



STUDY OF ELECTRICAL CONDUCTIVITY OF SISAL FIBER/ POLYPROPYLENE/ CENOSPHERE COMPOSITES

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Abstract

This paper reports the d.c. conductivity behaviour of power plant cenosphere obtained from flyash filled chopped sisal fibre reinforced polypropylene composites. Treated and untreated cenosphere with different concentration were loaded with chopped sisal in polypropylene. The loading of the polypropylene with the chopped sisal fiber and cenosphere increases the dc conductivity σ_{dc} . d.c. conductivity increases on increase of cenosphere content from 0.02 to 0.08 weight %. The activation energy values for untreated composites (2, 4,6, 8 phr) are 5.52, 6.85, 7.03 and 8.37 eV respectively which shows decrease in activation energy with increase of cenosphere content.

Key Words: Composite, electrical properties, fiber, flyash waste, cenosphere.

Introduction

Composites, based on thermoplastics, are now becoming popular due to their easy processing. Polypropylene (PP) possesses outstanding properties like low density, good abrasion resistance and excellent electrical properties. The physical properties of thermoplastics are insufficient for specific applications, and for this, different fillers such as glass, china clay, talc, CaCO_3 and natural lignocellulosic fibers such as jute, bast fiber are mixed to suit the requirements [1-2]. Conductivity in filled polymer composites is generally due to the formation of a continuous network of filler particles or fibers through the polymer matrix [3]. Clingerman [4] reported that the shape of the filler also changes the conductivity of the composite [4]. Lignocellulosic fibers can influence the conductance of the materials when they are used as the reinforcements [4].



Some studies have been reported, focusing on the mechanisms responsible for the conduction process within the composite [5]. The addition of fibers can modify the dc conductivity of the composite, but only when the added fibers are highly conductive compared to the matrix. Fibers, which are insulating in nature with respect to the matrix, will have little effect on the electrical properties [6]. The electrical properties of matrices—polymer, concrete, ceramic and short fibre filled composites were investigated by Chung et. al. [7-9].

dc conductivity of sisal fiber/polypropylene composites with and without cenosphere have not been determined. The aim of this work is to show the influence of cenosphere loading, treatment of sisal fiber and of cenosphere on dc conductivity of PP/sisal fiber cenosphere composites.

Materials and Methods

Materials

The sisal fiber used in the present study was collected from Bilaspure, India. Density of the sisal fiber was 1.45 g/cm³. Isotactic Polypropylene (PP) with density 0.915 gm/cm³ was obtained from IPCL Vadodra. Cenospheres of flyash used in this investigation were obtained from Cenosphere India Pvt Ltd. Kolkata of size 150 µm having density 0.6 gm/cm³.

Treatment of Fibers

Sisal fiber (length 5-7 mm) were immersed the of in a solution of 340 ml xylene having 18.5 gm of Maleic anhydride (MA) and 1.5 gm of benzoyl peroxide as catalyst and then heated at temperature of 140°C for 4 hours then dried at 70 °C in a vacuum oven until a constant weight is gained.

Surface Treatment of cenosphere

Cenospheres of size 150 µm were dried in an hot air oven at 50 °C for 20 minutes before treatment to make it moisture free and then treated with octadecyltrichloro (OTDC) silane with very low content of it. In a solvent of methanol and water having ratio 3:1, 0.4 wt % of silane is mixed. This solution was then hydrolyzed by 20 % acetic acid and cenospheres were

mixed in this solution with constant stirring. This slurry was stirred manually for 15 minutes gradually and kept at room temperature for 1 hour. These treated Cenospheres are then washed with distilled water 2-3 times. The slurry was then dried for 24 hours at room temperature and kept in vacuum oven at 120 °C for 20 minutes to make it moisture free.

Composite preparation

Table 1 lists the composites used in the study. Varying the weight % of treated and un treated sisal fibers (10, 20, 30), were mechanically mixed with PP granules and compounded on a two roll mill by keeping the rollers at 200°C, weighed amount of cenosphere (treated and un treated) were mixed gradually with fibers. Fourteen compositions (table 1) of PP/sisal/cenosphere (treated and un treated) were prepared respectively in identical conditions. Sheets were cut to 7x6 mm² size for making measurements.

