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A Study on Characterization and Comparison of Alkali Treated and Untreated Coconut shell Powder Reinforced Polyester Composites

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Abstract - Natural Lignocellulosics material like coconut shell has an outstanding potential as reinforcement in thermoset matrix. In this study, Composites were prepared by incorporating treated and untreated coconut shell powder at different filler contents (0, 10, 20, 30, 40, 50 php) into Unsaturated Polyester matrix. An alkali treatment was used to enhance both the matrix filler wetting and the chemical surface modification in order to improve the physicochemical interactions at the filler–matrix interphase. A catalyst, Methyl Ethyl Ketone Peroxide (MEKP) was used to initiate the polymerization reaction. The results from Universal testing machine revealed that increased in coconut shell content have increased the tensile & flexural strength. Treated coconut shell filled composite showed slightly higher mechanical properties and lower water absorption than the untreated one. The chemical bonding between the filler and polymer matrix were characterized by using the Fourier Transformer Infrared Spectroscopy (FTIR). Result from scanning electron microscopy (SEM) showed that increased in filler content have increased the tendency of filler-matrix interaction.

Keywords: Universal Testing Machine, Unsaturated Polyester Resin, Methyl Ethyl Ketone Peroxide, Alkaline Treatment, Scanning Electron Microscopy, Fourier Transformer Infrared Spectroscopy (FTIR).

1. INTRODUCTION

Composite materials are widely known for its high strength to weight ratio. These are the materials that having strong load carrying material (known as reinforcement) imbedded in weaker material (known as matrix) [1]. Natural fibers have been widely used to reinforce materials for over 3,000 years. The advantages of the natural fibers are low cost, low density, good thermal isolating properties, being recyclable and a renewable source that does not affect the environment [2].To improve the mechanical, physical and other properties, or to facilitate processing and reducing the cost, natural fibers has been used as reinforcing or filler materials [3].Natural filler like a coconut shell powder is made from the most versatile part of the coconut shell and it is organic in nature also. The incorporation of coconut shell powder as a matrix material into thermoset resin is reduced the production cost of the molded product. Polyester resins are group of general purpose thermoset having average mechanical properties, lower resistance to temperature, higher co-efficient of expansion and low cost [3]. Now days, the cost is one of the most important factor. The overall cost can be reduced by blending the polymer with low cost filler materials like coconut shell powder [4].

The objective of this paper is to prepare the alkali treated and untreated coconut shell powder Reinforced Polyester composites by using different content of coconut shell powder (0, 10, 20,30,40,50 php) and characterize it by using different testing equipment.

2. MATERIALS AND METHODS

2.1 Materials

Unsaturated Polyester Resin (UPR), grade "KPR 6600", the catalyst used, MEKP-Methyl Ethyl Ketone Peroxide (MEKP) and cobalt accelerator were supplied by KEMROCK Industries & Export Ltd, Halol. The Coconut shell powder (CSP) was obtained from VG Tinder Products (P) LTD, Salem. A Particle size analyzer was used to obtain an average filler size 80 µm.

2.2 Formulations

The UPR/CSP composites were prepared by mixing unsaturated polyester with treated and untreated CSP using different filler content. The formulation of UPR/CSP composites is given in Table 1 and the chemical composition of coconut shell is given in Table 2.

TABLE 1
FORMULATION OF UPR/CSP COMPOSITES
php = Parts per hundreds of total polymer

TABLE 2 : CHEMICAL COMPOSITION OF CSP

Composition	Wt.%
Lignin	29.4
Pentosans	27.7
Cellulose	26.6
Moisture	8
Solvent Extractives	4.2
Uronic Anhydrides	3.5
Ash	0.6

2.3 Alkali Treatment

This treatment was used to enhance both the matrix and filler wetting and the chemical surface modification in order to improve the physicochemical interactions at the filler–matrix interphase. Here soaking and stirring the CSP in 5%wt NAOH aqueous solution for about 3 hours at room temperature. Then CSP was filtered out and washed several times with distilled water until NAOH was eliminated that is until the water no longer indicated any alkalinity reaction before being dried up in an oven at 60°C for 24 hours.
The scheme of the reaction is:

FILLER -OH + NaOH => FILLER-O-Na^+ + H_2O

2.4 Sample Preparation

Mixing of the UPR/CSP composite was carried out by casting process. The UPR and the Pre requisite amount of filler content were homogeneously mixed by using Mechanical Stirrer. Accelerator and harder were added in the homogeneous mixture. Vacuum was applied for 5 min to remove air bubbles from the mixture. The homogeneous mixture was poured into the 300 x 300 x 5 mm casting mold. The casting mold was kept at room temperature for curing for the duration of 24 hrs. The UPR/CSP composites were prepared in different compositions i.e. 0wt%, 10wt%, 20wt%, 30wt%, 40wt%, 50wt% by mixing UPR with treated and untreated CSP.

