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Dielectric Properties of Parkia Powder Agar and Its Blends with Ethyl Cellulose†

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The dielectric properties and A.C. conductivities of parkia powder agar for different sample thicknesses and its blends with ethyl cellulose for various weight percentages have been studied as a function of frequency over the range 50 Hz-5 MHz at room temperature. The characterizations for crystallinity of parkia powder and its blends have studied using XRD. The morphological studies have been made using the SEM. The value of dielectric constant of parkia powder agar decreases gradually as thickness of the sample decreases. But as thickness of the sample increases the dielectric constant decreases exponentially over the frequency range 50 Hz to 10 KHz and afterwards it remains constant as frequency increases. This leads to the occurrence of changes in the properties of dielectrics of parkia powder agar as thickness of the sample increases. In case of parkia powder agar blends with ethyl cellulose the dielectric constant decreases gradually up to frequency of around 1 KHz and at higher frequencies it remains constant over all weight percentages of the ethyl cellulose. It is also observed that the dielectric constant of parkia powder agar blend with ethyl cellulose decreases as weight percentage of the ethyl cellulose increases. The A.C. conductivity of parkia powder agar remains constant over the frequency range 50 Hz to 300 KHz and afterwards the conductivity increases exponentially as frequency increases. Further, it is observed that A.C. conductivity of parkia powder agar increases as thickness of the sample increases in the frequency range of 300 KHz to 5 MHz. The dielectric loss of parkia powder agar increases as thickness of the sample increases. In case of its blends with ethyl cellulose the dielectric loss decreases as the weight percentage of ethyl cellulose increases.

Key Words: Dielectric constant, Dielectric loss, XRD-characterization, Activation energy, D.C. conductivity.

INTRODUCTION

The dielectric properties of polymer and its composites are dependent upon several factors, including method of preparation, chemical composition, doping concentration and grain structure or size. Polymer composites have steadily gained importance during past one/two decades, because of the need for electrostatic charges, dissipation, electromagnetic shielding etc. The electrical conduction in polymer film has much importance due to the discovery of the memory phenomenon[1] and currently has wide applications in thin film devices[2]. Electrical properties constitute one of the most convenient and sensitive methods for studying the polymer structure[3,4]. The electrical properties of regenerated cellulose (cello phone) film were studied by others[5]. The current-voltage characteristics of malachite green doped cellulose acetate films[6], for the measurement of electrical conductivity, as function of temperature, field, thickness and dopant concentration have been studied. The present work evolved the A.C. and D.C. measurements of naturally occurring parkia powder agar (PPA) material and its blends with different weight percentage of ethyl cellulose. The parkia powder taken from the pods of a plant belongs to the subfamily Mimosoideae and family Leguminosae[7]. In India, it is found in parks and as avenue trees. Pods attain maturity during March to May and hang from the peduncle in clusters. The pods produce a creamy-white powder with flour-like texture. A new substance named as parkia powder agar (PPA) is used separately for the dielectric and A.C. measurements and also as a doping material with ethyl cellulose polymer for the studies of electrical properties. The dried pods collected from Gulbarga University campus. The parkia powder agar is a good source of ascorbic acid, fat, proteins and minerals. The pod contains minerals such as Ca (97.47mg/100g), K (2400), Cu (2.3), Zn (2.77), Fe (57.1) and Mn (35.0 mg/100 g) as reported by Seanor[8].

EXPERIMENTAL

The parkia powder agar (PPA) is obtained from the pods of a leguminous plant. The different weights such as 250, 300, 350, 400, 450 and 500 mgs of parkia powder agar substance

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was taken to make the pellets of different thickness in circular shape. Thickness of the pellets is measured using screw gauge. The polymer of ethyl cellulose is obtained from S.D. Fine Chem. Ltd., Mumbai, India. The pellets of 10 mm diameter have been made by applying 2-3 tons of pressure using the pellet making machine with 10 mm diameter. Further the blends of parkia powder agar are obtained by doping with the ethyl cellulose in different weight per cent *viz.*, 30, 60 and 80 weight percentages. The pellets of pure parkia powder agar samples and its blends with ethyl cellulose at different weight per cent have been used for the measurements of electrical properties. The silver paste is coated on either side of the pellets for an Ohmic contact to provide the electrical connections for the measurements. The prepared samples are used to measure the capacitance, impedance, phase angle and dissipation to determine the dielectric constant (ε'), loss factors (ε") and A.C. conductivity as a function of frequency over the range from 50 Hz-5 MHz at room temperature using PC based LCR meter (Model: HIOKI 3552-50-LCR Hitester). The dielectric constant has been calculated from the capacitance values using the relation given below.