Methods

Resistance measurement

Sliced PP/sisal/cenosphere samples were coated by air-drying type conducting silver paint on both the sides. Resistance (R) values of these samples were measured by using a Kiethley Electrometer, model 610C in the temperature range from 30-120°C. Resistivity is calculated by following equation,

$$\rho = R \cdot A / L$$

Where, R is the resistance of the sample. A (cm²) is the area of the electrodes and L is the thickness.

dc conductivity calculation

dc conductivity was calculated by using the following formula

$$\sigma_{dc} = 1 / \rho = \frac{L}{RA}$$

Where, σ_{dc} is the dc conductivity of PP/sisal/cenosphere, R is the resistance, A is the area and L is the thickness of the sample.



Density Measurements

Density of the PP/sisal/cenosphere samples was determined by using a high precision Citizen machine, Model CX 265 as per ASTM D 792. Average of four values is reported here in table 2.

Surface Observation

Surfaces of the PP/sisal fiber /cenosphere composites were observed by using a FESEM (Nova Nanosem 430, FEI™ USA). Cross section of samples was polished before observation.

Result and Discussion

Table 1 lists the ingredients used for the preparation of the samples. Composites prepared had different ingredients concentrations which are listed in table 1.

Table 2 shows the density values of the composites prepared; it is clear that the addition of sisal fiber and cenosphere increases the density whereas treatment of sisal fiber and cenosphere decreased the density. This is because some impurities removed.

Fig.1a shows the effect of maleic anhydride treated and untreated chopped sisal fiber concentration on dc conductivity at different temperature from 30 to 120°C.

Fig.1b shows the effect of cenosphere content on the dc conductivity of untreated sisal/PP composites. It is clear that addition of cenosphere in chopped sisal fiber/PP composite (80/20) increases the dc conductivity. Increase in cenosphere content reduced the dc conductivity (80/20) concentration. Addition of cenosphere increases the porosity of the composites which tends to decrease the dc conductivity. It is clear from Fig.1b is that for the same sisal fiber content, the greater cenosphere proportion in the composite, shows the smaller is the dc conductivity of the composites. The other way to evaluate the fillers effect on the composite dc conductivity, is investigation of the effect of increasing cenosphere while sisal fiber concentration is kept constant. This



decrease is due to displacement of electrical charges being displaced inside the polymer at lower concentration. The decreases in dc conductivity by increase in cenosphere wt% may be due to particle blockage conduction path by cenosphere in chopped sisal fiber/PP composites. This plot shows that up to 75°C there is no increase in d.c. conductivity. There is a sudden increase in d.c. conductivity with increase of temperature for different cenosphere concentration is found at 75°C for 2,4,6 and 8 respectively. As the cenosphere content increased the interchain distance also increased due to which hopping between chains becomes more difficult, and thus decreases the dc conductivity. The factors that affect the dc conductivity of the cenosphere filled chopped sisal fiber/PP composite are filler concentration and porosity. In case of dc conductivity of filled polymers interface interaction is the most important parameter which influences the dc conductivity of matrix and will depend on type of fillers, concentration of filler and the chemical bonding between filler and matrix. In this figure it is also observed that there is a peak in dc conductivity at 95°C, 100°C, 100°C and 95°C for 2,4,6 and 8 weight % of cenosphere respectively.

Fig.1c shows that treatment of sisal fiber decreases the dc conductivity of the composites. This is because the treatment of fibers removed the waxes and impurities from the fiber surface. Fig. 1c shows the dc conductivity of the treated cenosphere filled PP/sisal (80/20) composites. It is clear from the graph that the addition of treated cenosphere in the PP/sisal fiber composites decreases the dc conductivity of the composites whereas as with increase in temperature of the composites dc conductivity increases.

