Effect of Chemical Treatment of Coir Dust on the Dielectric Behavior of Bio-waste Reinforced Polymer Composites

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ABSTRACT

The present work is focused on analyzing the effect of the chemical treatment of coir dust fibers on the dielectric properties of epoxy polymer matrix composites. Coir dust is a spongy, peat-like residue obtained from the processing of coconut husk for coir fiber. The coir dust was washed and sun dried. The dried coir dust was treated with chemicals such as HCl, CH₃COOH, KOH and NaOH. An untreated and treated surface of coir dust was characterized using FTIR. The dielectric parameters, viz., dielectric constant (κ), dielectric loss (tan δ) of untreated and treated composites were obtained in the frequency range of 100 Hz–1 MHz, and temperature range of 30°C–150°C. It is observed that dielectric loss and A. C. conductivity of the treated coir dust composite has decreased. The dielectric constant of the treated composite is lower than that of untreated composite, maybe because of reduction in interaction between polar–OH groups of coir dust fibers and water molecules.

Keywords: Chemical treatment, FTIR, natural fiber, orientation polarization, epoxy resin

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1. INTRODUCTION

Natural fibers such as rice husk, jute, sisal and pineapple leaf (PALF) have the potential to be used as a replacement for glass or other traditional reinforcement materials for making polymer composites. These fibers have many properties that make them an attractive alternative to traditional materials, which bear good mechanical, electrical, impact resistance, good thermal and acoustic properties [1–4]. In addition, they are available in large amounts, and are renewable and biodegradable. Other desirable properties include low cost, low density, less abrasion and less skin and respiratory irritation [5–7]. Natural fibers possess numerous advantages; unsatisfactory performance in natural fiber reinforcing polymer composites has limited the applications of natural fibers. The large amount of hydroxyl group exists in the cellulose chains, high lignin, low cellulose content and high level of moisture absorption which are the main factors which lead to debonding of natural fibers from the matrix [8–12]. To address this drawback, a number of fiber treatment methods have appeared in the literature [13–18]. Upon treatment, the hydrophilic nature of cellulose is significantly reduced, giving better fiber-matrix interfacial adhesion. Fourier transform infrared spectroscopy (FTIR) has been used to study the fiber/matrix bonding of treated and untreated natural fibers. The effect of alkali treatment on hemp fibers was studied by Ouajai et al. [19]. It was found that hemicellulose and pectin were removed by the treatment. FTIR was used to conform fiber/matrix bonding by Mohanty et al. [20]. Mwaikambo et al. used it to confirm that
hemicellulose was removed by the alkali treatment [21]. The chemical treatment of natural fibers affects the electrical properties along with mechanical properties. Naik et al. investigated the electrical properties of wood polymers composites based on agro waste and novolac [22]. Seena et al. analyzed the electrical properties of banana fiber reinforced phenol formaldehyde composites [23]. The study of the dielectrical properties of natural fiber reinforced polymer matrix composites indicates their suitability as insulating materials for special applications like suspension insulators, switch boards, bushing, studs and sleeves [24–26]. The present work is carried out to investigate the effect of the chemical treatment of coir dust fibers on the dielectrical properties of polymer composites.

2. MATERIALS AND METHODS

Coir dust is a spongy, peat-like residue obtained from the processing of coconut husk for coir fiber. The coir dust was washed in water and sun dried. Total Coir dust was divided into four equal weights and given the names untreated, Sample A (NaOH treated), Sample B (HCl treated) and Sample C (KOH treated).

The sample A was treated with 1.5M NaOH solution (500 mL) inside a beaker and this beaker was put in furnace at 60 °C for 12 h. The insoluble residue was treated with 5 wt% \( \text{H}_2\text{O}_2 \) (450) and 1.5M NaOH solution at 70 °C for 10 h and was finally treated with 2.5M NaOH solution (500 mL) at 75 °C for 3hr. After completion of the treatment, composite (with 20 wt% of coir dust) was prepared by hand lay-up method.

Sample B was treated with 1.5M NaOH solution (500 mL) inside a beaker and this beaker was put in a furnace at 60 °C for 12 hr. The insoluble residue was treated with 5 wt% \( \text{H}_2\text{O}_2 \) (450) and 1.5M NaOH solution at 70 °C for 10 h and was finally treated with 2M HCl solution (500 mL) at 60 °C for 3 h. After completion of the treatment, composite (with 20 wt% of coir dust) was prepared by hand lay-up method.