3. TESTING AND CHARACTERIZATION

3.1 Fourier Transformer Infrared Spectroscopy (FTIR)

FTIR is a very simple technique and widely used for analysis and determination of plastic/polymer structure. It is a powerful tool for identifying the types of chemical bonds in organic and inorganic molecule by producing an infrared absorption spectrum that was like a molecular "fingerprint". The range of the wave number studied was 4000 to 450 cm^{-1}.

Materials	Composites
Unsaturated Polyester Resin (php)	100
Treated and Untreated Coconut Shell Powder (php)	0,10,20,30,40,50
MEKP (php)	1.5
Cobalt (php)	7

3.2 Scanning Electron Microscopy (SEM)

Studies on the morphology of the tensile fracture surface of the composites were carried out by using a Scanning Electron Microscope (SEM).This test was conducted by using SEM equipment Model JEOL JSM- 5610LV at Faculty of Technology, MSU (Maharaja Sayajirao University), Baroda. The fractured surfaces of specimens were mounted on aluminum stubs and sputter coated with a thin layer of palladium to avoid electrostatic charging during characterization.

3.3 Tensile & Flexural Strength

Tensile test was carried out according to ASTM D 638 on an Instron Tensile Test Machine Model LR100KLloyds. Composite samples with 4 mm in thickness, 13 mm in width and 50 mm gauge length were cut from the molded sheet using cutting machine. The cross head speed of testing was 5 mm/min and the test was performed at 25 ± 3ºC. Tensile strength was measured on 5 identical samples for each composition and the average values were reported.
Flexural test was conducted as per ASTM D 790 using the same UTM. The width and thickness of the samples were 14mm and 4 mm respectively.

3.4 Swelling Behavior

The composite samples were dried at 80ºC for 24 hours and then immersed in distilled water at room temperature. To determine the water absorption, weighing of the samples at regular intervals has been done. All the specimens were taken out of the water periodically and wiped with tissue paper and then weighed. For each sample, at least three specimens were used. A digital scale-precise was used with a precision of ± 1 mg to weight the samples. The Percentage of water absorption, Mt was calculated by:

Mt = (Wn –Wd)/ Wd *100%

Where Wd and Wn are original dry weight and weight after exposure respectively.

4. RESULTS AND DISCUSSION

4.1 Fourier Transformer Infrared Spectroscopy

The FTIR spectra of Bio filler treated with NaOH confirmed the delignification. After mercerization the band at 1739.02 cm^{-1} and 1245.73cm^{-1} due to stretching vibration of >C=O and >C-O group respectively disappeared. These groups are present in lignin and hemi cellulose structure in both soft and hard wood the >C=O peak (1739.02cm^{-1}) due to xylem is realizably unmoved after alkaline treatment.

Delignification produces the very characteristics removed of the absorption bands at 1508.65 and weaker band at 1245.73cm^{-1}. The band at 1464.81cm^{-1} is due to –CH band of -CH_2 and -CH_3. As expected, such absorption band was not present in unmodified filler. The bond at 2901 cm^{-1} is due to –CH structure in glucose moiety.