The incorporation of hydrophilic natural fibers in polymers leads to heterogeneous systems whose properties are inferior due to lack of adhesion between the fibers and the matrix. Thus the treatment of fibers for better linkage is a significant step in the development of such composites [2]. Pothan et. al. [8] studied the effect of addition of fibers

on conductivity and reported that the conductivity increased upon the addition of lignocellulosic fibers. This is due to the presence of polar groups, which facilitate the flow of current [8]. The resistivity of fiber reinforced composites depend on the moisture content, crystalline and amorphous component present, presence of impurities, chemical composition, cellular structure, microfibrillar angle etc. The shapes of reinforcement determine the interparticle contact, which affect the conductivity of the system. Fibers and flakes having elongated shapes which affect the electrical conductivity [9]. Blythe [10] studied that the moisture content present in fibers increases the conductivity [10]. The hydrophilicity of cellulose fiber is generally responsible for greater conductivity of the composite. In polymeric materials most of the current flow takes place through the crystalline regions and non crystalline region allows current to pass through it mainly when moisture is present [11]. The hydroxyl groups in the hydrophilic fiber such as sisal fiber absorb moisture and hence the presence of the sisal fiber increases the conductivity of the composite.

Fig. 2(a) show the FESEM images of PP/sisal fiber/cenosphere composite (80/20/2). This microstructure of composite has untreated sisal fiber and untreated cenospheres in PP matrix with out any bonding, which is very clear in the microstructure. Fig. 2(b) shows the FESEM image of PP/treated sisal fiber/treated cenosphere composites (80/20/2). In this microstructure improved bonding between treated sisal fibre is clearly visible. Fig. 2(c) show the FESEM images of PP/sisal fiber/cenosphere composite (80/20/8). This microstructure of composite has untreated sisal fiber and untreated cenospheres in PP matrix with out any bonding, which is very clear in the microstructure. Fig. 2 d shows the FESEM image of PP/treated sisal fiber/treated cenosphere composites (80/20/8). In this microstructure improved bonding between treated sisal fibre is clearly visible. Cenosphere is also visible in this photo. With increasing of the content of the fibers conductive paths among the fibers

increases, and the average distance between the fibers becomes smaller; thus, the dc conductivity of the composites decreases. dc conduction in polypropylene is reported by Singh and Gupta [12]. They reported that the thermal agitation gives rise to defects in the material and conduction takes place by movement of ions from an occupied position to an unoccupied position. $\ln\sigma_{dc}$ vs $1000/T$ plot between for high temperature range has been analysed by using the following Arrhenius equation [12].

$$\sigma = A \exp(-W_E/kT)$$

Where, W_E is the activation energy of dc conduction, (eV), k the Boltzmann's constant, A is a constant and T is the temperature in °K.

Table 3 gives the values of activation energy of the samples. Figure 3 gives the activation energy curve analysed at different temperature for the samples. In case of untreated sisal fiber increase in cenosphere decreases the activation energy this is because due to the poor bonding of cenosphere with PP. Activation energy is increasing with the increase in treated cenosphere content in the composites because bonding has increased with treatment, mobility of dipoles reduced which increased the activation energy.

Chemical treatment of sisal fiber increased the activation energy from 4.66 to 5.15 eV for PP/sisal composites of 80/20 composition. On adding 2 phr treated cenosphere to PP/sisal (80/20) composite suddenly reduced the activation energy to 2.44 with increase in treated cenosphere concentration to 8 phr. It is found that PP/tr. Sisal/tr. Cenosphere (80/20/8) exhibited maximum activation energy of 6.30. In case of PP/ut sisal/ut cenosphere activation energy increased. PP/ut sisal/ut cenosphere having 2, 4, 6 and 8 phr untreated cenosphere had activation energy as 8.37, 7.03, 6.85 and 5.52 eV respectively. This shows that treated cenosphere gave good bonding and easy transport of charge carriers which reduced on increase of treated cenosphere concentration. In case of untreated cenosphere activation energy increased due to the increase in cenosphere concentration only. Lower values of activation



energy are observed in case of PP/tr. Sisal composite/ tr. Cenosphere (80/20/2) is probably due to impurity conduction domination by electronic conduction. Activation energy is calculated for high temperature region in which intrinsic conduction was often reported earlier [12]. Maximum resistance is observed in case of 2 wt% untreated cenosphere which reduced on increasing cenosphere content. This is because defects and ionic impurities increased. Increase in cenosphere concentration reduced the free spaces and reduced the hindrance which led to decrease in activation energy. Activation energy of cenosphere filled sisal fiber reinforced polypropylene composites are listed in table 3 and is always > 1 . On treatment of fibers and cenospheres reduced the activation energy to minimum for 2 wt 5 treated cenospheres filled composites and exhibited electronic conduction only. Increase in treated cenosphere content further increase the activation energy and exhibited ionic conduction. This decrease of activation energy with increase of cenosphere content is similar to the activation energy calculated for filled blends [12] which has been explained by the formation of border layer at the interface of the filled polymer.