Sample C was treated with 2M KOH (450 mL) solution at 60 °C for 12 h. The insoluble residue was delignified with 2% NaCl solution (500 mL) at pH 3, and adjusted with 10 wt% acetic acid at 60 °C for 1 h. Another treatment with KOH solution (300 mL) under the same conditions as the first step was used. After completion of the treatment, composite (with 20 wt% of coir dust) was prepared by hand lay-up method.

The coir dust fibers were tested with FTIR spectrophotometer interfaced with IR microscope operated in reflectance mode. Dielectric measurements were carried out with a Solartron 1296 dielectric interface. The instrument allowed measurements over the temperature range from 30 °C to 150 °C and frequency interval from 100 Hz to 1 MHz. In dielectric analysis, the upper and lower
surfaces of the sample are coated with silver paint for the purpose of conductance; the sample is placed between the two Al parallel electrodes. A sinusoidal signal is applied, creating an alternating electric field. This electric field produces polarization in the sample, which oscillates at the same frequency as of the applied electric field, but has a phase angle shift $\delta$. This phase angle shift is measured by comparing the applied voltage to the measured current, which is separated into capacitive and conductive components. Measurements of capacitance and conductance are used to calculate dielectric constant ($K$) and dielectric loss ($\tan \delta$).

3. RESULTS AND DISCUSSION

3.1. FTIR Experimental Analysis of Treated and Untreated Coir Dust

The bonding nature between the fiber and matrix depends on the chemical properties of the fiber, atomic arrangement, and chemical constitution of polymeric matrix. However, cellulose is the principle coupling agent in the fiber/matrix bonding in natural fiber composite. On the other hand, to prevent good adhesion, lignin might act as an obstruction to the coupling agent diffusion.

Changes in the chemical nature of the lignocelluloses and hemicellulose are expected to occur upon chemical treatment. These possible modifications were studied by infrared spectroscopy, as presented in Figure 1. The material coir dust has a typical lignocellulosic composition, presenting bonds at 3354 cm$^{-1}$ assigned to O-H stretch, at 2938 cm$^{-1}$ assigned to C-H stretch from methyl and methylene groups [27]. The peak 1625 cm$^{-1}$ represents the C=O bonds on hemicellulose from carboxylate groups and at 1462 cm$^{-1}$ assigned to CH$_2$ symmetric bending peaks [28].

The sample treated with NaOH is not presenting the peaks 3354 cm$^{-1}$, 2938 cm$^{-1}$ and 1625 cm$^{-1}$. The removal of lignin and hemicellulose from the coir dust surface causes these peaks to disappear [29].

The peak 1620 cm$^{-1}$ present in KOH treated sample indicates the presence of hemicellulose content and the disappearance of peak 2938 cm$^{-1}$ and presence of 3354 cm$^{-1}$ indicates the partial removal of the lignin content. The peaks 3354 cm$^{-1}$ and 2938 cm$^{-1}$ are not present in the HCl treated sample. The removal of lignin from the coir dust surface causes these peaks to disappear. The peak at 1658 cm$^{-1}$ in case of HCl treated coir dust indicates the presence of hemicellulose content.
3.2. Effect of the Chemical Treatment on Dielectrical Properties

Dielectric constant of untreated and treated coir dust filled composite is shown in Figure 2. The untreated sample has the high dielectric constant of 8.75 followed by sample C (KOH treated) with a value of 5.457; sample B (HCl treated) was next with a value 4.88. Again, sample A (NaOH treated) has the least value of 4.77. Compared with the untreated sample, the treated samples are having lower dielectric constant. The main reason for this is decrement of orientation polarization due to less interaction between water molecules and polar–OH groups of coir dust. Due to this chemical treatment of coir dust, hydrophobic nature of coir dust increases, causing reduction of moisture absorption [30, 31].

![Figure 2](image2.png)

**Fig. 2:** Variation of Dielectric Constant of Treated and Untreated Coir Dust Filled Polymer Composites.
Variation of dielectric constant as a function of frequency for untreated and treated composite samples is shown in Figure 3. The NaOH treated sample having low dielectric constant compares to the untreated and other treated samples.

![Figure 3](image)

**Fig. 3:** Frequency Dependence of Dielectric Constant for Treated and Untreated Composites.

Figure 4 presents the dielectric constant of treated coir dust composites as a function of frequency at different testing temperatures. From the figure, it is observed that the nature of the plots for all NaOH, KOH and HCl treated samples is similar. It is evident from these figures that dielectric constant of the composites is decreased with increasing frequency, at any constant temperature. It is also observed that low frequency and high temperature have more prominent effect on the dielectric constant. Decreasing nature of the K value with increasing frequency is due to the dielectric relaxation, which is the cause of anomalous dispersion. At higher frequencies, the orientation of polar molecules along the direction of the applied electric field is disturbed.

