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DIELECTRIC BEHAVIOR OF BIO-WASTE REINFORCED POLYMER COMPOSITES

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Abstract

A dielectric is an electrical insulator that may be polarized by the action of an applied electric field. When a dielectric is placed in an electric field, electric charges do not flow through the material, as in a conductor, but only slightly shift from their average equilibrium positions causing dielectric polarization. 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. A novel low-cost polymer composite using coir dust reinforcement is prepared. Investigation of dielectric behavior of this polymer composite proves its efficiency as a high-value marketable product. As the composite is made using bio-materials from local resources, its cost is less as compared to other polymer composites available today.

Keywords:- Dielectric, Bio-waste, electric field etc.

1. INTRODUCTION

A dielectric is an electrical insulator that may be polarized by the action of an applied electric field. When a dielectric is placed in an electric field, electric charges do not flow through the material, as in a conductor, but only slightly shift from their average equilibrium positions causing dielectric polarization. Because of dielectric polarization, positive charges are displaced along the field and negative charges shift in the opposite direction. This creates an internal electric field which partly compensates the external field inside the dielectric **[1,2]** if a dielectric is composed of weakly bonded molecules, those molecules not only become polarized, but also reorient so that their symmetry axis aligns to the field**.**

While the term "insulator" refers to a low degree of electrical conduction, the term "dielectric" is typically used to describe materials with a high polarizability. The latter is expressed by a number called the dielectric constant. A common, yet notable, example of a dielectric is the electrically insulating material between the metallic plates of a capacitor. The polarization of the dielectric by the applied electric field increases the capacitor's capacitance **[3].**
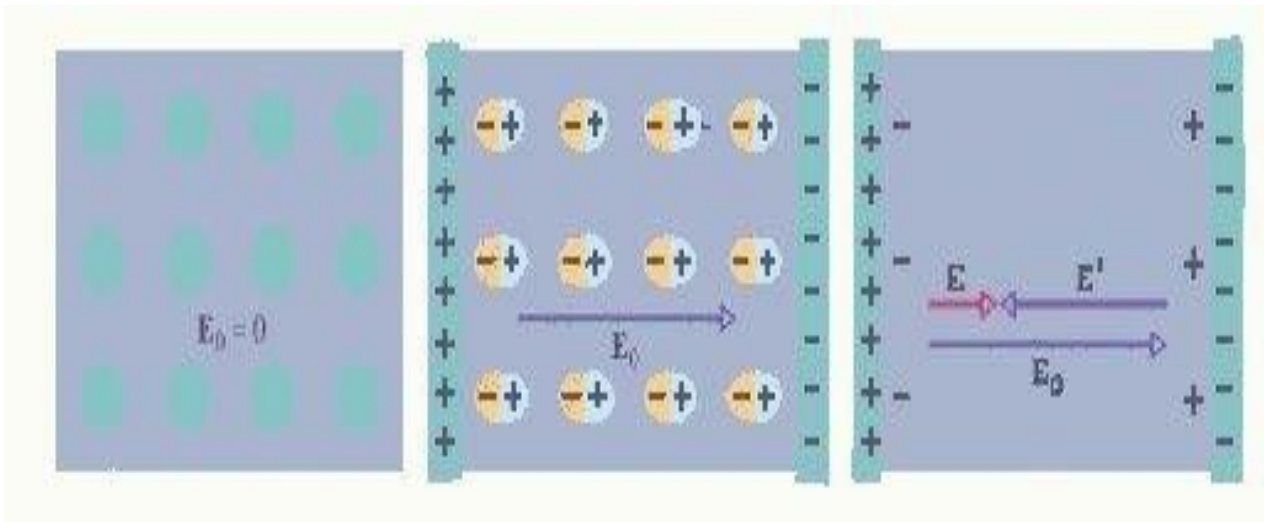

Figure 1: Polarization of the dielectric material between the capacitor plates.

The study of dielectric properties is concerned with the storage and dissipation of electric and magnetic energy in materials **[4]**. It is important to explain various phenomena in electronics, optics, and solid-state physics.

1.1 Dielectric Mechanisms

There are a number of different dielectric mechanisms, connected to the way a studied medium reacts to the applied field (see the figure illustration). Each dielectric mechanism is centered around its characteristic frequency, which is the reciprocal of the characteristic time of the process. In general, dielectric mechanisms can be divided into relaxation and resonance processes. The most common, starting from high frequencies, are:

1.2 Electronic polarization

This resonant process occurs in a neutral atom when the electric field displaces the electron density relative to the nucleus it surrounds. This displacement occurs due to the equilibrium between restoration and electric forces. Electronic polarization may be understood by assuming an atom as a point nucleus surrounded by spherical electron cloud of uniform charge density.

1.3 Atomic polarization

Atomic polarization is observed when the electronic cloud is deformed under the force of the applied field, so that the negative and positive charge is formed. This is a resonant process.

1.4 Dipole relaxation

This originates from permanent and induced dipoles aligning to an electric field. Their orientation polarization is disturbed by thermal noise (which miss-aligns the dipole vectors from the direction of the field), and the time needed for dipoles to relax is determined by the local viscosity. These two facts make dipole relaxation heavily dependent on temperature and chemical surrounding.

1.5. Ionic relaxation

Ionic relaxation comprises ionic conductivity and interfacial and space charge relaxation. Ionic conductivity predominates at low frequencies and introduces only losses to the system. Interfacial relaxation occurs when charge carriers are trapped at interfaces of heterogeneous systems. A related effect is Maxwell-Wagner-Sillars polarization, where charge carriers blocked at inner dielectric boundary layers (on the macroscopic scale) or external electrodes (on a macroscopic scale) lead to a separation of charges. The charges may be separated by a considerable distance and therefore make contributions to the dielectric loss that are orders of magnitude larger than the response due to molecular fluctuations[2].

1.6 Dielectric relaxation

Dielectric relaxation as a whole is the result of the movement of dipoles (dipole relaxation) and electric charges (ionic relaxation) due to an applied alternating field, and is usually observed in the frequency range 10^2-10^{10} Hz. Relaxation mechanisms are relatively slow compared to resonant electronic transitions or molecular vibrations, which usually have frequencies above 10^{12} Hz.

2. BACKGROUND

2.1 Dielectric nature of the polymer composites

It is well known that composites can be produced exhibiting enhanced properties that the constituent materials may not exhibit [5-8]. For instance, from the combination of different fibers or fillers with polymer matrices one can produce polymer-matrix composites, a material important to the electronic industry for its dielectric properties in the use of capacitors [9-11]. The effective utilization of filled polymers depends strongly on the ability to disperse the fillers homogeneously throughout the matrix [12]. The interface properties also strongly affect the characteristics and performance of these composites [13]. One of the most attractive features of these filled composites is that their dielectric properties can be widely changed by choice of shape, size, and the conductivity of filled constituents in the polymeric matrix.

Most of the interesting properties of polymers are attributable to the complex motions within their molecular matrix. In the polymeric system, molecular relaxations exhibit various transitions [14]. As very little work has been reported on double-layer systems, the intention in the present work is to study dielectric properties of such a system. The polymeric interfaces act as charge-carrier trapping sites [15]. Therefore, it has become essential to study the effect of interfaces on the charge-carrier generation, transport and storage in polymeric systems. The study of dielectric constant and dielectric loss, as a function of temperature and frequency is one of the most convenient and sensitive methods of studying polymeric structure.