Conclusions

1. In this paper, experimental study about the effects of sisal fiber/cenosphere content on the dc conductivity of composites of polypropylene has been described.
2. Composites with higher concentration (8gm) of cenosphere shows low values of dc conductivity compared to less concentration (2gm) of cenosphere composites.
3. Activation energy decreases with increase of cenosphere content.



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FIGURE CAPTIONS:

Figure 1 shows the dc conductivity for all samples in the temperature range 30-130°C.

Figure 2 shows the FESEM images of PP/sisal fiber/cenosphere composites.

(a) PP/ut sisal fiber/ut cenosphere (80/20/2)

(b) PP/tr sisal fiber/tr cenosphere (80/20/2)

(c) PP/ut sisal fiber/ut cenosphere (80/20/8)

(d) PP/tr sisal fiber/tr cenosphere (80/20/8)

Figure 3 shows the $\ln \sigma_{dc}$ vs. $(1000/T)$ curve for all samples at high temperature range 353-393 K.

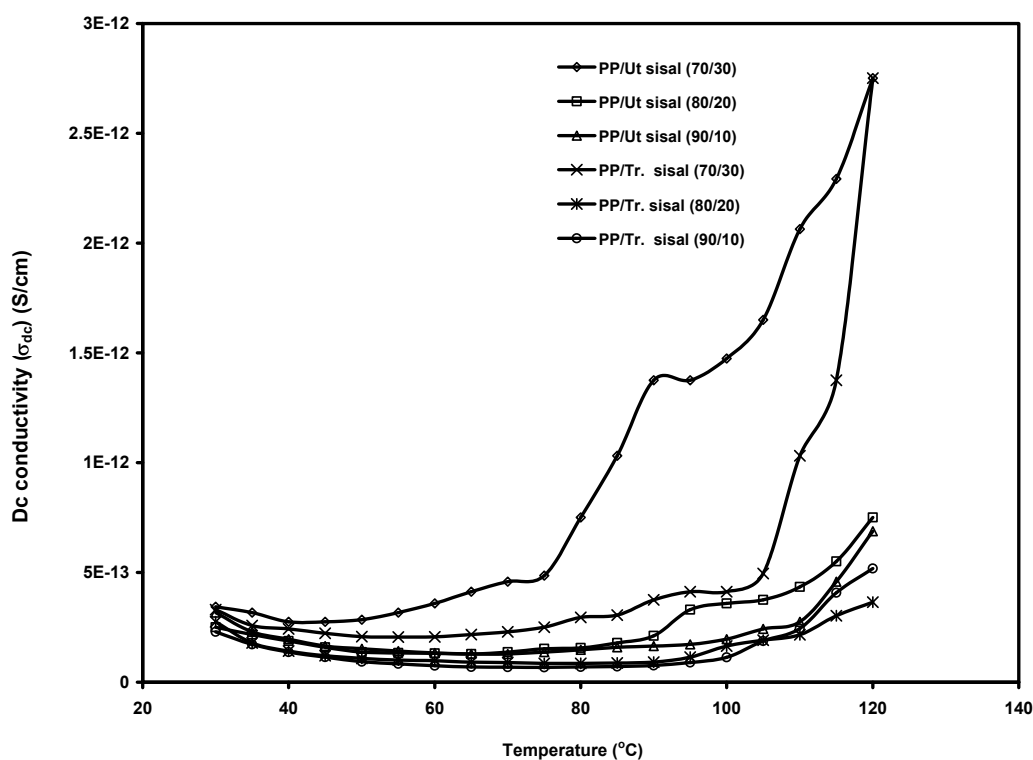


Figure 1. (a) Shows the dc conductivity for all samples in the temperature range 30-130°C

