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Experimental Investigation on Surface Characterisation of Chemical Treated Natural Fiber Composite

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Abstract: ***In this research work removal of lignin and hemicelluloses for untreated coir fiber reinforced composites was accomplished by suitable chemical treatment namely new method at optimum fiber loading such as 25% and 30%. To avoid the problem of untreated coir fiber, chemical treatment was done. Final treated fiber extraction process was gone through new method. It results in treated cellulose composite at 25% and 30% lignin and hemicelluloses was found to be decreased was revealed in Fourier transform infrared spectroscopy analysis, it shows defibrillation, depolymerization in treated composite was revealed in scanning electron microscopy analysis, and shows high oxygen content percentage than carbon content percentage about 22.06% which demonstrates removal of lignin in treated composite, was revealed in element detection analysis.. The main objective was accomplished by the removal of lignin and hemicelluloses of natural fiber through suitable chemical treatment and at optimum fiber loading.***

Keywords: Lignin, Hemicelluloses, Scanning Electron Microscopic, Fourier Transform Infrared Spectroscopy, Element Detection Analysis

1. Introduction

Now a day's composites are used in wide variety of applications because of its constituent material can regain their original properties even after mixing. In polymers many research under gone through synthetic because of their promising properties. In this study natural Fiber reinforced polymer was investigated and chosen material was coir fiber because it is natural, low cost, low density, high specific strength and environmental friendly. The drawback of natural fiber has high moisture absorption capacity, and it consists large amount of lignin, hemicelluloses , pectin and wax which was amorphous in nature, due to which it possess low strength, and poor dynamic characteristics.. Thus in this study various chemical extraction processes was carried out namely New Method[1] and cross and Bevan method [2] to remove lignin, pectin, hemicelluloses content , reducing wear and to improve adhesion between fiber and matrix. Finally Treated cellulose fibril is extracted from New Method because large quantity of treated cellulose was obtained from new method, and in new method fiber was subjected to mild chemical treatment it does not affect cellulose content which tends to reduce lignin and hemicelluloses contents alone. Thus further extraction process gone through new method for 800 grams. After Extraction process obtained treated cellulose was 350 grams, for which composite sheets were made at different weight percentage such as 25% and 30% in case of both treated and untreated fiber by compression molding .To reduce moisture content and to improve strength reinforcement is done and matrix used for reinforcement is epoxy. For obtained composite sheets morphological analysis was carried out in scanning electron microscopic (SEM), for chemical composition analysis FT-IR (Fourier Transform Infrared Spectroscopy) analysis was carried out, and finally element detection analysis was carried out by EDX Technique to check weight percentage of carbon and oxygen. The microscopic analysis results at treated cellulose sample defibrillation, Depolymerization occurs, and material becomes soft comparison to untreated sample. In FT-IR absence of lignin and hemicelluloses was observed in both 25% and 30% of fiber loading, in element detection analysis it reveals that oxygen content is larger than carbon in case of treated composite ,it proves that amount of lignin content was reduced. Thus due to chemical treatment it demonstrates removal of lignin and hemicelluloses in case of treated cellulose composite at 25% and 30% of fiber loading comparison to untreated composite. The main theme of this research was to remove lignin and hemicelluloses content of untreated coir fiber reinforced composites through suitable chemical treatment namely new method [1] thus it results high removal Rate at 25 % and 30 % of treated cellulose composite.

2. Experimental

a) Materials

Coconut fiber is extracted from the outer shell of a coconut. Brown fibers are thick, strong and have high abrasion resistance. Density 1.4 g/cc Single fiber, Breaking Elongation 30%, Moisture regain at 65% RH 10.5%,

b) Methods for the extraction of Treated cellulose

In this research two extraction processes were carried out namely cross and Bevan method [2] and new method [1].

1. New Method [1]

Coir fiber is chopped for 3-4 mm length and it is soaked for

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2% caustic soda (NaOH) in the fiber liquor ratio of 1:10 and kept for 6 hrs at a room temperature [1]. Then fiber is washed several times with distilled wasted to remove sticking of NaOH in fiber thus weight reduction was achieved [1]. Steam exploded treatment is done for mercerized fiber for 1 hrs around 200-250 degree Celsius[1] in pressure Cooker thus removal of lignin content and hemicelluloses content is observed weight loss is Obtained after steam explosion[1]. Then it is washed thoroughly with water, dried and weighted. During this process heat is applied around 200-250 degree Celsius [1]. Bleaching process is done for steam exploding Fiber using Naclo2 (Sodium chlorite) at ph. 2.3 [1]. The ph. of water is 7 is reduced to low ph at 2.3 value using Concentrated sulphuric acid (H2SO4) and by using sodium chlorite ph. is maintained at ph. 2.3. Heated for 1 hr. at 50- 60 degree [1]. Then it is washed with distilled water to reduce the effect of sodium chlorite [1]. Oxalic acid treatment is subjected to bleached fiber for mild acid treatment [1]. Oxalic acid 5% is taken for 800 grams of coir fiber and heated for 1 hrs at 50-60 degree followed by steam exploded treatment for 1 hrs at 200-250 degree [1] maintained in pressure cooker. Then fibers are then washed thoroughly with water and dried. Thus due to mild chemical treatment cellulose content of fiber may not decrease [1]. Finally after acid treatment the dried fiber is subject to constant stirring process on Remi motor at constant speed of 200 rpm for further cellulose.