For polymer composites in the solid or viscoelastic state, the physical structure is of great importance in determining the dielectric behavior [16]. The dielectric properties of polymer composite materials have been studied with a view to modifying the properties of polymer systems for practical applications. The conventional inorganic insulators and dielectrics have to a large extent been replaced by polymers on account of their unique ability to be tailor made for specific needs. Epoxies and polyesters have been used in electronics as insulators, dielectrics, substrates, potting compounds, embedding materials and conformal coatings [17].

2.2 Chemical treatment of natural fibers

One of the major problems associated with the use of natural fibers in composites is their high moisture sensitivity leading to severe reduction of mechanical properties and delaminating. The reduction in mechanical properties may be due to poor interfacial bonding between resin matrices and fibers. It is therefore necessary to modify the fiber surface to render it more hydrophobic and also more compatible with resin matrices. An effective method of chemical modification of natural fibers is graft copolymerization. The resulting co- polymer displays the characteristic properties of both fibrous cellulose and grafted polymer [18, 19]. One of the most explored chemical modifications is the acetylation-esterifi cation of cellulose-OH, by reaction with acetic anhydride. This reaction reduces hydrophilicity and swelling of lignocelluloses and their composites [20].

The effect of chemical treatment of natural fibers with sodium alginate and sodium hydroxide has also been reported for coir, banana and sisal fibers by Mani *et al.* [21]. This modification results in an increase in adhesive bonding and thus improves ultimate tensile strength up to 30%. Mitra *et al.* [22] have reported that treatment of jute with polycondensates such as phenol-formaldehyde, melamine-formaldehyde and cashew nut shell with liquid formaldehyde improves the wet ability of jute fibers and reduces water regain properties. Samal and Ray [23] have studied the chemical modification of pineapple leaf fibers using alkali treatment, diazo coupling with aniline and cross-linking with formaldehyde. These chemical treatments result in significant

improvements in mechanical properties, chemical resistance and reduced moisture regain. Finally, Joseph *et al*. [24] have investigated the influence of chemical treatment with sodium hydroxide, isocyanate and peroxide on the properties of sisal/polyethylene composites. The observed enhancement in properties of the composites and attributed this to the strong bonding between sisal and polyethylene matrix.

In an effort to improve the mechanical properties of recycled HDPE/wood fiber composites, Selke *et al.* [25] investigated the use of several additives with possible effect on the fiber/matrix adhesion or fiber dispersion into the matrix. They found that maleic anhydride modified polypropylene appears especially promising, since its use at a concentration of 5% in composites with 30% wood fiber results in an increase in tensile strength and elongation at break. Similar results have been obtained by Dalvag *et al*. [26], who have reported that the composite's elastic modulus remains unchanged. Zadorecki and Flodin [27] have found that some coupling agents, namely trichloro-striazine and di-methylol melamine can produce covalent bonds between cellulosic materials and polymer matrices, leading to modified performance and reduced sensitivity to water. This approach has been further explored by Maldas and Kokta [28], who used ophthalmic anhydride as coupling agent for wood fiber/polystyrene composites. In addition to the chemical affinity of the benzene rings of ophthalmic anhydride with those of polystyrene, the anhydride group can directly attack the – OH group of cellulose. Furthermore, Razi *et al.* [29] found that the treatment of wood with sodium hydroxide followed by drying with vinyltrimethoxysilance is superior, for obtaining maximum bonding strength at the wood/polymer interface that yields improved mechanical properties.

3. COMPOSITE PROCESSING AND CHARACTERISATION

3.1 Matrix material

Epoxy LY 556, chemically belonging to the epoxide family is used as the matrix material. Its common name is Bisphenol A Diglycidyl Ether. The hardener with IUPAC name NN0-bis (2-aminoethylethane-1, 2-diamin) used with the epoxy has the designation HY-951. The epoxy resin and the hardener were supplied by Ciba Geigy India Ltd. Resin and hardeners are mixed in a ratio of 10:1 by weight as recommended. Density of the epoxy resin system is 1.28 g/cc.

3.2 Chemical treatment

Coir dust is the spongy, peat like residues get this from the processing of coconut husks (mesocarp) for coir fiber .The coir dust was washed and sun dried. The dried coir dust was treated with chemicals such as HCl, CH_3COOH, KOH and NaOH. Total Coir dust was divided in to three equal weights and given the names Sample A, Sample B and Sample C.

The sample A was treated with 1.5M NaOH solution (500ml) inside a beaker and this beaker was put in furnace at 60^0C for 12 hours. The insoluble residue was treated with 5 wt% H_2O_2 (450) and 1.5M NaOH solution at 70^0C for 10 hours and was finally treated with 2.5M NaOH solution (500ml) at 75^0C for 3 hours. After completion of the treatment, composite (with 20 wt% of coir dust) has prepared by hand lay-up method.

The sample B was treated with 1.5M NaOH solution (500ml) inside a beaker and this beaker was put in furnace at 60^0C for 12 hours. The insoluble residue was treated with 5 wt% H_2O_2 (450) and 1.5M NaOH solution at 70^0C for 10 hours and was finally treated with 2M HCl solution (500ml) at 60^0C for 3 hours. After completion of the treatment, composite (with 20 wt% of coir dust) is prepared by hand lay-up method.

The sample C was treated with the 2M KOH (450ml) solution at 60^0C for 12 hours. The insoluble residue was delignified with 2% NaCl solution (500ml) at pH 3, and adjusted with 10 wt% acetic acid at 60^0C for 1 hour. Another treatment with KOH solution (300ml) under the same conditions as the first step was used. After completion of the treatment, composite (with 20 wt% of coir dust) has prepared by hand lay-up method.

3.3 Composite fabrication

Coir dust was mixed with the epoxy resin by stirring at room temperature and poured in moulds of dimensions (300 $\times$ 300 $\times$ 5) mm. Five samples i.e. sample A(pure epoxy),sample B(epoxy+10 wt% of coir dust),sample C(epoxy+20 wt.% of coir dust),sample D(epoxy+40wt% of coir dust),and sample E(epoxy+60 wt.% of coir dust) are prepared . Test specimens of suitable dimensions are cut from the composite sheets for erosion wear test. For abrasive wear test, composite pins of length30 mm and diameter of 10 mm are prepared by using cylindrical moulds.

Designation	Composition
ECD1 (Sample A)	Pure Epoxy
ECD2 (Sample B)	Epoxy + 10 wt. % Coir Dust
ECD3 (Sample C)	Epoxy +20 wt. % Coir Dust
ECD4 (Sample D)	Epoxy +40 wt. % Coir Dust

ECD5 (Sample E)	Epoxy + 60 wt. % Coir Dust

Table-1. Different wt. % percentages of the coir dust in epoxy resin.

3.4 FTIR Spectroscopy

Fourier Transform Infrared (FTIR) spectroscopy is an important analysis technique which detects various characteristic functional groups in molecules of any matter [**30**]. On interaction of an infrared light with the matter, chemical bonds will stretch, contract and bend and as a result, each chemical functional group tends to absorb infrared radiation in a specific wavelength range regardless of the structure of the rest of the molecule. Based on this principle, functional groups present in composite materials are identified. It is performed in a FTIR spectrophotometer interfaced with IR microscope operated in reflectance mode. The microscope is equipped with a video camera, a liquid nitrogen cooled mercury cadmium telluride (MCT) detector and a computer controlled translation stage, programmable in the x and y directions. The spectra are collected in the 400 cm^{-1} to 4000 cm^{-1} regions with 8 cm^{-1} resolution, 60 scans and beam spot size of 10µm-100µm. The FTIR imaging in the present investigation is carried out using a Perkin Elmer Spectrum RX (1).