$$\varepsilon' = \frac{Cd}{\varepsilon_0 A} \quad (1)$$

sample and ε_0 is the permittivity of free space. Further, from the measured values of dielectric where C is capacitance of the dielectric material, d is thickness of the sample, A is area of the constants, dielectric loss and A.C. conductivities (σ_{ac}) are calculated using the eqns. 2 and 3, respectively, which are given below:

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \quad \text{or} \quad \varepsilon'' = \varepsilon' \tan\delta \quad (2)$$

$$\sigma_{ac} = \varepsilon' \varepsilon_0 \omega \tan\delta \quad (3)$$

Further, we have also observed the presence of some of the minerals in the parkia powder agar sample using the atomic absorption spectrometer.

RESULTS AND DISCUSSION

X-Ray diffraction studies: The crystallinity of parkia powder agar have been studied using powder X-ray diffractometer (XRD) (Philips Analytical) and observed the strong intensive peak of pure parkia powder agar indicate its crystalline nature but the diffuse peaks of the composite show its amorphous behaviour. In the parkia powder agar sample sharp and diffuse peaks observed at 2θ angles 19º and 22º with peak heights 133 and 211, respectively (Fig. 1). The values of lattice parameter (d) at angles 19º and 22º are 4.6559 Å and 4.0226 Å, respectively which were calculated using the Bragg's Law. The ethyl cellulose has also been characterized separately for study of the crystallinity (Fig. 2). The XRD shows peaks at angles 90, 11.280 and 20.980 with peak heights 387.00, 473.35 and 436.50, respectively. The values of lattice parameter (d) at angles 9º, 11.28º and 20.98º are 9.800 Å, 7.8373 Å and 4.2294 Å, respectively. Further, crystallinity of the blend of parkia powder agar with ethyl cellulose has been characterized (Fig. 3). In case of the blend of parkia powder agar and ethyl cellulose, we observed a peak at an angle 22º with peak height 3895.

