Mechanical and Morphological Properties of Cellulose Reinforced Composites

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Abstract. The development of bio-based polymer composite materials not only promotes a better environment but also assists to compensate for the gap between demand and supply of non- renewable materials. In the current research work, an attempt was made by fabricating cellulose reinforced polymer bio-composites in a step towards the development of sustainable and safe materials. Cellulose is extracted from the kenaf fibre using water pre hydrolysis synthesis. Bio composite materials are fabricated utilizing cellulose as reinforcement and isophthalic polyester as matrix phase. Mechanical (tensile and flexural) properties of the composite materials are studied as a function of cellulose loading. Tensile strength of the composite materials exhibited an increase (up to 10% cellulose) and decrease (from 15% cellulose) pattern with the respective percentage of reinforcement. Composite with 10% cellulose reported an almost 25% increase in tensile strength compared to neat resin. The fractured surfaces of composite materials were analysed by scanning with an electronic microscope. The fractured surface of 15% cellulose composite featured voids and non-uniform distribution of reinforcement. From morphological observations, the drop in strength in 15% cellulose composite materials can be attributed to the presence of voids and agglomeration of reinforcement.

Keywords: Composite materials · Bio-composites · Kenaf cellulose · SEM · Morphological

1 Introduction

With the rapid global industrial growth, there has been great demand for the supply of engineering materials. In view of industrial demand for engineering materials and also concerns over global warming, there is a huge necessity of materials which are environmentally friendly (biodegradable) and also abundant in nature so as to meet the gap between demand and supply. Over the past decades research fraternity has been working on the natural polymer composite materials for the utilization of these composite materials as an alternative to non-renewable metal, ceramic and synthetic materials in different engineering applications. Natural fibre reinforced polymer composite materials were found effective in many engineering fields and proved their potential for a

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wide range of applications automobile [1], marine [2], construction etc. This can be attributed to excellent properties like high specific strength, low density, non- toxic, biodegradability, economical, low abrasiveness and abundance availability of natural fibres [3]. Researchers have experimented fabrication of natural fibre polymer composites by reinforcing different types of natural materials like coir, jute, kenaf, sisal, banana fibre, coconut shells (in the form of both fibre and particulates) etc., with an intention to minimize utilization of synthetic reinforcements like glass fibre, carbon fibre etc., to maximum extent. All the natural fibre reinforcements are lignocellulose materials which generally have three common constituents: cellulose, hemicellulose and lignin in different proportions [4]. Cellulose is one of the imperative linear chain polymer constituents of natural fibres which generally consists of β-1,4 linked D-glucose units [5]. Cellulose promotes some of the significant properties like high specific strength and modulus, reactive surface and low density [6]. Apart from direct utilization of cellulosic natural fibres as reinforcements, researchers also exploited cellulose as reinforcement in fabrication of biodegradable polymer composite materials.

William J Orts et al. [7] experimented fabrication of polymer nanocomposites by reinforcing cellulose microfibrils. Authors reported a 5-fold increase in tensile strength and modulus of cellulose microfibrils starch plastics compared to neat polymer gels. While the authors reported decrease in mechanical properties of cellulose thermoplastic composites. From the study it was concluded that reinforcement of cellulose microfibrils involves complex interaction with matrix phases. M Hazwan Hussin et al. [8] presented a comprehensive study about the preparation methods, modification techniques, structural and chemical characterization of cellulose fibrils. With the detailed demonstration of significance of cellulose fibrils and their physicochemical characteristics, authors concluded that more focus is required for the development of efficient cellulose fibrils owing to high performance cellulose nanofiber composites for various science and technology applications. N Saba et al. [9] developed cellulose epoxy composite materials with different cellulose fibres percentage. In their study bleached softwood kraft pulp was used as precursor material. Authors analysed the mechanical properties of the cellulose epoxy composites with respect to cellulose fibre content. From the experimental results it was concluded that addition of cellulose fibres to epoxy resin resulted in composites with improved mechanical properties. Pronounced results were witnessed in the composites with 0.75% cellulose nanofiber loading.

The objective of present research study is to explore and exploit the natural lignocellulosic materials for the development of sustainable composite materials. In this context, the research work is carried out to prepare biodegradable bio composites by extracting cellulose from kenaf fibers of Malvaceae family. Utilizing polyester thermosetting resin as matrix material and cellulose fibrils as reinforcement composites were prepared by hand lay- up technique. These are then characterized accordingly for mechanical properties as per ASTM standards.