3.5 Dielectric studies

Dielectric measurements were carried out with a solartron 1296 Dielectric Interface instrument allowing measurements over the temperature range from 30^0 C to 150^0C and frequency interval from 100Hz to 1 MHz In dielectric analysis, the upper and lower surface of the sample is 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 produce polarization in the sample, which oscillates at the same frequency as of the applied electric field, but has a phase angle shift δ .This phase angle shift is measured by comparing the applied voltage to the measured current , which is separated in to capacitive and conductive components **[31, 32]** .
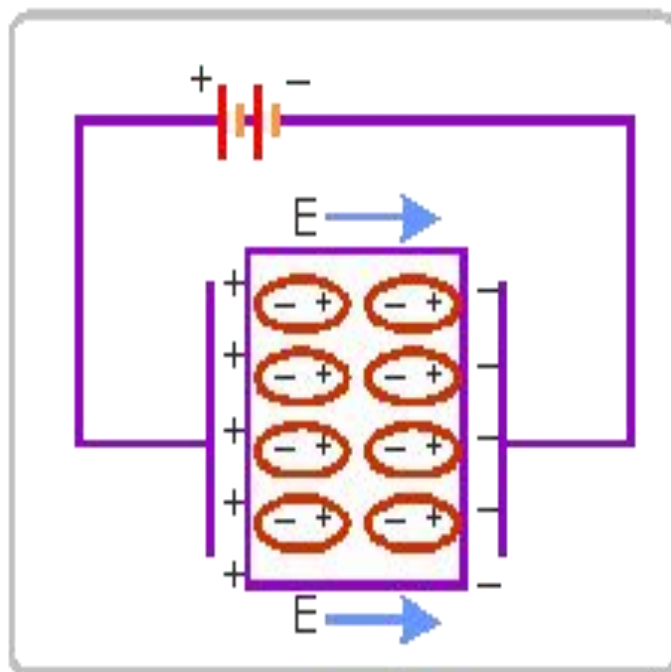

Figure 2: Schematic diagram of the applied electric field for a composite.

The dielectric constant, dielectric loss, conductivity and resistivity are determined as follows.

Dielectric constant K = C$^{'}$ /C.

Where C' (pF) is measured capacitance and C (pF) is calculated using the formulae, C = ε_0. (A/d);

A (m.m²) - area of the electrode, d (m. m)-thickness of the sample.

The dielectric loss is given by, tan δ =G(S)/ (w C' (F)),

w=2πf, f-is the measuring frequency. And   ,G=G$_0$(R-R$_0$) **[33]**

4. RESULTS AND DISCUSSION

4.1 FTIR Analysis

The content of the coir dust including moisture, cellulose lignin, ash, and extractives. The content of lignin (35.5%) is relatively high compared to the other natural fibers, while the cellulose content (36%) is higher compared to the other fiber species e.g jute (16-22%), hardwood (19-26%) . Figure 3, presents the FTIR experimental analysis of the pure coir dust and composites with different wt.% of coir dust. 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 **[34]**.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. The peaks for pure epoxy presenting at 1046 cm-1 assigned to C-O-C bending in benzo-ether, at 2922 cm-1 represents the C-H stretching in methyl group, at 3430-3450 cm-1 represents O-H stretching in free alcohol. The peak range about 1000cm-1 to 1200cm-1 become more wide and short in case of 10wt% of coir dust composite as compared to corresponding peaks in pure epoxy resin, due to the formation of hydrogen bonding hydrogen atom of the methyle groups in coir dust and oxygen atom of the epoxy resin.
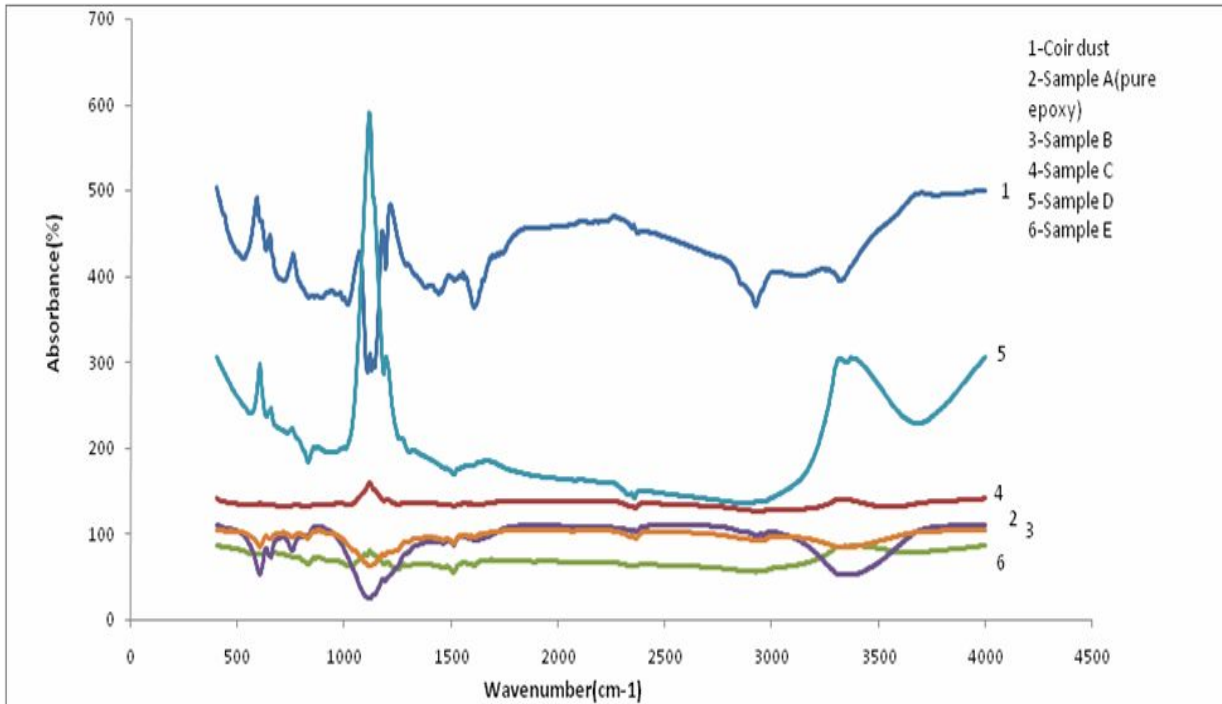
Figure 3: FTIR experimental analysis of pure coir dust and composites with different wt. %
of coir dust.