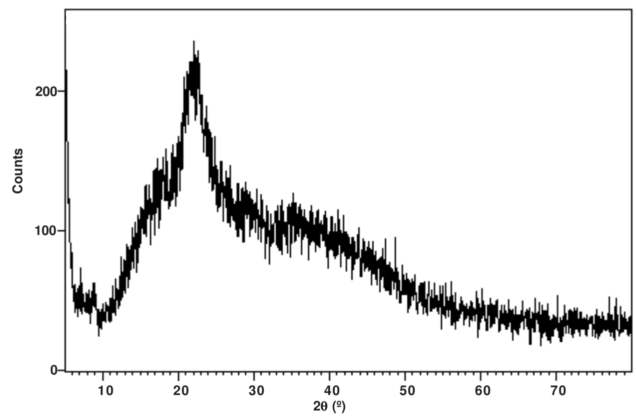

Fig. 1. XRD spectrum of pure parkia powder agar

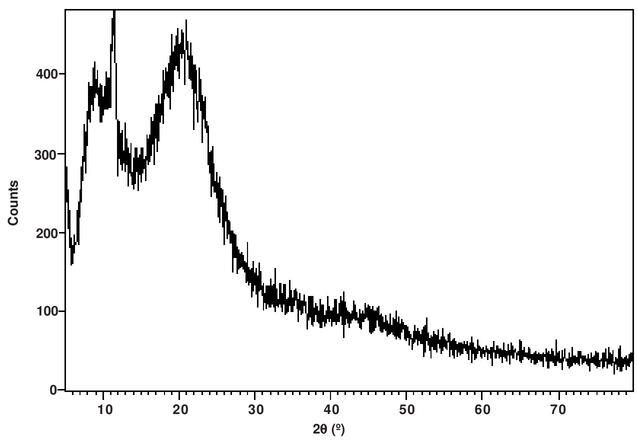

Fig. 2. XRD spectrum of pure ethyl cellulose

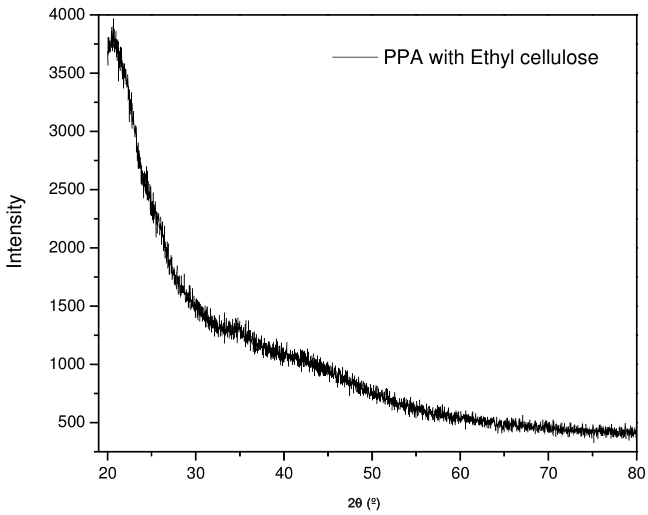

Fig. 3. XRD spectrum of the blend of parkia powder agar (PPA) with ethyl cellulose

Scanning electron microscopy (SEM): The morphological studies of parkia powder agar sample and its blend have been made using SEM. The micrographs of parkia powder agar and its blend are shown by Figs. 4 and 5. The morphological studies of the pure parkia powder agar show the microcrystalline particles. The surface of these particles in a magnified scale are showing reflecting in nature but in case of the blend of parkia powder agar with ethyl cellulose (Fig. 5), shows the crystallites possess diffusive surface of

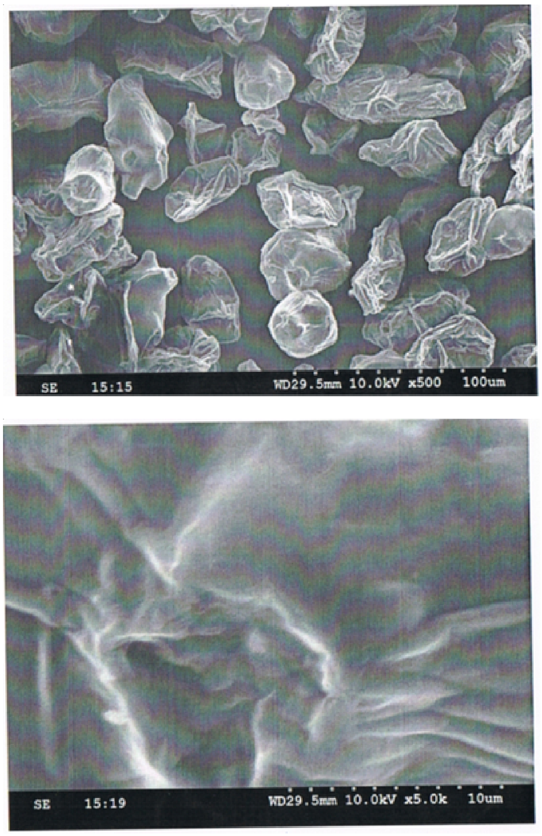

Fig. 4. SEM micrograph of pure parkia powder agar

Fig. 5. SEM micrograph of the blend of parkia powder agar with ethyl cellulose in the ratio 4:6 wt %

reflectance and also the micro scale image exhibits formation of the gel type polymerization.

Atomic absorption spectra: The standard solution of parkia powder agar sample is prepared by dissolving 1 g of parkia powder agar powder in 1:1 ratio of 25 mL of concentrated H_2SO_4 and double distilled water. The solution is stirred well for complete dissolution and further added 950 mL of double distilled water, to make 1000 mL solution, to examine the presence of minerals in parts per million (ppm). The 10 mL of standard solution was used for analysis of the ingredient mineral/materials present in the parkia powder agar using an atomic absorption spectrometer (Thermo Scientific, iCE 3000 series). The results of the spectrometer reveals that the solution contains 0.1061 mg/L of copper, 0.1995 mg/L of zinc, 0.7569 mg/L iron, 0.0417 mg/L of lead and 2.3126 mg/L of magnesium.