Extracted treated cellulose from new method was shown in Figure 3.1.

Figure 3.1: Final Stage Extracted Treated Cellulose after Stirring Process

2. Cross and Bevan method [2]

In this step chopped coir fiber is first boiled with 1% of NaOH for 30 min. after that it is washed with water until moist chlorine gas is removed and it is dried, weight reduction is achieved [2]. Then after the fiber is treated with 2% of sodium sulphite and heated it for 5 min. Finally it is washed with water and then it is dried and weighted [2]. Fiber after sodium sulphite treatment is subjected to bleaching process by maintaining ph. at 4 thus ph. value is reduced using sulphuric acid and it heated with of potassium permanganate for 1 hrs. At 50-60 degree thus low molecular weight components partially get reduced [2]. Thus it is washed with water again and again. Finally it is dried and weighted [2]. The obtained treated cellulose from cross and Bevan method was shown in figure 3.2

Figure 3.2: Obtained Treated Cellulose from Cross and Bevan method

c) Composite sheet fabrication

The matrix used in this process is epoxy because it is able to improve adhesion between fiber and matrix and low cost. The epoxy matrix and epoxy hardener is mixed in ratio of 10: 1, in slow manner to avoid bubble formation in prolonged duration [10].

The coir fiber of 25% and 30%of both treated and untreated by weight was spread over mold cavity. The fiber is placed in mold impression and mixture of resin and hardener is also placed in mold impression [10]. The other half of mold plate is placed to complete whole setup. Force is applied at the rate of 2 ton per square inch which squeezes the fiber and resin mixture which is able to take mold impression shape and it is kept at constant pressure at 85-95 degree for 3-4 hrs [10]. And then it is cooled for 9-12 degree for 4-5 hrs, to avoid hardener effect in composite plate [10]. After the whole process is completed sheets are withdrawn from mold impression [10]. The fabricated composite sheets were shown in Figure 3.3 & 3.4.

Figure 3.3: Untreated Composite Sheets

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Figure 3.4: Treated Composite Sheets

d) Scanning Electron Microscopic (SEM)
Morphological analysis of samples at different weight percentage such as 25% and 30% in case of both treated and untreated composite were evaluated by scanning electron microscope Carl Zeiss EVO 18 at 20Kv. The samples are made for 1mm x 1mm x 3mm .for the prescribed size of sample morphology analysis were carried out for both treated and untreated composite.

e) Fourier Transform Infrared Spectroscopy (FT-IR)
Fourier transform infrared spectroscopy (FT-IR) of the samples was recorded by an IR-tracer 100 Schimadzu spectrophotometer. About 1mm x 1mm sample was placed into small particles of liquid nitrogen. The samples were mixed with kbr and pressed into small disc about 1mm thickness [1].

f) Element Detection Analysis
Energy-dispersive X-ray spectroscopy (EDS, EDX, or XEDS), sometimes called energy dispersive X- ray analysis (EDXA) or energy dispersive X-ray microanalysis (EDXMA), is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on an interaction of some source of excitation and a sample [14]. In this study element detection analysis were carried out for both treated and untreated sample at both 25% and 30% weight in the dimension of 1mm x 1mm x 3mm sample size. Element detection analysis was carried out in SEM along With EDX technique to determine chemical composition in which able to determine weight percentage of carbon and oxygen in samples [1].