4.2 Dielectric properties

Figure 4, gives the variation of dielectric constant and tangent loss with frequency for different weight percentage of coir dust reinforcement at room temperature. From the figure it is clear that the K and εǁ are increases with increasing coir dust content up to certain limit(i.e. up to 40 wt.%) due to the absorption of moisture at the fiber resin interface, as the dielectric of water is very high, with further increase in reinforce amount both K and εǁ are decreases. It also be noted that with increasing the frequency the dielectric constant was decreased, it is the expected behavior in most of the materials**[35]**. As frequency increases more and more of the bound charges will start to oscillate out of phase with the applied voltage and will contribute to A.C conductivity causing k decreases, and also observed that with increasing frequency dielectric loss decreased.
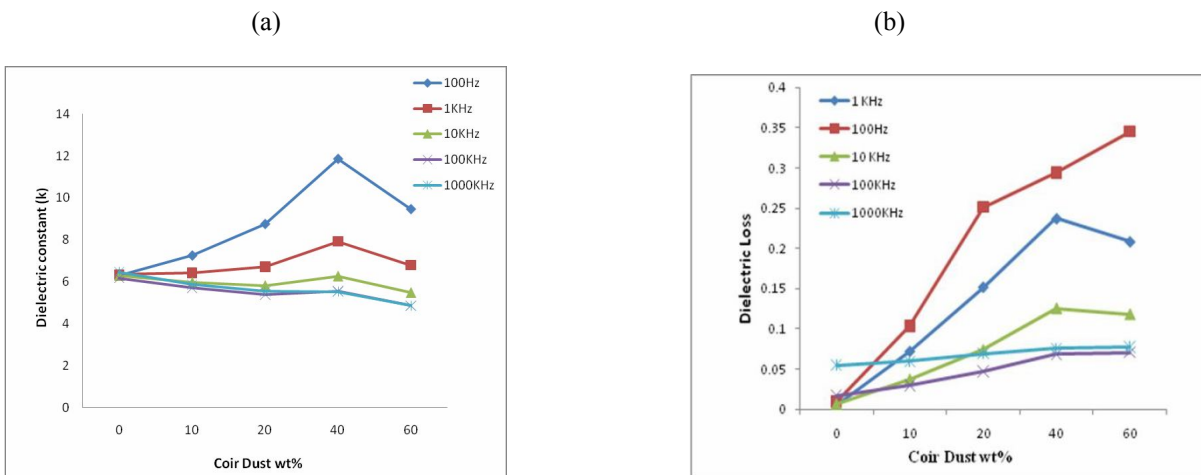
(a)   (b)

Figure 4. Frequency dependence of (a) Dielectric constant, (b) Dielectric loss for different wt.% of coir dust in epoxy
resin at room temperature.

Figure 5, shows the variation of dielectric constant (K) with frequency at different temperatures for samples A, B, C, D and E, respectively. From the figures it is clear that dielectric constant decreased with increasing frequency at constant temperature, and increased with increasing temperature at constant frequency ,low frequency and high temperature have more prominent 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 **[36]**.
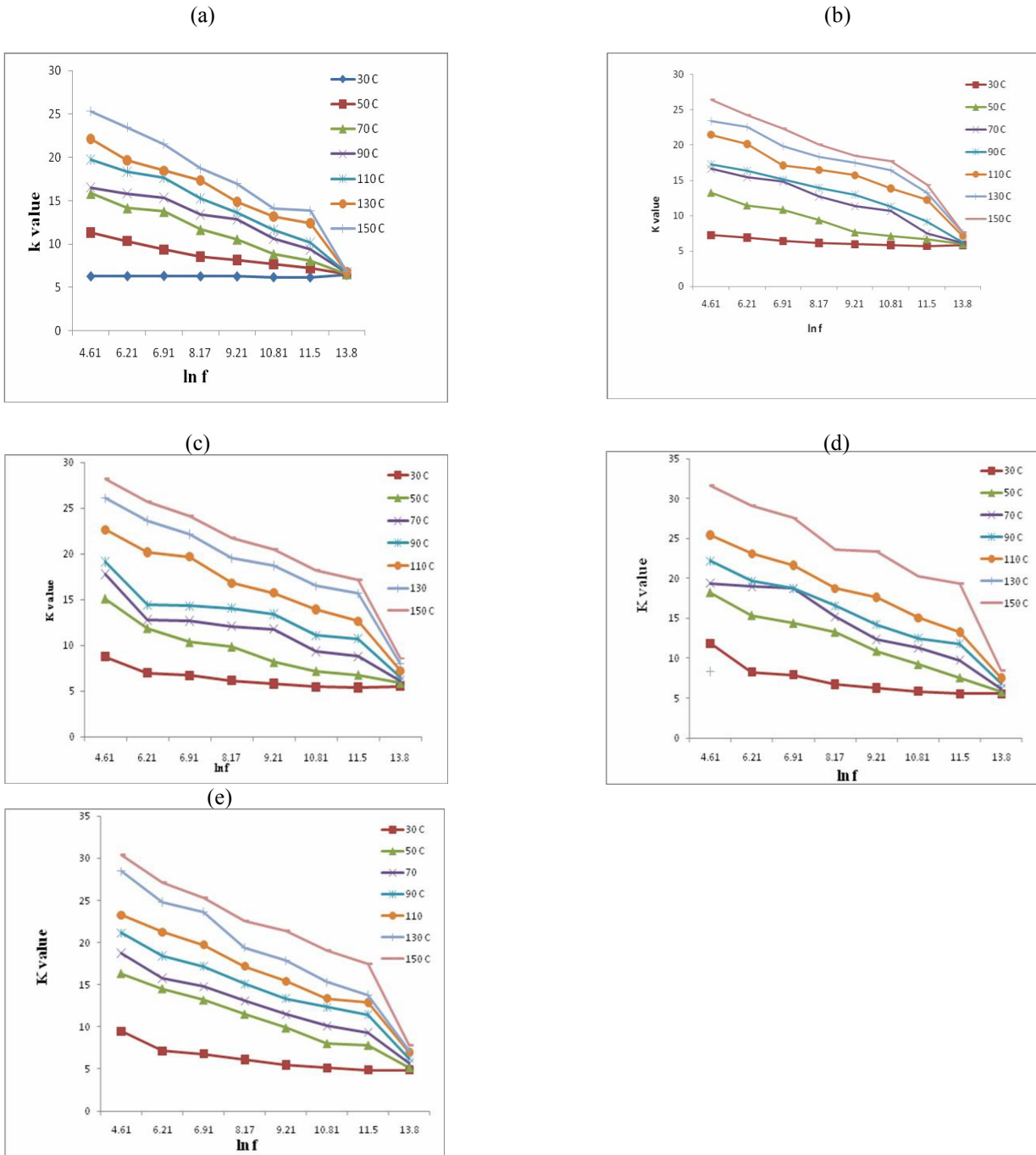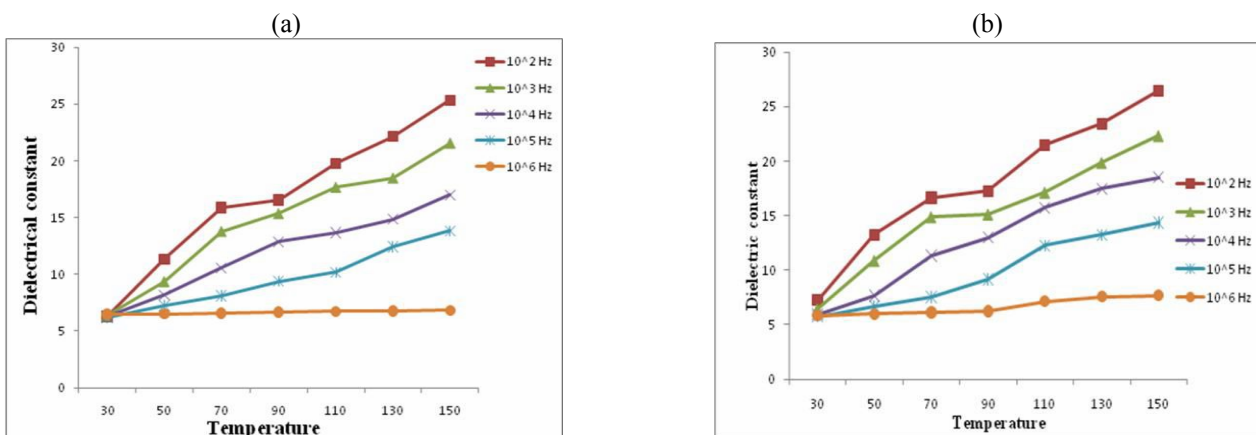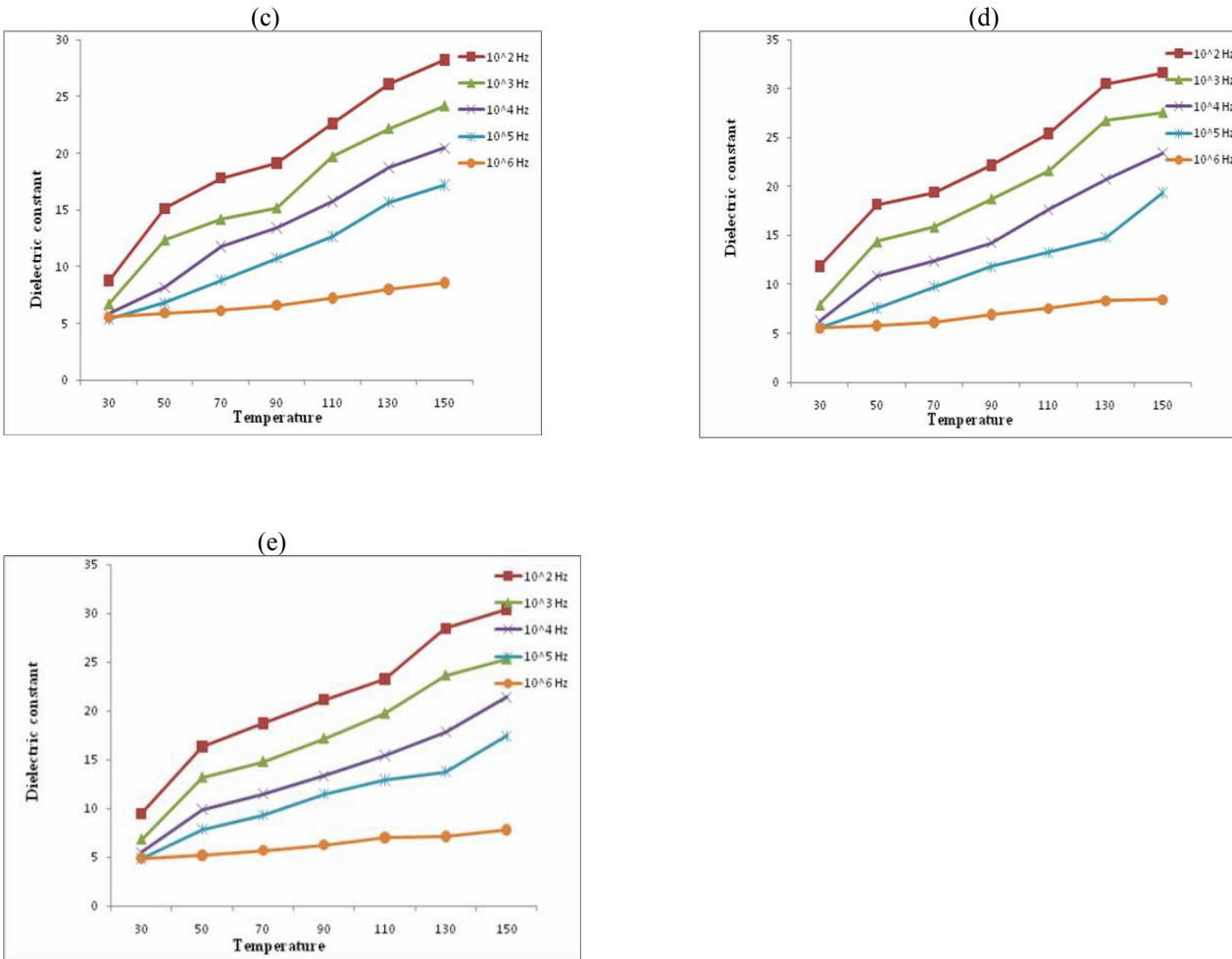
(a)