Capacitance measurements: The capacitance of parkia powder agar sample has been studied as a function of frequency at different thicknesses. The plots of these samples as a function of frequency at ambient temperature for different thicknesses are given in Fig. 6. It is observed from Fig. 6 that as frequency increases the capacitance of the parkia powder agar decreases exponentially at lower frequency ranging from 100 Hz to 10 KHz, afterwards it decreases gradually and remains constant at higher frequencies. The same trend shows for all the different thicknesses of parkia powder agar as frequency increases, Further it is observed that as thickness of the parkia powder agar increases the capacitance of these samples decreases.

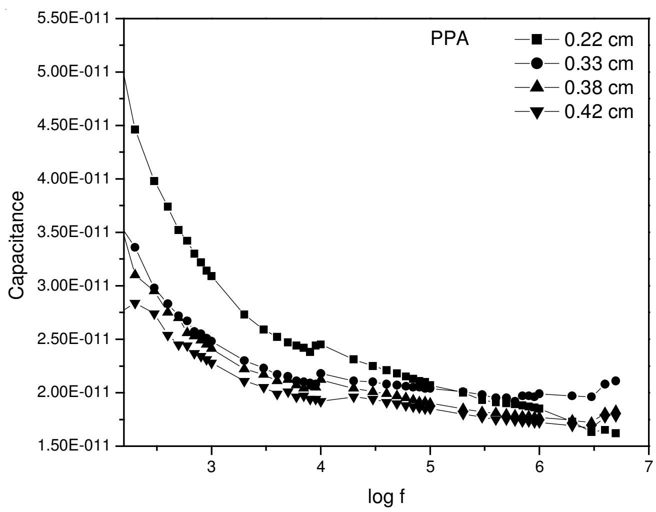

Fig. 6. Capacitance of parkia powder agar (PPA) at different thickness as function of frequency

Dielectric properties: The dielectric constant of parkia powder agar decreases exponentially as the frequency increases up to 3 KHz and gradually decreases up to 10 KHz. But at higher frequencies the dielectric constant remains same and is independent of the higher frequencies (Fig. 7). Further, it is observed that as thickness of the parkia powder agar samples increases the values of dielectric constant also increases appreciably with in the frequency range 100Hz-1 KHz. But at higher frequency range that is above 1 KHz a noticeable increase in the values of dielectric constant have

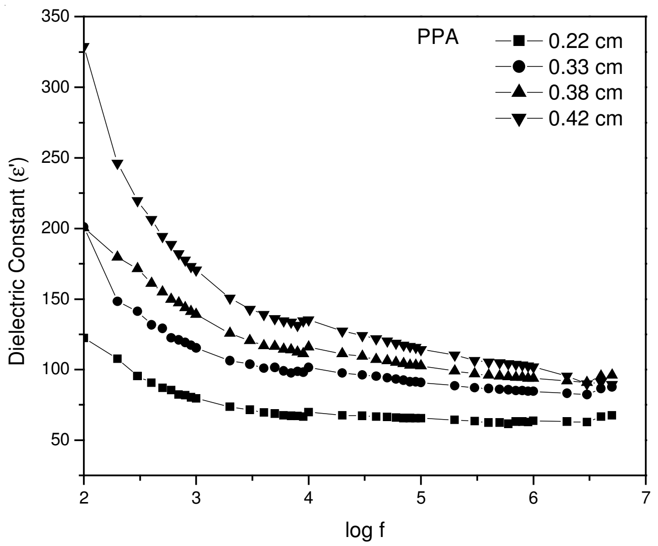

Fig. 7. Dielectric constant of parkia powder agar (PPA) at different thickness as function of frequency

been observed and those remains constant at higher frequencies.