2 Materials and Methods

2.1 Raw Materials

In the current research study Kenaf fibres were procured from the local farms and are used as precursor materials for the synthesis of cellulose. Isophthalic polyester resin bought from local suppliers was chosen as matrix phase. Polymerization of resin is initiated by utilizing Methyl Ethyl Ketone Peroxide (MEKP) as a catalyst. The curing of the resin is accelerated by Cobalt naphthenate.

2.2 Synthesis of Cellulose Kenaf Fibres

Figure 1 Illustrates the preparation procedure of cellulose from kenaf fibres. The process adopted for the preparation of cellulose was taken from the research work of Sun et al. [10] Initially the precursor materials were cleaned with distilled water and chopped into small pieces of length 5 mm–10 mm. The dried moisture free kenaf fibres were subject to dewaxing in which fibres were boiled in toluene for almost 6 h followed by washing with ethanol. Subsequently the fibres were treated with 0. M NaOH under continuous stirring for 3 h in 50% volume of ethanol. The resulting fibres carried for hydrogen peroxide buffer solution treatment at four different concentrations of H_2O_2 (i.e., 0.1%, 1%, 2% and 3%) with continuous agitation. Then the mixture was handled for 15 h stirring with NaOH and $Na_2B_4O_7.10\ H_2O$. Lastly, the mixture was treated with HNO_3, 70%. Cleaning of cellulose fibre was carried out by ethanol followed by distilled water and then dried in the oven at 60 °C temperature till moisture free.

2.3 Preparation of Cellulose Polyester Composites

The Cellulose microfibrils obtained from the pre hydrolysis of kenaf fibres were utilized as reinforcement in the fabrication of cellulose polyester resin. The composites were developed with three different reinforcement loadings. A simple hand layup technique was used for the preparation of composites. An open wooden mould in rectangular geometry with dimensions of $180 \times 280 \times 5\ mm^3$ was prepared. A plastic glassy sheet placed in mould to serve the bottom surface of the composites, which assists in easy removal of composites from mould after curing. Measured quantity of cellulose fibrils i.e., 5% taken into the beaker and mixed with appropriate calculated amount of polyester resin. Methyl ethyl ketone peroxide is added to the resin which acts as catalyst and resin also blended with cobalt naphthenate as an accelerator. Both the catalyst and accelerator assist in the polymerization process of the polyester resin composite composites. The composite mixtures in the mould left for curing at room temperature for 72 h. Similar procedure is adopted for the preparation of 10% and 15% cellulose polyester composites. The cured composite samples taken out from the mould and cut as per dimensions of ASTM standards.

2.4 Mechanical Tests

Cellulose polyester composite sample of dog bone shape with dimensions length - 140 mm, width at centre-10mm and thickness 4 mm tested for tensile strength on per-cievestar digital universal testing machine (UTM) as per ASTM D3039–76. The tensile

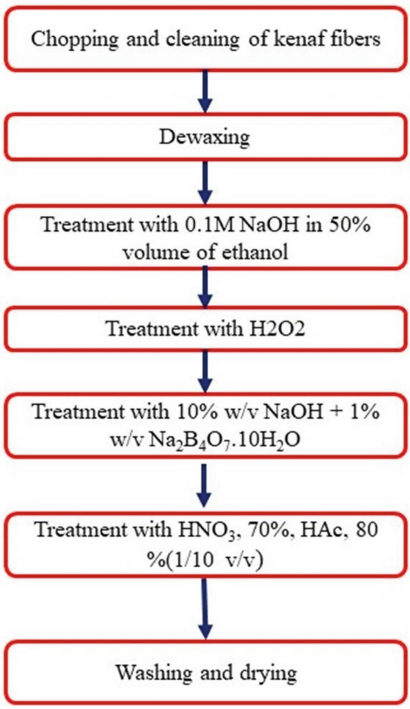

Fig. 1. Extraction of cellulose fibrils from kenaf fibres

test was carried out with cross head speed / rate of loading of 2 mm/min. Three point bending test was performed according to ASTM D2344–84 standards on percievestar universal testing machine. Each test is carried on five composite samples and average values are recorded.

2.5 Scanning Electron Microscope (SEM) Analysis

Fracture surfaces of the samples tested for tensile strength were investigated using a scanning electron microscope using Nova Nano SEM 450 at an accelerating voltage of 15 kV. The analysis was carried out at different scales and resolutions. The samples were gold coated prior to analysis.