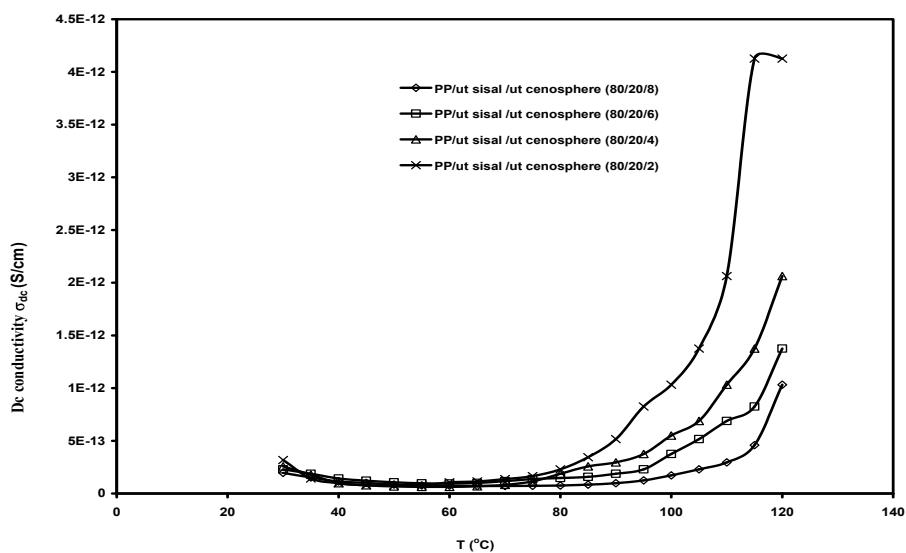


Figure 1 (b) shows the dc conductivity for all samples in the temperature range 30-130°C

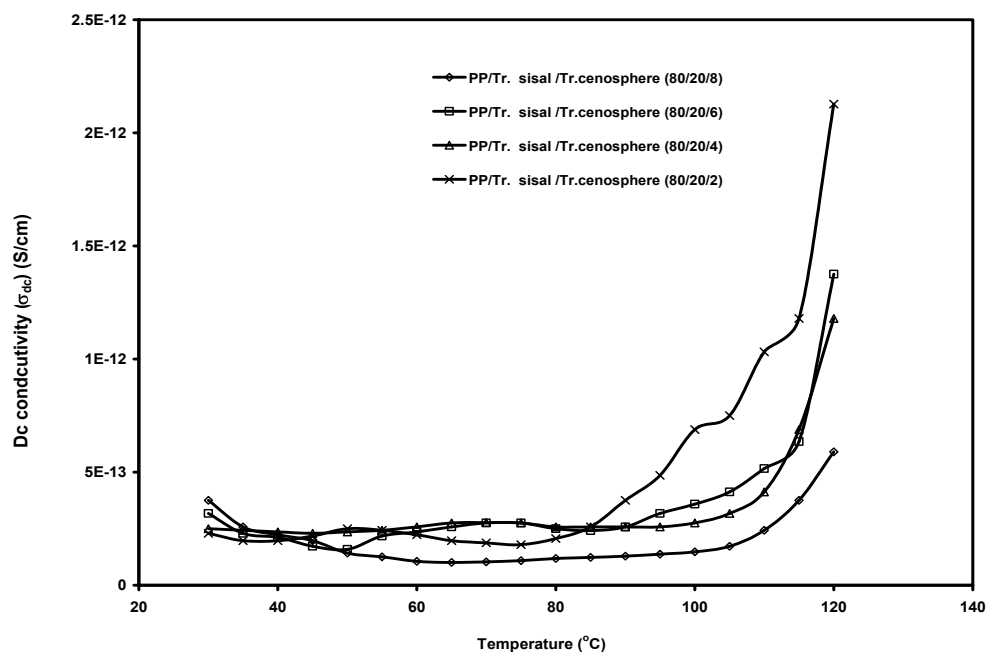


Figure 1 (c) shows the dc conductivity for all samples in the temperature range 30-130°C