We have also studied the dielectric properties of the blend of parkia powder agar with 30, 60 and 80 weight percentages of ethyl cellulose as function of frequency (Fig. 8). It is noticed that the values of dielectric constant of parkia powder agar have been reduced by doping with 30, 60 and 80 wt % of ethyl cellulose (Fig. 8). This implies that as dielectric constants of the blend of parkia powder agar with ethyl cellulose decrease as the weight percent of ethyl cellulose increases. It is also observed that the values of dielectric constant of pure parkia powder agar are significantly higher than those of the values of ethyl cellulose for the given thickness. Here we have seen the modified dielectric properties of the blend that is the dielectric constant lies in between the values of pure parkia powder agar and ethyl cellulose. A typical plot for dielectric constant *versus* weight per cent of ethyl cellulose at a given frequency of 200 Hz is given in Fig. 9.

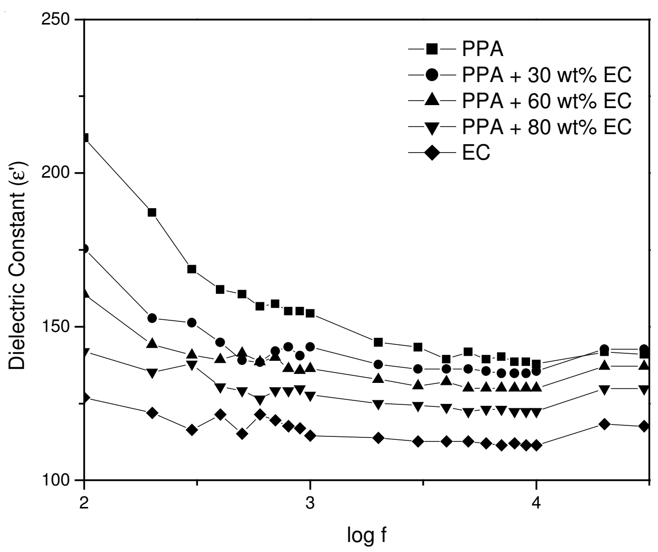

Fig. 8. Variation of dielectric constant of parkia powder agar (PPA) at different weight per cent of ethyl cellulose (EC) and its blends as function of frequency

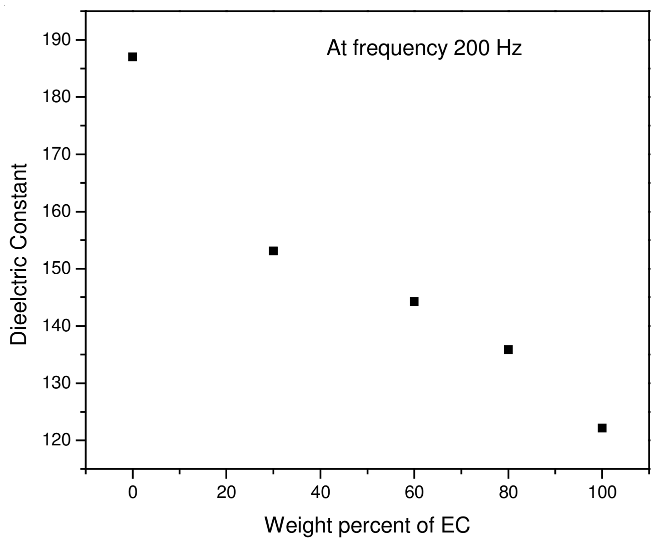

Fig. 9. Dielectric constant of parkia powder agar with different weight per cent of ethyl cellulose (EC)

Dielectric loss: The dielectric loss of parkia powder agar decreases exponentially as the frequency increases up to 1 KHz and gradually decreases up to 3 KHz but at higher frequencies the loss remains constant (Fig. 10). Further, it is observed that as thickness of the parkia powder agar increases the dielectric loss also increases with in the low frequency range 50 Hz-3 KHz, whereas at higher frequency range it remains constant.

