Nanoparticles, thus no binding occur. These two sheets ES and NDES possess lowest value of tensile and flexural strength because their absence of reinforcement. In the two sheet and four composites the order for tensile and flexural strength are same direction. According to the above table the tensile strength and flexural strength value is orderly written as ES<NDES<MJREC<MJRNDEC<BJREC<RJREC.
These orders with tensile and flexural value are more understood from the Figure **3**. The flexural strength also measured by universal flexural strength testing machine. From the Figure **3,** ES possesses lowest flexural strength value and RJREC possesses highest value. Presence of Lignin and reinforcements are parameter that affects the flexural strength values. The highest Tensile Modulus value of four composites is 1095 MPa by (RJREC). RJREC contains much more structural ingredients 'Lignin' than other composites. On the other hand, the other composites contain the bleached and modified jute fibre as reinforcement. The bleached jute fibre contain less amount of Lignin and MJREC contain lowest value of Lignin because further chemical treatments *e.g.* scouring, bleaching, modification etc. The MJRNDEC possesses higher Tensile Modulus and Flexural Modulus value than MJREC because it contain nanoparticles, which act as a binder and made strong mechanical bond with enhancing surface properties. Between the two sheets, ES contain poor Tensile Modulus and Flexural Modulus value than NDES because ES contain no Nanoparticles. These two sheets ES and NDES possess lowest value of Tensile Modulus and Flexural Modulus because their absence of reinforcement shown in Figure **4**. According to the Figure **4**, Tensile Modulus and Flexural Modulus value is orderly written as ES < NDES < MJREC < MJRNDEC < BJREC < RJREC.

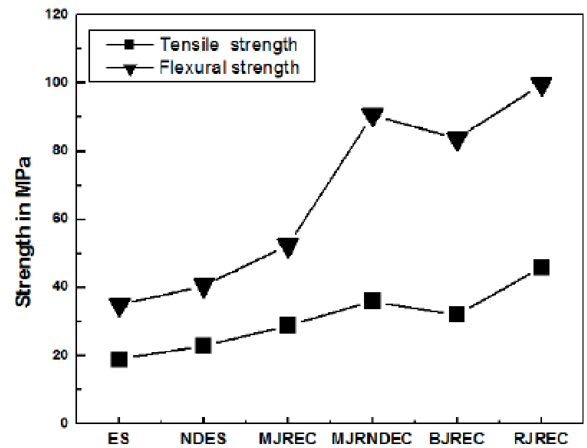

Figure 3: Comparison of Tensile strength and Flexural strength of various composites.

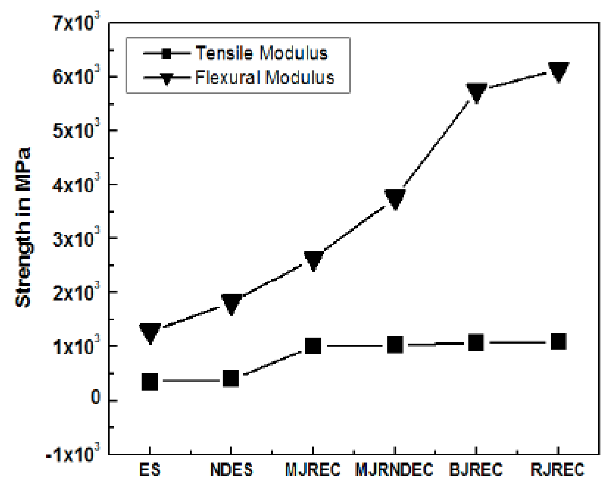

Figure 4: Comparison of Tensile Modulus and Flexural Modulus of various composites.