3 Results and Discussions

3.1 Tensile Strength

Figure 2a illustrates the tensile test results of the cellulose polyester composites as function of reinforcement loading percentage. From the results it can be noticed that irrespective of percentage of filler loading the tensile strength of the cellulose polyester composites is enhanced compared to polyester resin. The strength of the polymer matrix

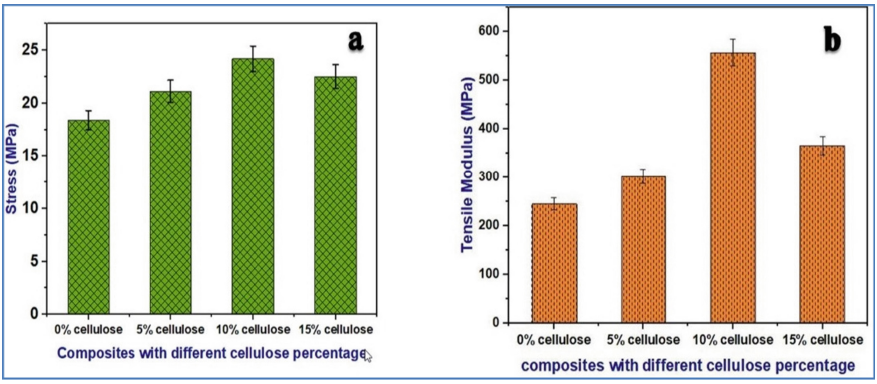

Fig. 2. Tensile strength and tensile modulus of the cellulose polyester composites materials respectively

composites mostly relies on the interaction and interfacial bonding between the polymer matrix and reinforcement [11]. The increase in tensile strength of the cellulose polyester composites demonstrated in Fig. 2a can be attributed to addition of cellulose filler and strong interfacial bonding between resin and cellulose. The cellulose fibrils reinforcement in composites act as load bearing sites by distribution of the stress thereby increasing the resistance to tensile loading. H D Rozman et al. [12] studied tensile behaviour of kenaf polyester composites. From the results presented in their study it can be noticed that addition of kenaf fibre to polyester improved tensile strength of the composites. The current study tensile strength results of cellulose composites observed higher than the tensile strength of 40% kenaf fibre reinforced polyester composites reported by H D Rozman et al. [12]. The values of tensile Modulus of cellulose polyester composites are shown in Fig. 2b. It is noticed that tensile modulus cellulose composites enhanced with filler loading up to 10% cellulose composites and then decreased with further increase in filler loading. The maximum tensile modulus is reported in the 10% which is 178.1442% higher than the tensile modulus of 15% composites. The descent of tensile modulus in 15% composites may be due to poor wetting.

3.2 Flexural Strength

The bending strength of cellulose composites was investigated using a three-point bending test and results obtained are shown in Fig. 3. Flexural strength of the composite mainly refers to the resistance of the composites to bending load. From the Fig. 3 it can be noticed that flexural strength of the cellulose polyester composites exhibited similar behavior as tensile strength i.e., bending strength of the composites improved with the addition of cellulose fibrils to polyester resin. Maximum flexural strength observed in the 10% cellulose composites, this may be considered due to strong interfacial bonding at the interface of cellulose fibrils and polyester matrix. Similar flexural strength behavior reported was in Cuong Manh Vu et al. [13] study.

Authors observed that upon addition of cellulose fibrils polymer matrix the bending strength of the cellulose thermosetting composites have increased due to strong bonding between the matrix and reinforcement.

In the current it is also evident from Fig. 4 bending resistance of the composite materials reduced in 15% cellulose composites. Because when the filler reinforcement increases beyond 10%, the polymer resin is insufficient for wetting of matrix and reinforcement which lead to early failure of the composites when tested in bending.