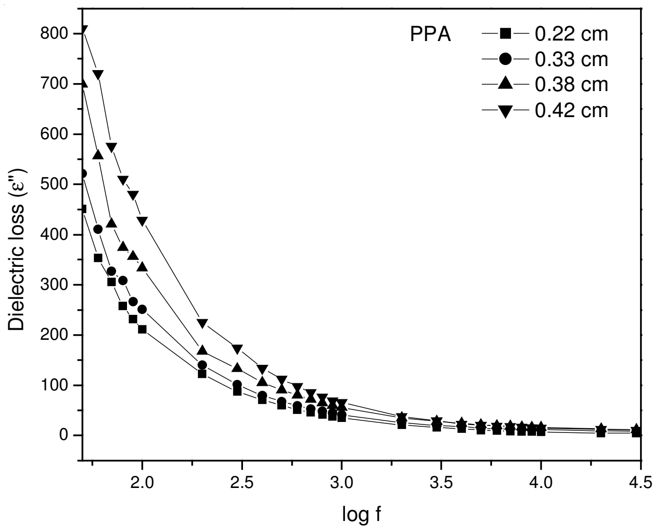

Fig. 10. Dielectric loss of parkia powder agar (PPA) at different thickness as function of frequency

Further the measurements of dielectric loss of the blend of parkia powder agar with 30, 60 and 80 weight percentages of ethyl cellulose are given in Fig. 11. It is observed that the dielectric loss of the blend of parkia powder agar decreases as the weight per cent of ethyl cellulose increases within the frequency range of 50 Hz to 3 KHz, whereas at higher frequencies the loss of the blend remains constant as frequency increases.

A.C. Conductivity: The A.C. conductivity of parkia powder agar and its blends with different weight percentages of ethyl cellulose are shown in Fig. 12. The A.C. conductivity of parkia powder agar remains constant up to 10 KHz and

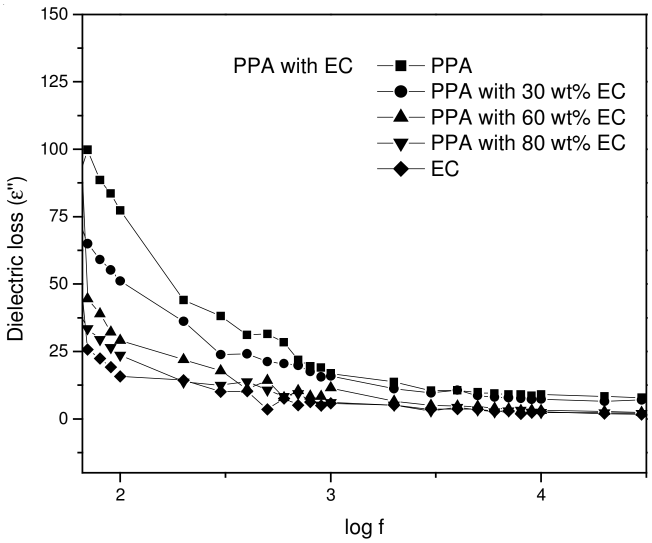

Fig. 11. Dielectric loss of parkia powder agar (PPA) with ethyl cellulose (EC) at different weight per cent as function of frequency

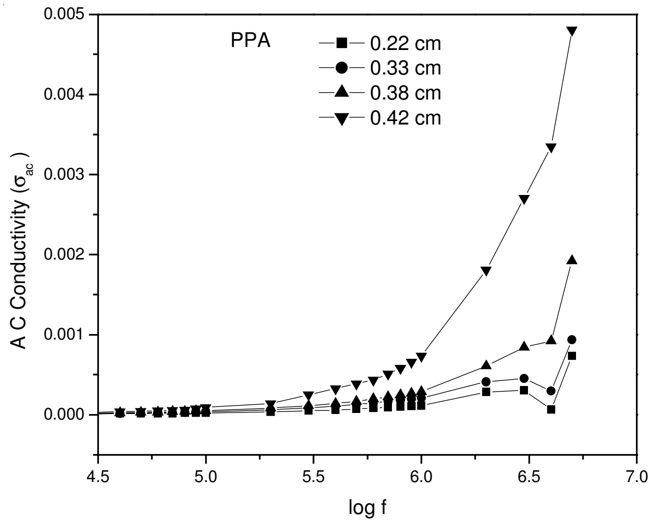

Fig. 12. A.C. conductivity of parkia powder agar (PPA) as function of frequency at different thickness

afterwards it increases exponentially as frequency increases. It is noticed that as thickness of the parkia powder agar increases the A.C. conductivity also increases at frequency 300 KHz onwards. The A.C. conductivity of the parkia powder agar at thickness 0.42 cm increases compared to other thicknesses.