3.1.2. Vicker's Micro Indentation (Hardness) and Leeb Rebound

Jute reinforced composites have low tensile value than other hard materials. In order to evaluate hardness test low load are given and the hardness test are done by Vicker's micro indentation (hardness) test method. In this test method low load, *e.g*. up to 2 KG are given during Vicker's micro indentation (hardness) load [19].Leeb rebound (hardness) test also low load hardness test .This test isalso done for four composites and two sheets. This test is also in similar order as like as Vickers's micro indentation (hardness) test. The epoxy sheet has low value of Leeb Rebound hardness No. 653. The Modified Jute Reinforced Nano Disperse Epoxy Composite has high value of Leeb Rebound hardness No. 694.This cause epoxy sheet contains no reinforcement phase. The other composites like Modified Jute Reinforced Epoxy Composite, Bleached Jute Reinforced Epoxy Composite, Raw Jute Reinforced Epoxy Composite, Modified Jute Reinforced Nano Disperse Epoxy Composite posses higher value of hardness due to presence of jute as a reinforcement phase. Modified jute shows less value of Leeb Rebound hardness No. than bleached jute and raw jute reinforced composites. Modified jute contain low amount of lignin when it come under several chemical treatment for modification. NDES and MJRNDEC contain nanoparticles which arises strong bond on surface to the composite and this hard surface are liable for high Leeb Rebound hardness No.

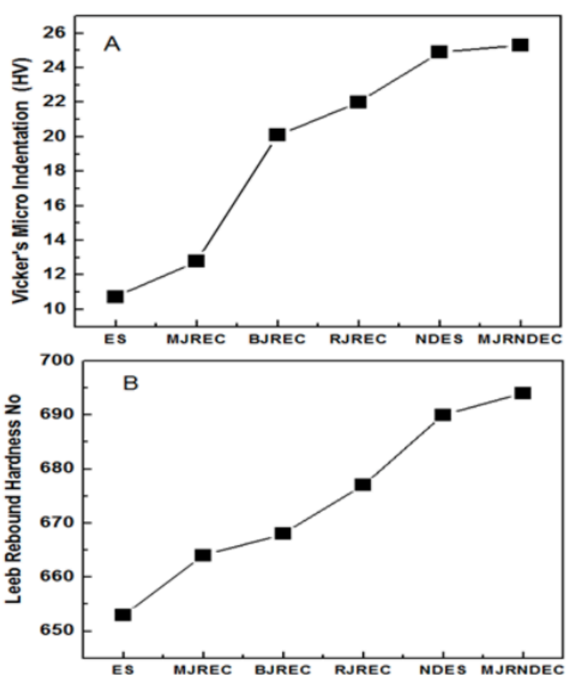

Figure 5: Hardness of various composites (**A**) for Vicker's Micro Indentation (HV) and (**B**) for Leeb Rebound Hardness No.

3.1.3. Epoxy Sheet vs. Nano (Fe_2O_3) Disperse Epoxy Sheet: Comparison

Epoxy sheet and Nano (Fe_2O_3) disperse Epoxy sheet both are absence of reinforcement phase. Epoxy sheet are fabricated by using hardener and epoxy resin with suitable temperature and pressure. Nano (Fe_2O_3) disperse Epoxy sheet are fabricated by using hardener, epoxy resin and Nano (Fe_2O_3) particles with suitable temperature and pressure discussed in earlier section 3. Special types of shape are used for make this sheets. There are several mechanical test are done for this two type of sheet for their characterization. The result of their mechanical test shows Figure **6** the difference between two sheets. Hence the epoxy sheet which contains nanoparticles that gives the better mechanical properties. The better mechanical properties comprises with the presence of nanoparticles.

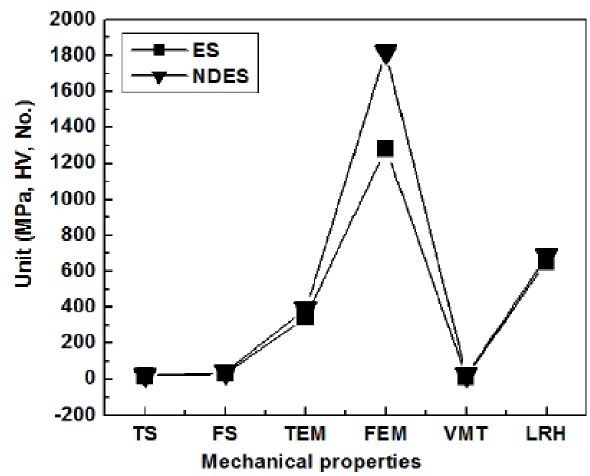

Figure 6: Comparison of various mechanical properties between ES and NDES.