3. Results and Discussion

A. Scanning Electron Microscopy (SEM)
The results showed in Fig 4.1 & 4.2 as untreated raw sample, and chemically treated fiber sample. It shows that rough surface and little porous in case of untreated raw sample at both 25% and 30% weight of fiber loading, and shows strong binding of cementing components lignin and hemicelluloses in untreated raw with average diameter of 100µm in case of 25% untreated and 30% untreated samples. In general alkali treatment at 2% remove lignin, and hemicelluloses content in fiber further steam explosion and bleaching process removes lignin, hemicelluloses, pectin, and tannin from inner part of fiber by Depolymerization and defibrillation [1], lignin oxidized by bleaching agent which allows to lignin degradation that leads to formation three functional groups such as hydroxyl, carbonyl, and carboxylic that access lignin to be soluble in alkali solution [1]. From the above study it demonstrates that in untreated raw sample it reveals rough surface, porous and shows impurities such as lignin and hemicelluloses at both 25% and 30% of weight in Fig 4.1. In case of treated composite it demonstrates removal of lignin and hemicelluloses due to alkali treatment followed by stream explosion, bleaching and oxalic acid treatment it allows to defibrillation and Depolymerization that reveals removal of lignin, hemicelluloses, wax and pectin that contained in fiber. Each elementary fiber shows a compact structure; exhibiting an alignment in the fiber axis direction with some non-fibrous components in the fiber surface [1]. It shows that chemically treated fiber shows removal of lignin and hemicelluloses with microfibrills average diameter around 20-200µm shown in Fig 4.2

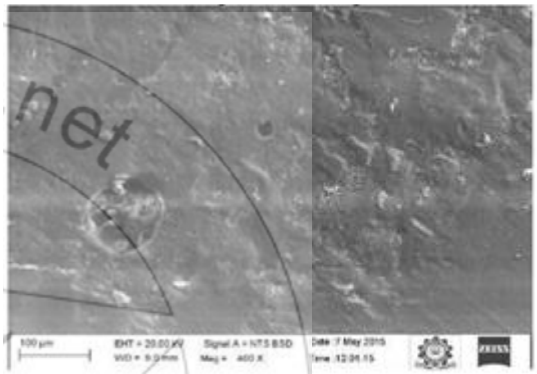

Figure 4.1: Untreated coir fiber SEM Image

Figure 4.2: Treated coir fiber SEM Image

B) Fourier Transform Infrared Spectroscopy (FT-IR)
The results shows that lignin and hemicelluloses content wave number was absent in case of both treated cellulose at 25% and 30% weight comparison to untreated composites. The composite material is composed of alkaline, esters, ketone, alcohol; aromatics with different oxygen functional group same as observed in literature [1]. An infrared red transmittance spectrum with some main observed peaks for different weight percentage in case of both treated and untreated was shown in table 4.1. All samples should be dry because it is difficult to extract due to cellulose water interaction [1].

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Table 4.1: Infrared Transmittance spectrum (%) for the different weight percentage in case of both treated and untreated composite

FT-IR (cm-1) Spectra (%T) [1]	O-H Stretch [1]	C-H Vibration [1]	C=O Stretch [1]	Absorbed water [1]
25% Untreated	3969.5	2731.2	1753.29	1608.63
30% Untreated	3975.2	1865.17	1755.22	1610.56
25% Treated	3980.9	2106.27	-	1608.46
30% Treated	3983.3	-	-	1600.34

From the above table 4.1 it shows that the characteristics peak wave number 1753.2 - 1755.22 cm-1 is responsible for hemicelluloses and lignin content and it was presented in both untreated composite at 25%and 30% weight. At chemical treated composite it was chiefly absent in both weight percentage. The wave number from 1600-1690 cm-1 is responsible for moisture absorption capacity. It shows that increasing to decreasing range from untreated to treated sample at different weight percentage in the range 1610.56-1600.34 cm-1.

Table 4.2: Infrared Transmittance spectrum (%) for the different weight percentage in case of both treated and untreated composite

C-C Stretching [1]	Aromatic ring Vibration of lignin [1]	C-H Stretching [1]	FT-IR (cm-1) Spectra (%T) [1]
1060.13	-	1365.60	25% Untreated
1053.10	1276.88	-	30% Untreated
1068.13	-	-	25% Treated
1072.42	-	-	30% Treated

From the above table 4.2 it shows that the characteristics peak wave number 1200-1390 cm-1 is responsible for hemicelluloses and lignin content and another possibility carboxyl adsorption [1], it was presented in both untreated composite at 25% weight and aromatic ring vibration was presents in 30% untreated. At chemical treated composite it was chiefly absent in both weight percentage. The wave number from 1000-1080 cm-1 is responsible for cellulose to cellulose interaction. It shows that decreasing range to increasing range from untreated to treated sample at different weight percentage in the range 1053.10 – 1072.42 cm-1. From FT-IR we can conclude that lignin and hemicelluloses binding components were removed in chemical treatment during NaOH treatment it breaks hydrogen bond, thus it reduces OH Concentration, further steam explosion, bleaching process it leads to formation of three function groups that access lignin and hemicelluloses to be soluble in chemical treatment, thus raw fiber have this characteristics peak 1700-1760 cm-1 and 1200-1390 cm-1 chiefly responsible for lignin and hemicelluloses. Above results were shown as graph with respect to wave number Vs Infrared transmittance (%), in Fig 4.3 & 4.4.