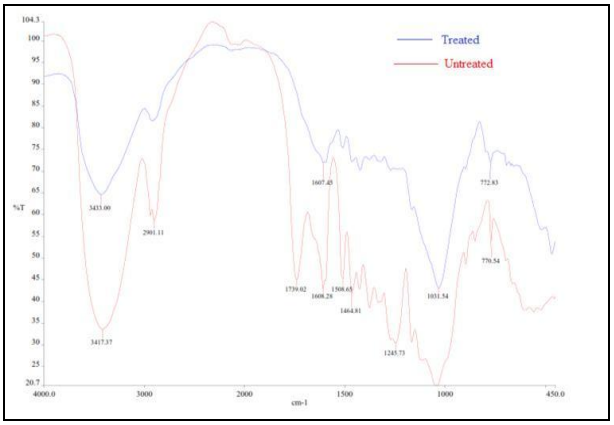

Fig. 1 FTIR Spectrum for treated and untreated CSP

4.2 Scanning Electron Microscopy (SEM)

The Scanning Electron Microscope was used to examine the tensile fracture surface of Polyester/CS composites at 30% of treated and untreated filler content as shown in figures 2-3. It shows that the occurrence of agglomeration in the composites in the presence of fillers. The formation of agglomerates is less in treated composites compared to untreated one. This tendency is due to good filler-matrix interaction. Figure 2 shows that agglomeration is less compared to figure 3.

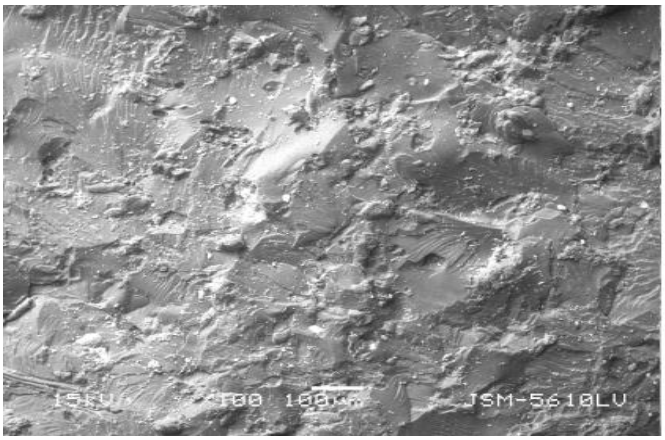

Fig. 2 SEM micrograph of tensile fracture surface of UPR/CSP composites (30% treated) at magnification of 100X.

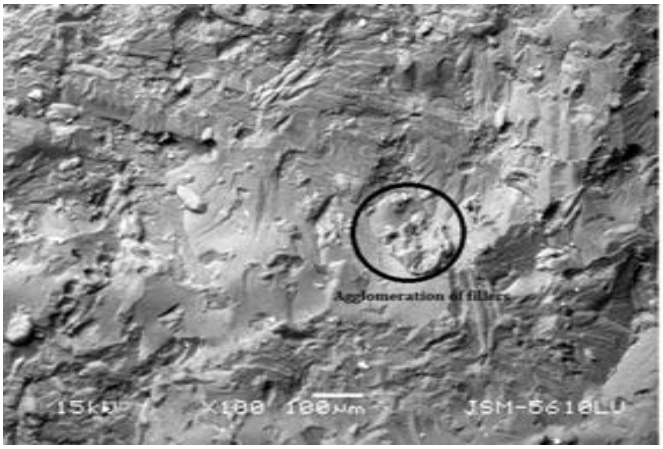

Fig. 3 SEM micrograph of tensile fracture surface of UPR/CSP composites (30% untreated) at magnification of 100X.

4.3 Tensile and flexural strength

Figure 4 & 5 shows the Effect of filler content on Tensile strength and Flexural strength of Polyester/CS Composites. As we can see, the strength of the composite decreased with addition of 10 php filler content and after that it starts to increase with addition of 20, 30 and 40 php [5]. The decreased of strength may due to the insufficient of fillers to reinforce the matrix of the composites. As the filler content increased, filler is sufficient to reinforce the matrix of the composites as there is a strong interface between the filler and the resin. Now with the filler content of 50 php, the strength is decreased as there is an excess filler to reinforce the matrix of

the composite. So there is no strong interface between all filler particles and resin. The lignin content in coconut shell is very high i.e., 29.4%. The bio-flour materials are a complex network of three polymers: cellulose, hemicelluloses and lignin. According to Kim et al., [6] lignin holds the bio- flour together and also acts as a stiffening agent for the cellulose molecules within bio-flour cell wall. So, the lignin and cellulose content of CS increased the tensile and flexural strength of coconut shell filled Polyester composites.