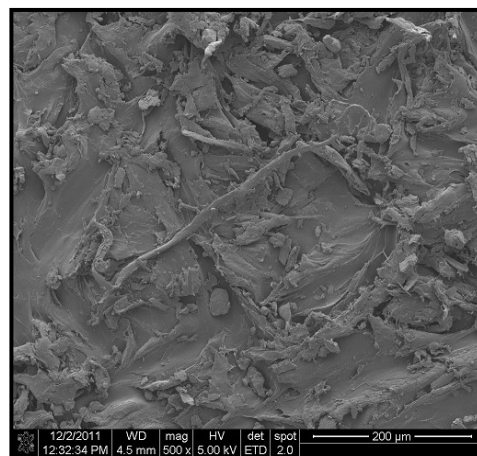
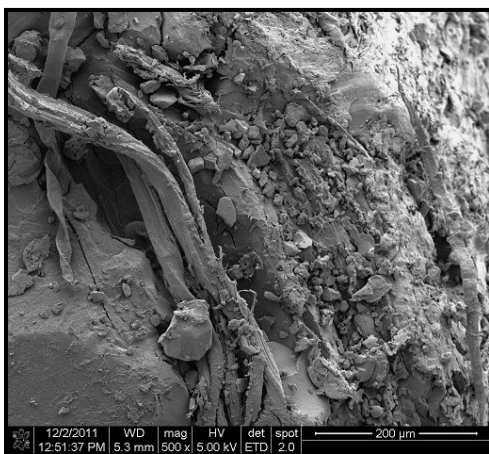


Figure 2 (a) PP/ut sisal fiber/ut cenosphere (80/20/2)

Figure 2 (b) PP/tr sisal
fiber/tr cenosphere
(80/20/2)

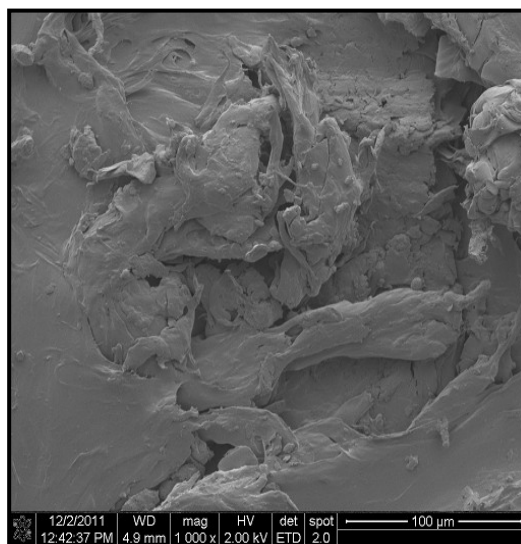
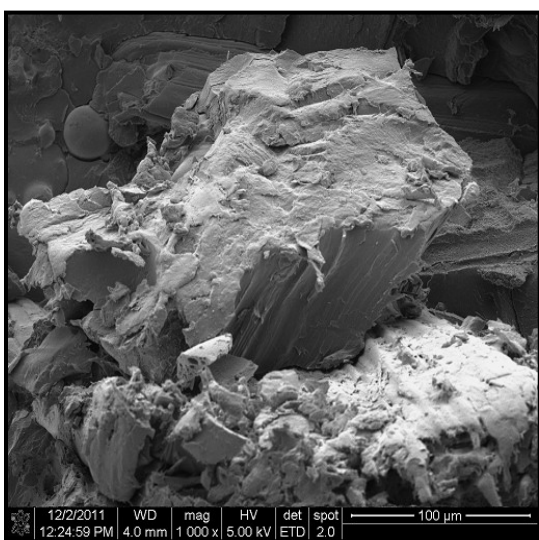


Figure 2 (c) PP/ut sisal fiber/ut cenosphere (80/20/8)

Figure 2 (d) PP/tr sisal
fiber/tr cenosphere
(80/20/8)