(b)

(c)

(d)

(e)

Figure 5. Frequency dependence of K-value at different temperatures for; (a) sample A, (b)sample B, (c) sample C, (d)sample D , and (e) sample E.

Figure 6 (a-e), shows the variation of dielectric constant with temperature at different frequencies for samples A, B, C, D, and E, respectively. From the figure it is clear that the value of K is increased with increasing temperature at constant frequency. The increase in K value with increasing temperature is due to the greater freedom of movement of dipole molecular chains. With increasing temperature the polarization increases and hence the dielectric constant is also increased **[37]**.
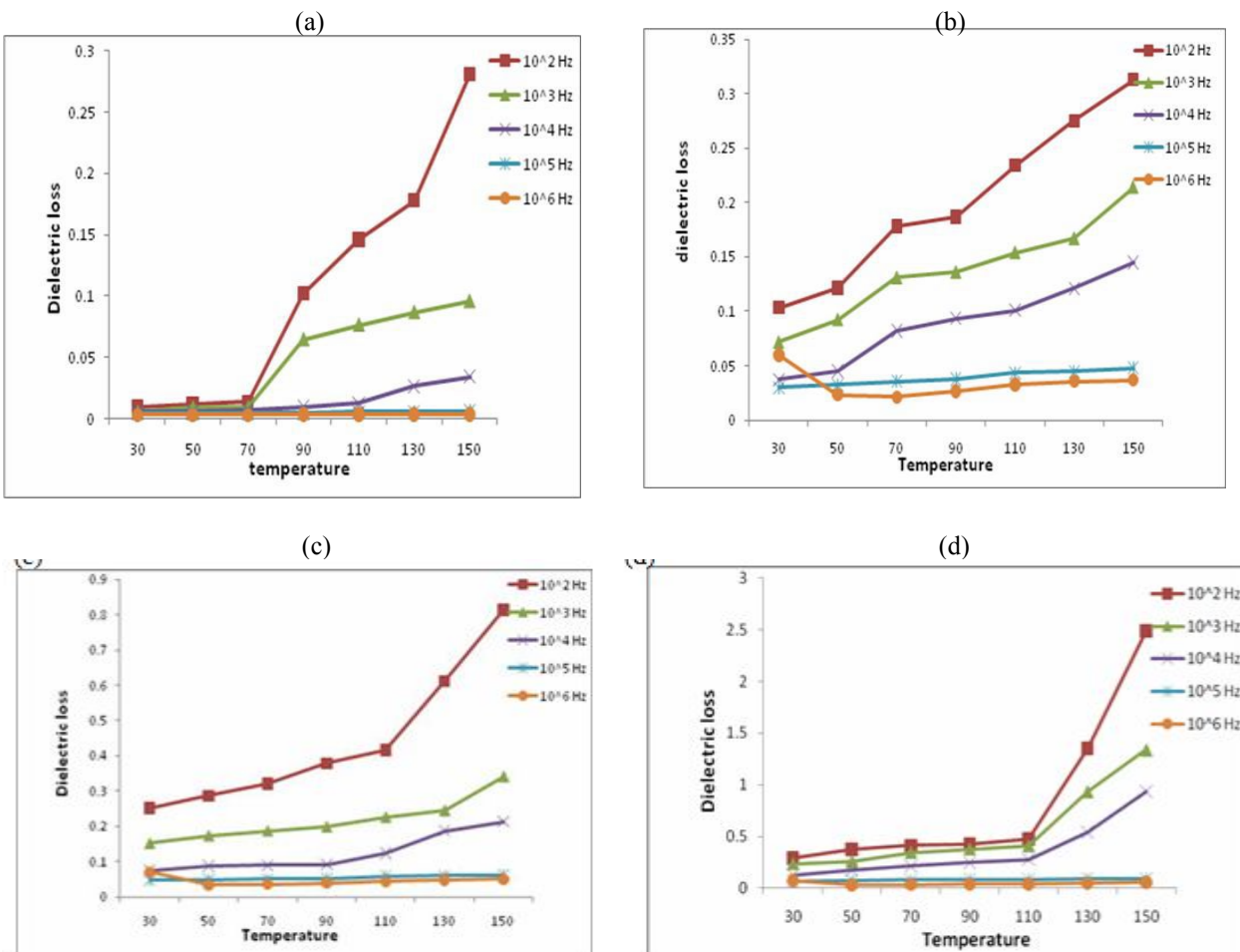
(a)