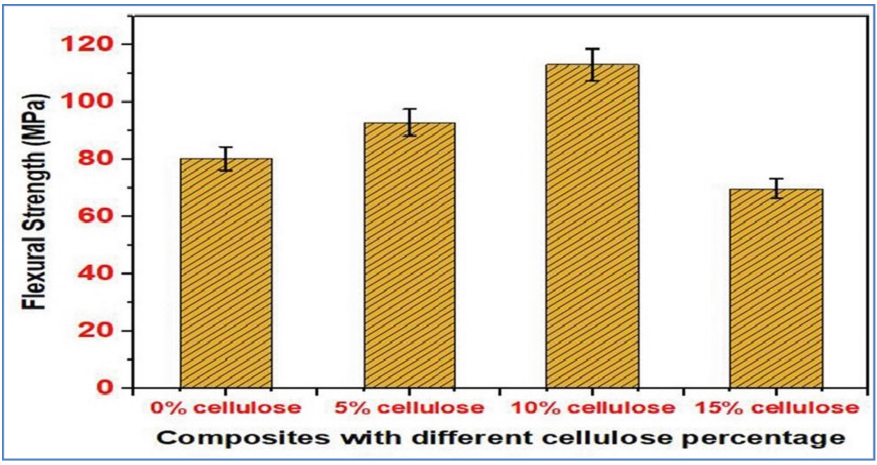

Fig. 3. Flexural strength of cellulose polyester composites

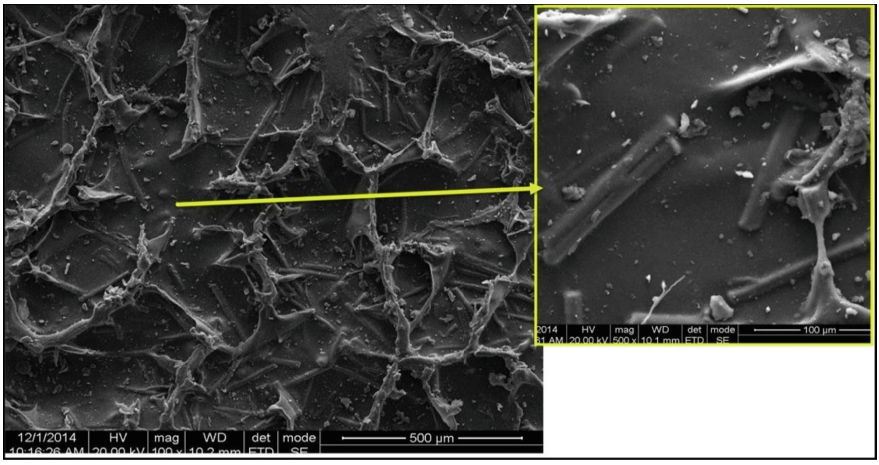

Fig. 4. Morphology of 10% cellulose composites fracture under tensile load

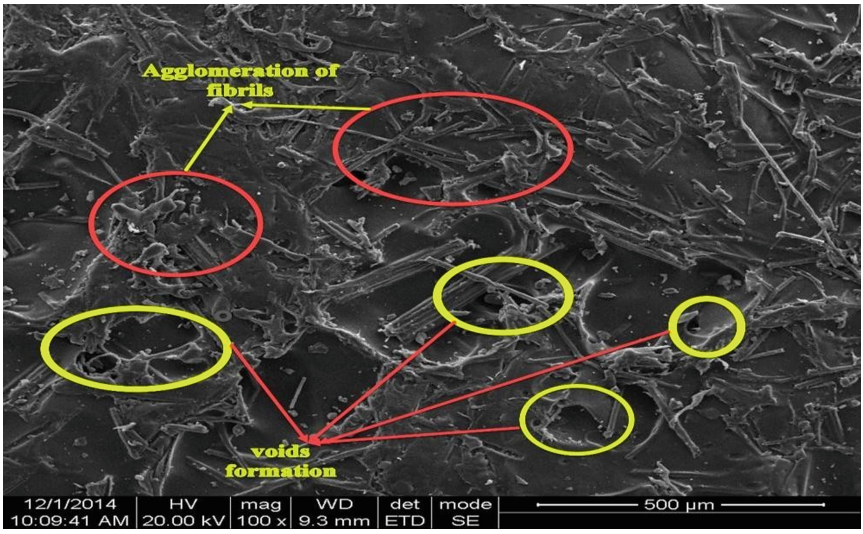

Fig. 5. Morphology of 15% cellulose composites fracture under tensile load

3.3 Morphological Studies

The fractured surfaces of composite materials tested for tensile strength were analysed using a scanning electron microscope. Morphology of tensile tested 10% cellulose composites presented in Fig. 4. The surface witnesses randomly blended cellulose fibrils into the polyester resin. The magnified view of the fracture surface has smooth exterior with no evidence of crack which indicates the brittle mode of failure [14].

Figure 5 illustrates the morphology of the fractured surface of 15% cellulose polyester composite material. It can be clearly observed that cellulose fibrils are randomly distributed in the polyester resin. It is also evident from Fig. 5 that the surface has a number of void formations and lumps of fibrils. The presence of voids in the composite materials results in poor mechanical properties [15]. Therefore, the reduced tensile and flexural strength in 15% cellulose composites can be ascribed to void formation and poor interfacial bonding. Detachment, voids and air bubble formation are relatively high when compared to 10% composites.

4 Conclusions

From the observations of experimental results of the cellulose polyester composites, it could be realized that reinforcement of cellulose fibrils that extracted from kenaf fibre have improved mechanical properties of the cellulose polyester composites. 10% cellulose reinforcement yielded relatively better mechanical properties; therefore, it can be concluded that 10% cellulose fibrils is optimum reinforcement for fabrication of cellulose polyester composites**.** From the comprehensive morphological studies, it can be comprehended that void formation and poor wetting lead to failure of composites.

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