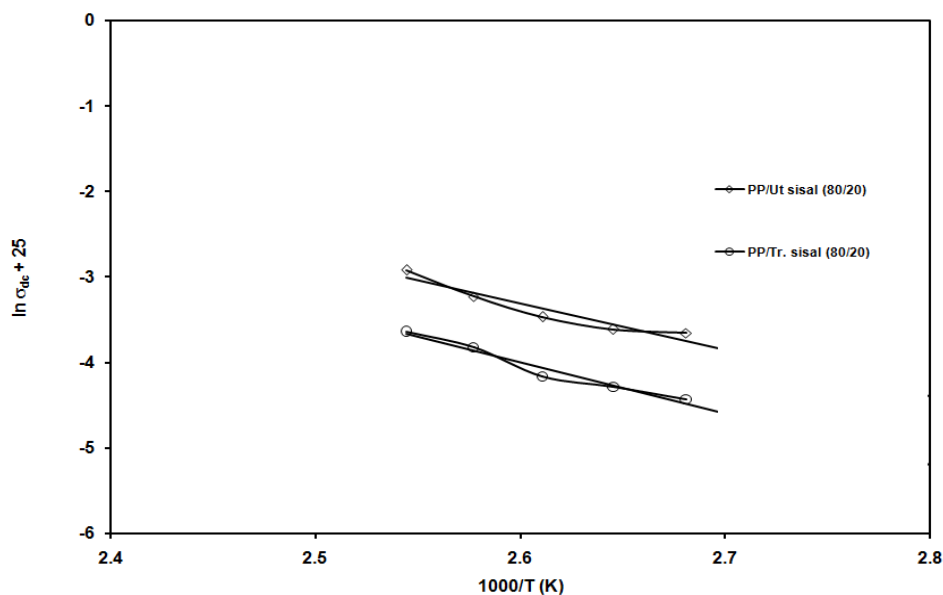


Figure 3 (a) shows the $\ln \sigma_{dc}$ vs. $(1000/T)$ curve for all samples at high temperature range 353-393 K

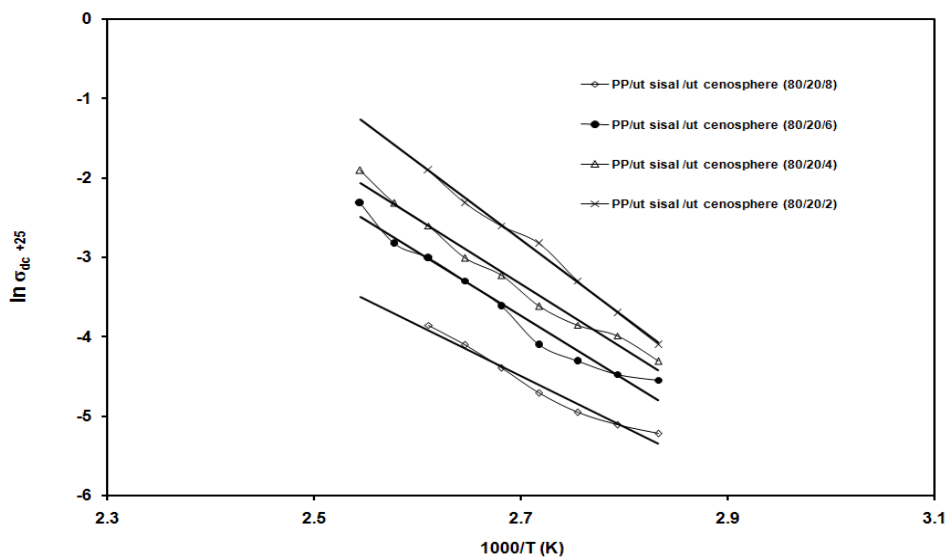


Figure 3 (b) shows the $\ln \sigma_{dc}$ vs. $(1000/T)$ curve for all samples at high temperature range 353-393 K

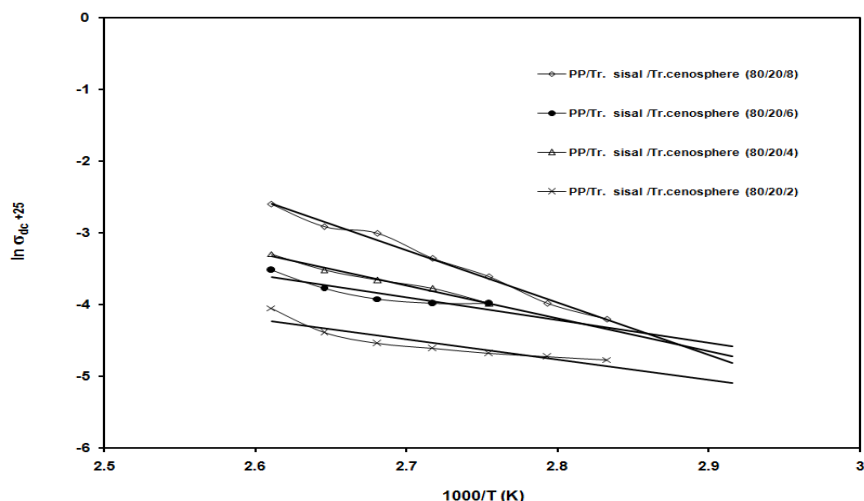


Figure 3 (c) shows the $\ln\sigma_{dc}$ vs. $(1000/T)$ curve for all samples at high temperature range 353-393 K