(b)

Figure 6. Temperature dependence of K-value at different frequencies for; (a) sample A,(b)sample B, (c)sample C,(d)sample D, and (e) sample E.

Figure 7(a-e), shows the variation of the dielectric loss with temperature at different frequencies for samples A, B, C, D, and E respectively. From the figure it is clear that dielectric loss is highly increases with increasing temperature at lower frequencies ,at higher frequencies the increment in the dielectric loss is negligible.
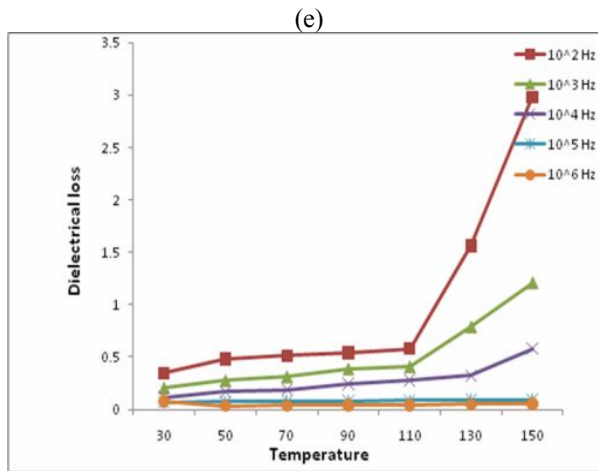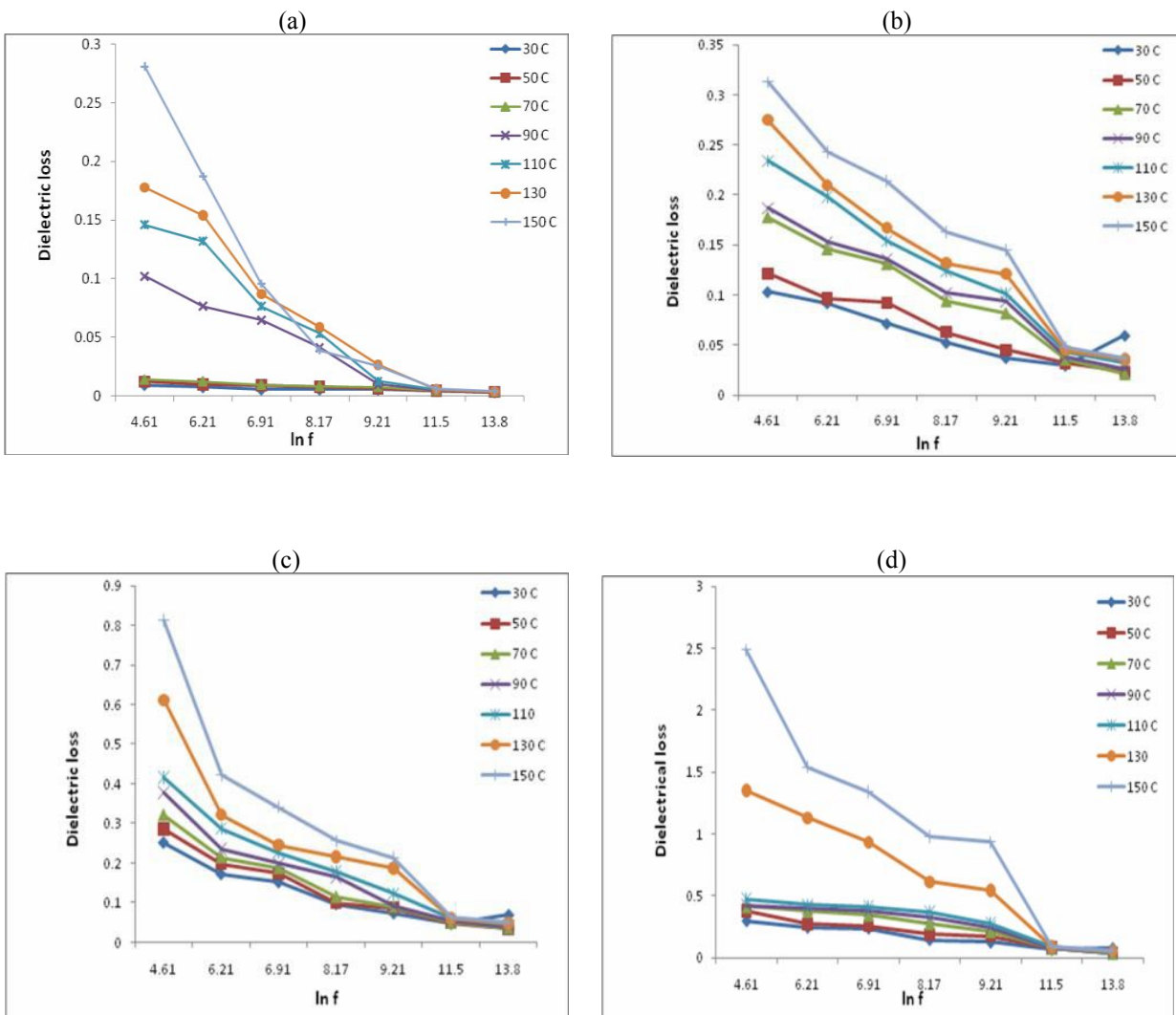
Figure 7. Temperature dependence of loss factor at different frequencies for (a) sample A, (b)sample B, (c)sample C,(d)sample D, and (e)sample E.