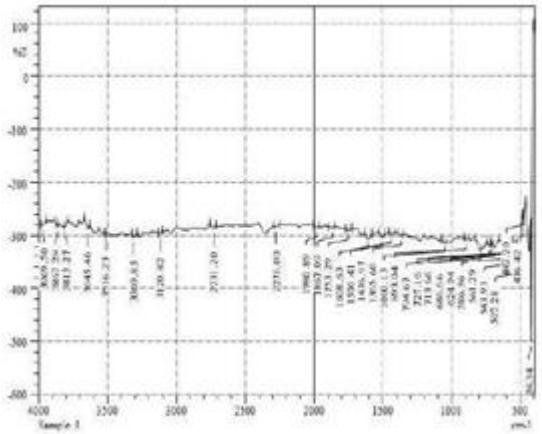

Figure 4.3: Graph Shows For Untreated 25% Weight Sample Chemical Composition With Respect To Infrared Transmittance (%)

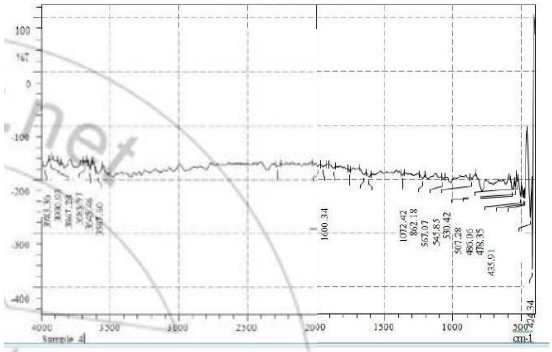

Figure 4.4: Graph Shows For Treated 30% Weight Sample Chemical Composition With Respect To Infrared Transmittance (%)

C) Element Detection Analysis

The results shows that at untreated sample at 25% and 30% weight of fiber loading it shows higher carbon content weight percentage than oxygen it clearly demonstrates due to higher lignin content, because lignin which was aromatic in nature with high carbon content comparison to cellulose and hemicelluloses. Thus during chemical treatment cellulose will be isolated from impurities which shows higher weight percentage of oxygen than carbon content. At 25% and 30% of fiber loading higher weight percentage of oxygen was observed at 30% of treated sample, but both 25% and 30% of treated sample demonstrates lignin content elimination and results higher oxygen percentage comparison to untreated samples at both 25% and 30% weight. The results of weight percentage of oxygen and carbon content was shown in Fig 4.5.

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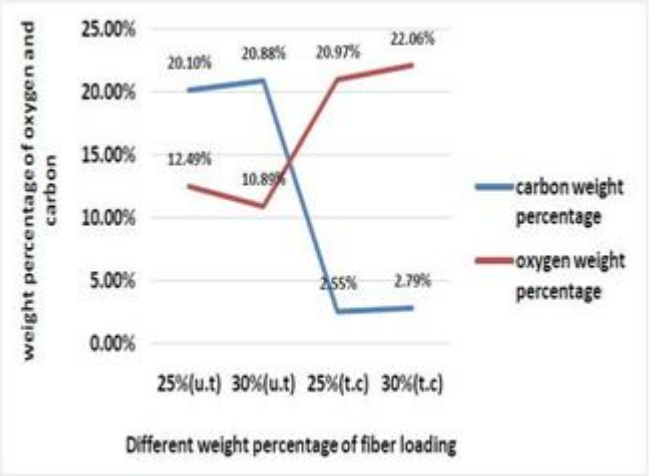

Figure 4.5: Weight Percentage of Carbon and Oxygen Vs Fiber Loading

4. Conclusion

The main theme of this research was to remove lignin and hemicellulose content of untreated coir fiber reinforced composites and it was accomplished by suitable chemical treatment namely new method thus it possess low amplitude level, and high damping factor in treated composites at both 25% and 30% of fiber loading. Microscopic Analysis Such as Scanning Electron Microscopic, Fourier Transform Infrared Spectroscopy, and Element Detection Analysis were Carried out for Treated and Untreated Composite ,it Shows Defibrillation And Depolymerization in Case of Treated Cellulose at 25%And 30% was recorded in SEM. The Absence of Lignin, Hemicelluloses, Pectin, and Some wax Content was observed in Treated Cellulose at 25% and 30% Comparison to Untreated. In Element Detection Analysis Weight Percentage of Oxygen is higher than Carbon at 30% of Treated Cellulose, Secondly at 25% of Treated Cellulose Comparison to Untreated Composites. So we conclude that removal of lignin and hemicelluloses was observed in Case of Treated Composite Comparison to Untreated Composite at 25% and 30% of weight. In future scope at 30% of treated composite, damping characteristics can be enhanced.

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