TABLE CAPTIONS:

Table.1 Ingredients used in making PP/sisal/cenosphere Composite

Table.2 Density of all the samples (with and with out) cenosphere.

Table.3 shows the activation energy of the samples

Table.1

S . N	Sample	Polypropylene (gm)	Sisal fiber (gm)	Treated Sisal fiber (gm)	Cenosphere (gm)	Treated Cenosphere (gm)
1	Pure PP	100	-	-	-	-
2	PP/Sisal (70/30)	70	30	-	-	-
3	PP/Sisal (80/20)	80	20	-	-	-
4	PP/Sisal (90/10)	90	10	-	-	-
5	PP/ MATr. Sisal (70/30)	70	-	30	-	-
6	PP/MATr. Sisal (80/20)	80	-	20	-	-
7	PP/ MATr. Sisal (90/10)	90	-	10	-	-
8	PP/ Sisal/Ceno (80/20/8)	80	20	-	8	-
9	PP/ Sisal/Ceno (80/20/6)	80	20	-	6	-
10	PP/Sisal/Ceno (80/20/4)	80	20	-	4	-
11	PP/Sisal/Ceno (80/20/2)	80	20	-	2	-
12	PP/MATr. Sisal/ Silane Tr.Ceno (80/20/8)	80	-	20	-	8
13	PP/MATr. Sisal/Silane Tr.Ceno (80/20/6)	80	-	20	-	6
14	PP/MATr. Sisal/ Silane Tr.Ceno (80/20/4)	80	-	20	-	4
15	PP/MATr. Sisal/ Silane Tr.Ceno (80/20/2)	80	-	20	-	2

Table.2

S.N	Sample	Density (g/cc)
1	Pure PP	0.905
2	PP/Sisal (70/30)	0.9371
3	PP/Sisal (80/20)	0.92895
4	PP/Sisal (90/10)	0.91245
5	PP/ MATr. Sisal (70/30)	0.92754
6	PP/MATr. Sisal (80/20)	0.92107
7	PP/ MATr. Sisal (90/10)	0.91018
8	PP/ Sisal/Ceno (80/20/8)	0.95180
9	PP/ Sisal/Ceno (80/20/6)	0.94517
10	PP/Sisal/Ceno (80/20/4)	0.93862
11	PP/Sisal/Ceno (80/20/2)	0.93281
12	PP/MATr. Sisal/ Silane Tr.Ceno (80/20/8)	0.94665
13	PP/MATr. Sisal/Silane Tr.Ceno (80/20/6)	0.93676
14	PP/MATr. Sisal/ Silane Tr.Ceno (80/20/4)	0.92653
15	PP/MATr. Sisal/ Silane Tr.Ceno (80/20/2)	0.92358

Table.3

S.N.	Sample	Slope	Activation energy (eV)
1	PP/Sisal (80/20)	4.8623	4.1864
2	PP/MATr. Sisal (80/20)	4.7552	4.094
3	PP/ ut Sisal/ut Ceno (80/20/8)	7.077	6.093
4	PP/ ut Sisal/ut Ceno (80/20/6)	7.9659	6.853
5	PP/ut Sisal/ut Ceno (80/20/4)	8.1732	7.0371
6	PP/ut Sisal/ut Ceno (80/20/2)	10.315	8.8812
7	PP/Tr.Sisal/Tr.Ceno (80/20/8)	4.6181	3.9761
8	PP/Tr.Sisal/Tr.Ceno (80/20/6)	5.1405	4.4259
9	PP/Tr.Sisal/Tr.Ceno (80/20/4)	5.1990	4.4763
10	PP/Tr.Sisal/Tr.Ceno (80/20/2)	8.7464	7.5306