![Figure 4](image)

(a) (b)
Figure 5 presents the variation of the dielectric constant with temperature for treated and untreated coir dust composites and Figure 6 represents the temperature dependence of the K-value at different frequencies for treated samples. The value of K is found to increase with temperature at fixed frequency for all treated and untreated samples. This is due to the greater freedom of movement of dipole molecular chains. With increasing temperature, the polarization increases and hence the dielectric constant also increases.

**Fig. 4: Frequency Dependence of K-Value at Different Temperatures for (a) NaOH, (b) KOH, and (c) HCl Treated Composites.**

**Fig. 5: Temperature Dependence of Dielectric Constant for Treated and Untreated Composites.**
Fig. 6: Temperature Dependence of k-Value at Different Frequencies for (a) NaOH, (b) KOH, and (c) HCl Treated Composites.

The results of the dielectric loss of treated and untreated coir dust filled composites are presented in Figure 7. The treated sample A (NaOH) has the lowest dielectric loss 0.0115 followed by sample B (HCl) with a value of 0.0405. Sample C (KOH) was the next value 0.0413. Untreated Sample has the high value of 0.251. The dielectric loss of the treated samples was decreased; this is due to the decrement in the A. C. conductivity of the composite.
Fig. 7: Variation of the Dielectric Loss of Treated and Untreated Coir Dust Filled Polymer Composites.

Figure 8 presents the frequency dependence of dielectric loss for treated and untreated samples and Figure 9 represents the dielectric loss of treated samples as a function of frequency at different temperatures. The value of dielectric loss decreases with increase of frequency at constant temperature in all treated and untreated samples. The decrement in the loss factor for high temperature and at low frequency is more prominent which is the usual behavior of most materials.
Variation of the dielectric loss with temperature for treated and untreated composites is shown in Figures 10 and 11, indicating the temperature dependence of the loss factor at different frequencies. The nature of the plots for treated and untreated samples is the same. The dielectric loss increases sharply with increasing temperature at lower frequencies whereas at higher frequencies the increment in the dielectric loss is negligible.

Fig. 9: Frequency Dependence of Dielectric Loss at Different Temperatures for (a) NaOH, (b) KOH, and (c) HCl Treated Composites.
**Fig. 10:** Temperature Dependence of Dielectric Loss for Treated and Untreated Composites.

**Fig. 11:** Temperature Dependence of Dielectric Loss at Different Frequencies for (a) NaOH, (b) KOH, and (c) HCl Treated Composites.
Figure 12 shows the frequency dependence of A. C. conductivity and the A. C. resistivity for untreated and treated coir dust composites. The treated samples have lower conductivity than the untreated sample. This is because chemical treatment reduces the moisture absorption nature at the fiber/matrix interface of composites. It is evident from the figure that the conductivity of the material is increased with increasing frequency at room temperature. It is also observed that at middle of the frequency range the change in the A. C conductivity of NaOH treated sample is high. The resistivity of the material is decreased with increasing frequency at room temperature.

![Graph](image)

(a) ![Graph](image)

(b)

**Fig. 12:** Frequency Dependence of (a) Conductivity, and (b) Resistivity.

4. CONCLUSIONS

The effect of chemical treatment of coir dust fibers on the dielectrical properties (dielectric constant $K$, dielectric loss $\tan \delta$) of epoxy polymer matrix composites has been studied in the frequency range of 100 Hz–1 MHz, and temperature range of 30 °C –150 °C. The experimental result indicates that the dielectrical loss ($\tan \delta$) and A. C. conductivity decreased with the chemical treatment due to reducing the moisture absorption nature at the fiber/matrix interface. Dielectric constant of treated coir dust composite was lower than that of the untreated coir dust composite because of reduction in interaction between polar–OH groups of coir dust fibers and water molecules. This decrement of $K$ and $\tan \delta$ increases the dielectric strength of the composites and the increased dielectric strength makes these composites quite suitable for use as insulators. Samples treated with NaOH gave the low dielectric loss and low dielectric constant properties followed by the samples treated with HCl and KOH. The decrease in $K$ with increase of frequency is due to the orientation polarization and increase in $K$ with increase in test temperature is due to greater freedom of movement of dipole molecular chain.
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REFERENCES