Figure 8(a-e) shows the variation of dielectric loss with frequency at different temperatures for samples A,B,C,D,and E, respectively. From the figure it is clear that the loss factor decreased with increasing frequency at constant temperature, the decrement in the loss factor for high temperature at low frequency is more prominent, it is usual behavior of most materials[38].
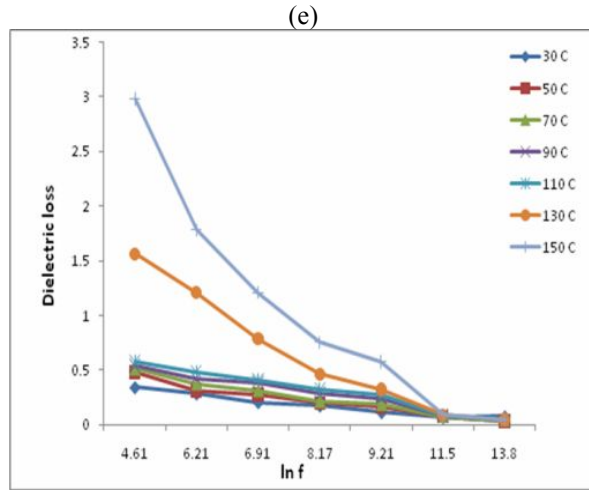
(e)

Figure 8. Frequency dependence of loss factor at different temperatures for(a) sample A, (b)sample B, (c)sample C,(d) sample D, and (e) sample E.

4.3 Electrical conductivity and resistivity

Figure 9(a-b) shows the variation of conductivity and resistivity with frequency at different wt. % percentage of coir dust. From the figure it is clear that the conductivity of the material is increased with increasing frequency at room temperature, it is also observed that at 40 wt. % coir dust the A.C conductivity is more pronounced. The resistivity of the material is decreased with increasing frequency at room temperature, the decrement of A.C resistivity at 40 wt. % coir dust is more pronounced.
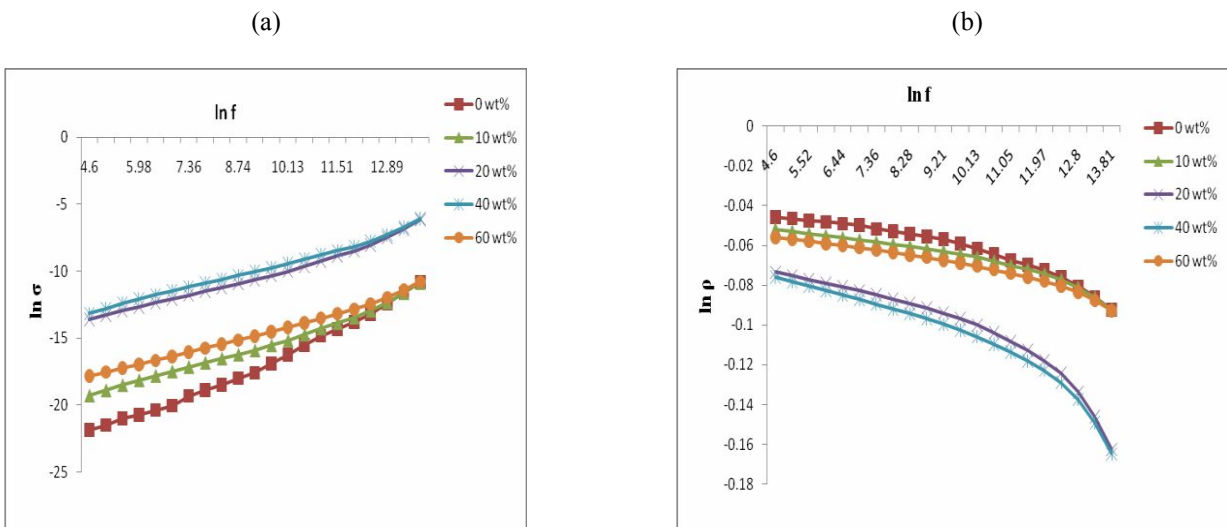
(a) (b)

Figure 9. Frequency dependence of (a) conductivity and (b) resistivity.

Figure 10, shows the variation of the A.C conductance with frequency and temperature for different samples. With increasing the frequency and temperature the conductance of the material increases due to the oscillation of the dipolar molecules in out of phase with the applied field and random movement of the dipolar molecules.

Figure 11, gives the variation of the A.C Resistance with frequency of applied electric field and testing temperature for different samples. It is observed that with increasing frequency and temperature the A.C resistance decreases due to increment in the conductivity.
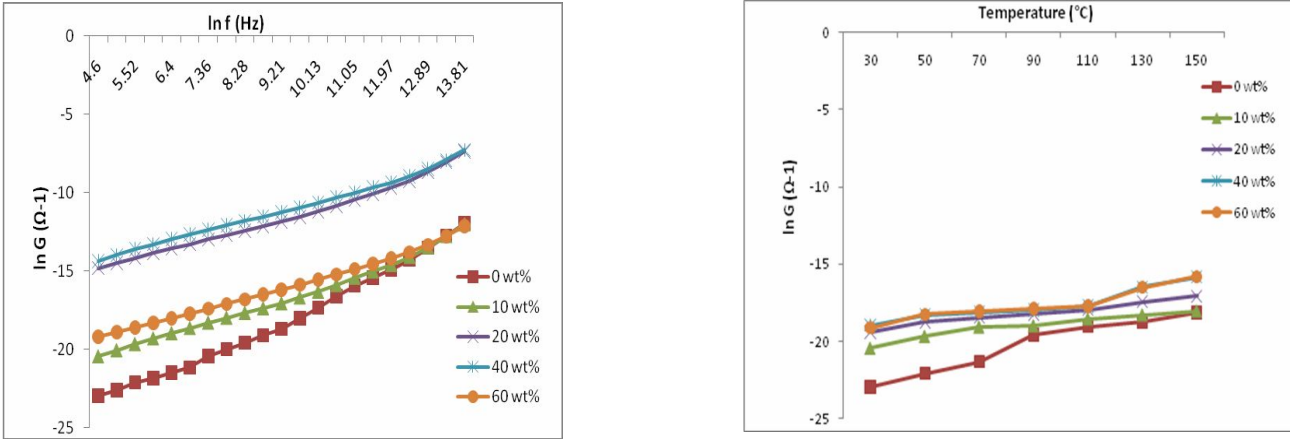
(a) (b)