3.1.4. Modified Jute Reinforced Epoxy Composite vs. Modified Jute Reinforced Nano (Fe_2O_3) Disperse Epoxy Composite

Modified Jute Reinforced Epoxy Composite is a composite where the modified jute is used as a reinforcement phase. The better mechanical properties obtain when nanoparticles are adding with the modified jute. Epoxy resin, hardener, nanoparticles, modified jute is used in this composite. The latter composite termed as Modified Jute Reinforced Nano (Fe_2O_3) Disperse Epoxy Composite shows better mechanical properties than Modified Jute Reinforced Epoxy Composite. Because it contain no nanoparticles, as a result the surface properties are lower than when nanoparticles adding. The tensile strength of Modified Jute Reinforced Epoxy Composite is 29 MPa when Modified Jute Reinforced Nano (Fe_2O_3) Disperse Epoxy Composite is 36 MPa. The Vickers hardness of Modified Jute Reinforced Epoxy Composite and Modified Jute Reinforced Nano (Fe_2O_3) Disperse Epoxy Composite is 12.8 HV and 25.3 HV respectively.

In this case, the epoxy sheet with nanoparticles gives the better mechanical properties *e.g.* tensile strength, flexural strength, micro hardness etc. For an example, the tensile strength of epoxy sheet is 19MPa where the tensile strength of nano disperse epoxy sheet is 23MPa shown in Figure **7**.

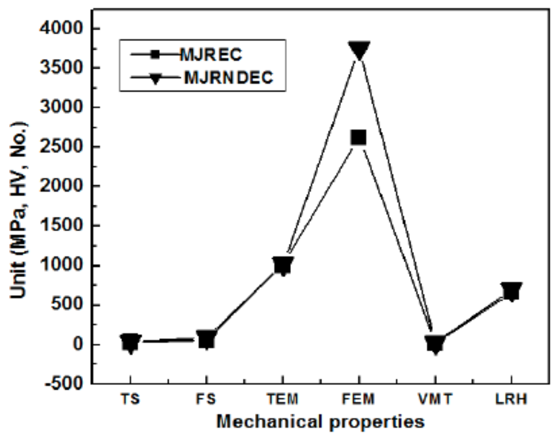

Figure 7: Comparison of mechanical properties between MJREC and MJRNDEC.

With this example the other properties also improved when Nanoparticles added with epoxy sheet. Nanoparticles improve the surface properties of the sheet. The mechanical bond and the interfacial bond are enhancing by the adding of Nanoparticles. By dint of this phenomenon, the nano disperse epoxy sheet shows the better mechanical properties. The other mechanical properties Tensile Modulus, Flexural Modulus, Vickers hardness, Leeb rebound hardness are improved.

4. CONCLUSION

Alpha-Fe_2O_3 nanoparticles size reduces with rising annealing temperature. It modifies the mechanical properties like tensile and flexural properties. Tensile strength and Flexural strength of epoxy sheet is greatly increased by 21% and 14% with combining with Fe_2O_3 respectively.

When this Fe_2O_3nano particles use to make Modified Jute Reinforced Nano disperse composite then the tensile strength increase with 24%. This result shows the improvement of properties, which enumerates that Fe_2O_3, modify and raise mechanical properties. Such as Tensile strength. Young's modulus, Flexural strength, Hardness etc. The raw jute fibre contain more lignin than bleached and modified jute fibre. Therefore, in this case naturally raw jute fabric ensure better mechanical properties than bleached and modified jute. Mechanical properties of this bleached and modified jute can enrich by adding nanoparticles. Fe_2O_3 raise the properties by virtue of its good surface properties. These led to greater demands for nano disperse natural fibre reinforced composites.

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