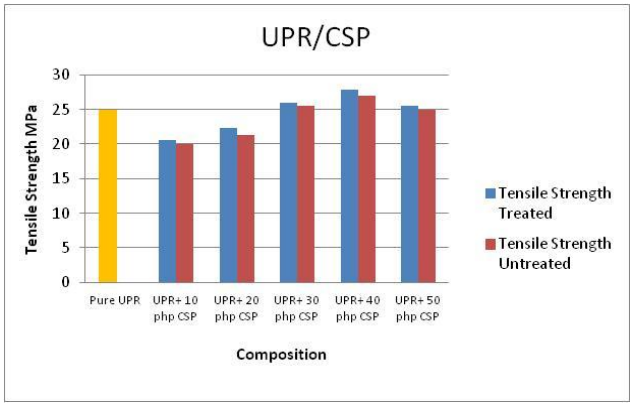

Fig. 4 The effect of filler content on the tensile Strength of UPR/CSP composites

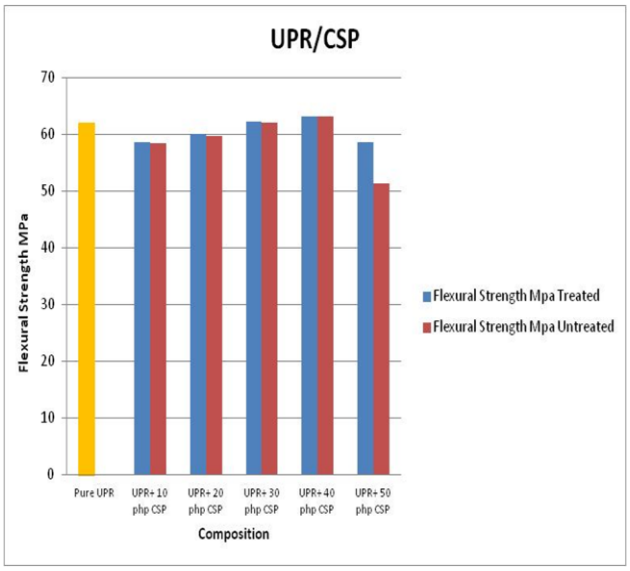

Fig. 5 The effect of filler content on the flexural strength of UPR/CSP composites

4.4 Swelling behavior

Natural fillers are hydrophilic materials with many hydroxyl groups (-OH) in the structure [3]. The hydrophilic nature of the CS leads to the formation of hydrogen bonds between filler and water molecules and absorbs water. Due to the presence of (-OH) groups, Coconut shell filler shows low moisture resistance. Treated filler shows lower moisture absorption than the untreated one. The reason is during the treatment the number of –OH group is reduced. Figure 6 shows the graph of Percentage of Water absorption versus Time on Polyester/CS Composites.

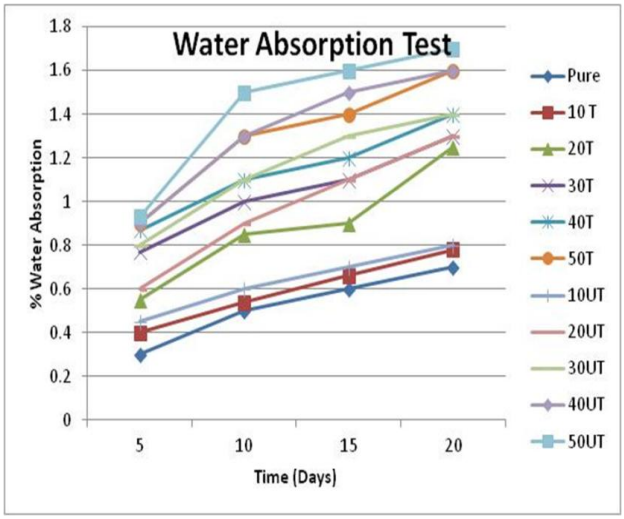

Fig. 6 Percentage of Water absorption versus Time on UPR/CSP Composites.

5. CONCLUSIONS

The effect of Coconut shell content of UPR/CSP composites on mechanical properties, swelling behavior, and FTIR was studied. The results show that the Tensile strength, Flexural strength and Water absorption of UPR/CSP composites increased with the increasing CSP content. The treatment on filler shows better properties than untreated one. FTIR Study indicates that the tendency of filler-matrix interaction improved with the increasing filler in Polyester matrix.

6. ACKNOWLEDGEMENT

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