Figure 10. Variation of A.C Conductance with (a) Frequency (b) Temperature for different wt. %of coir dust reinforced composite.
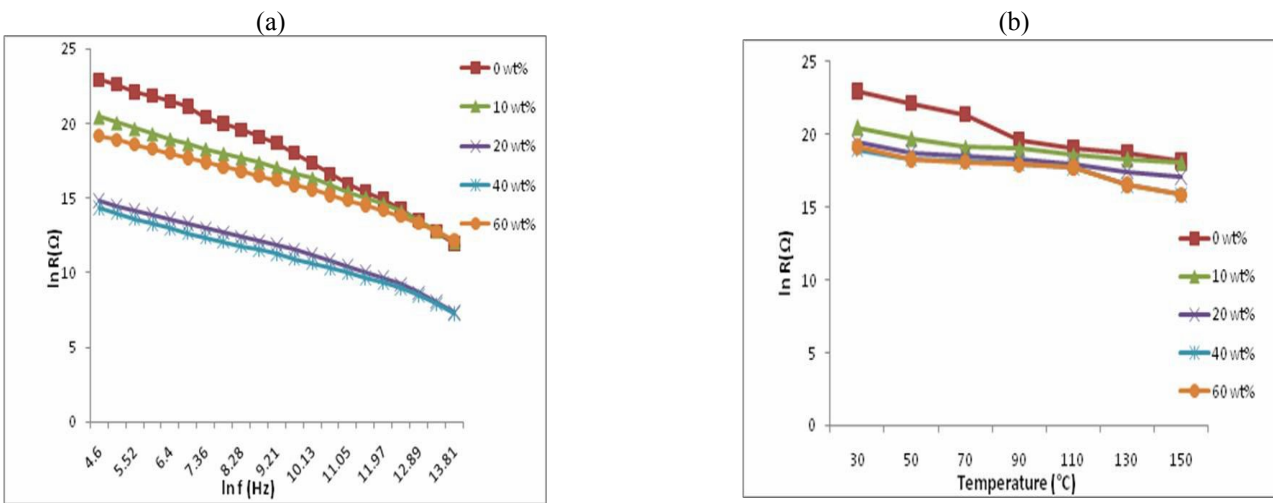

Figure 11. Variation of A.C Resistance with (a) Frequency (b) Temperature for d i f f e r e n t wt. % ofcoir dust in epoxy resin composite.

4.4 Effect of chemical treatment on the properties of composite

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 12.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 **[39].**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 **[40, 41]**.

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 **[42, 43]**. The peak 1620 cm^{-1} is present in KOH treated sample indicates the presents of hemicellulose content and the disappearance of peak 2938 cm-1 and presents of 3354 cm^{-1}, indicates that partially 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 1658 in case of HCl treated coir dust, indicates the presence of hemicellulose content.
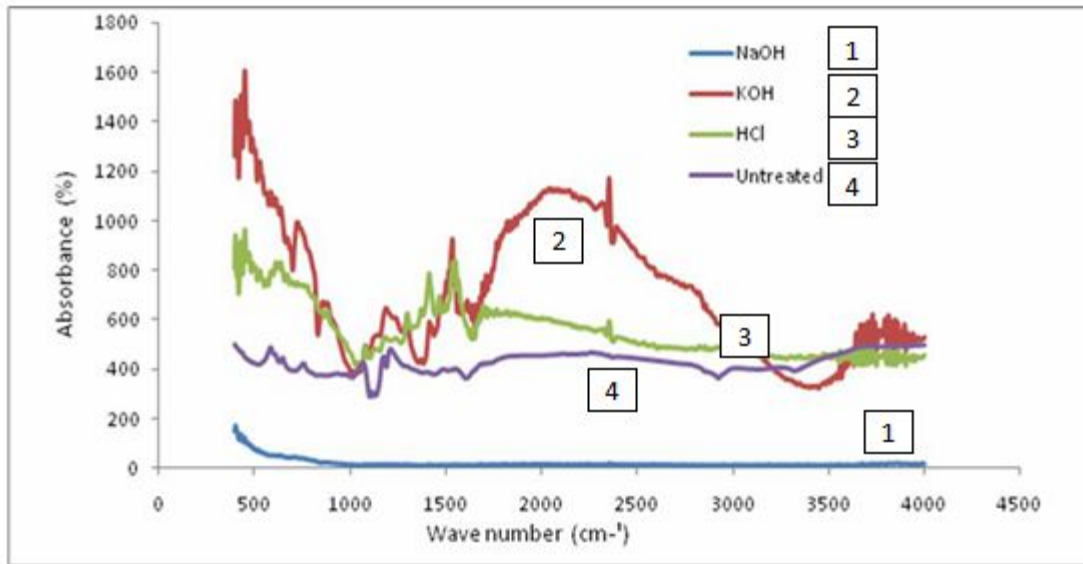
Figure 12: FTIR experimental analysis of treated and untreated coir dust.

4.5 Effect of the Chemical Treatment on Dielectric Properties

Dielectric constant of untreated and treated coir dust filled composite is shown in Figure 13. It is observed that the treated samples are having the lower dielectric constant compare with the untreated composite. The main reason for this is decrement of the orientation polarization due to the less interaction between the 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 the moisture absorption **[44, 45].**
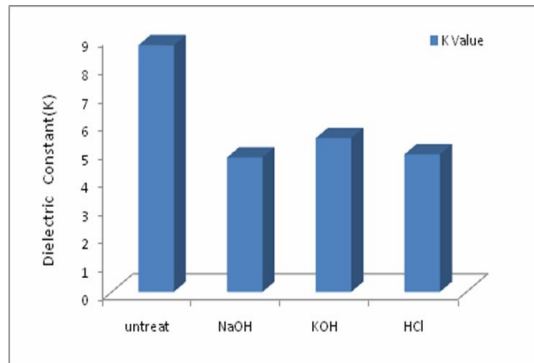

Figure 13. Variation of the dielectric constant of treated and untreated coir dust filled polymer composite.

Dielectric loss for treated and untreated composites was presented in Figure 14. From the figure it is observed that the dielectric loss of the treated samples is decreased; this is due to the increment in the density of the composite, causing decrement in the charge storage inside the voids.
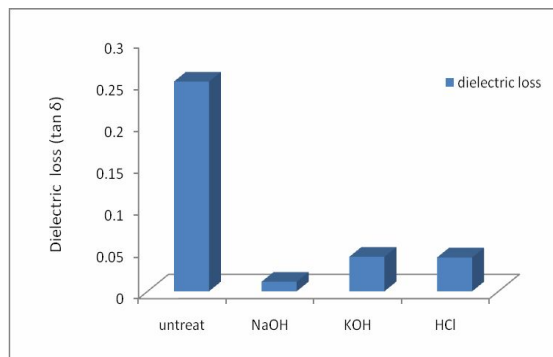

Figure 14: Variation of the dielectric loss of treated and untreated coir dust filled composite.

5. CONCLUSIONS

➢   Dielectric properties (viz. relative permittivity and loss factor), A.C conductivity, and A.C resistivity of pure epoxy resin and coir dust composites have been studied in the frequency range 100Hz-1MHz and in the temperature range 30-150°C .The experimental results indicate that the dielectric constant (varies between 6.29 to 11.84 with wt.% of coir dust) and dielectric loss factor (varies between 0.00934 to 0.34512 with wt.% of coir dust) are decreases with increasing frequency; due to the orientation polarization and increases with increasing temperature due to greater movement of polar molecular dipole. The A.C conductivity has increased and A.C resistivity is decreased with   increasing frequency at room temperature. Maximum variation of conductivity and resistivity with frequency is observed at 40 wt.% coir dust addition.

➢   These coir dust reinforced composites can be useful in the electronics industry for its dielectric and insulating properties in the use of capacitors, microelectronic components (such as transistors and ICs), and safe

insulators for low to moderate voltages (hundreds of volts).

➢ Coir dust was treated in different environmental conditions. It is found that, the dielectric constant of treated coir dust composite is lower than t h a t of the untreated coir dust composite. As a whole it can be concluded that, the coir dust reinforced polymer composite, improves the dielectric behavior by (~15-46%) than that of the epoxy (matrix material). A low cost composite could be processed and high valued utilization as well as pollution caused by the coir dust can be prohibited.

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