D.C. Conductivity: The measurements of resistivity of samples such as ethyl cellulose, parkia powder agar and its blends with 50 and 80 weight percentages of ethyl cellulose are carried out using Kiethly source meter for different temperatures over the range 45-100 ºC. The experimentally measured values of D.C. conductivity and the activation energies of the ethyl cellulose, parkia powder agar and its blends with ethyl cellulose are calculated using Arrhenius equation (4).

$$\sigma_{dc} = \sigma_0 \exp - \frac{E}{k}\left(\frac{1}{T} - \frac{1}{T_0}\right) \quad (4)$$

where σ_{dc} and σ_0 are the conductivities at temperatures T and T_0, respectively, k is the Boltzmann constant and E is the corresponding activation energy. The plot of ln (σ_{dc}) *versus* 1000/T and is given in Fig. 13. The data is fitted for straight line using least square fit. The values of slope of fitting parameters are used to calculate the activation energies (E) and are listed in Table-1. It is observed that as thickness of the samples increased the activation energy decreases. Fig. 13 showed that the D.C. conductivity of pure samples of parkia powder agar and ethyl cellulose are more or less same, but in case of its blends with 50 wt % in each shown increase in its conductivity than those of pure samples. In case of 80 wt % of ethyl cellulose with parkia powder agar shown decrease in its conductivity. It is also noticed that the D.C. conductivity is decreased as temperature increases for all the samples.

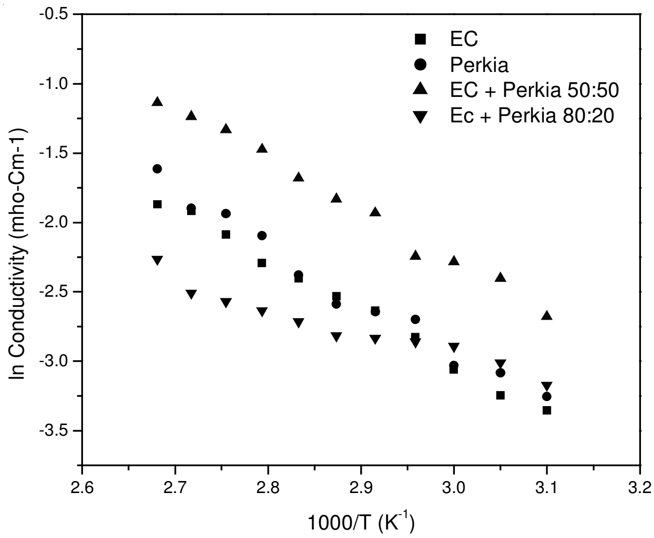

Fig. 13. Plot of ln (σ_{dc}) *versus* 1000/T of parkia powder agar (PPA) and its blends

TABLE-1
ACTIVATION ENERGIES OF THE ETHYL CELLULOSE, PARKIA POWDER AGAR AND ITS BLENDS

Sample	Thickness (cm)	Activation energy (eV)
Parkia powder agar	0.384 0.429	0.659 0.332
Ethyl cellulose	0.429 0.537	0.792 0.410
Blend of parkia powder agar with ethyl cellulose	0.429 0.654	0.321 0.152

Conclusion

The XRD spectrum of the blend of parkia powder agar with ethyl cellulose shows diffuse peak of broader area implies the particle size is very small. The SEM images, shows the crystallites possess diffusive surface for reflectance and also the micro scale image exhibits formation of the gel type polymerization. The capacitance of the parkia powder agar decreases exponentially at lower frequency ranging from 100 Hz to 10 KHz; afterwards it decreases gradually and remains constant at higher frequencies. On the other hand, it is observed that as thickness of parkia powder agar sample increases the values of dielectric constant also increases appreciably with in the frequency range100 Hz-1 KHz. In case of blends of parkia powder agar with ethyl cellulose the dielectric constant decrease as the weight per cent of ethyl cellulose increases.

Further, the A.C. conductivity of parkia powder agar remains constant up to 10 KHz and increases at frequency 300 KHz onwards. In case of D.C. conductivity it is noticed that as temperature increases the D.C. conductivity decreases, for all the samples. It is also observed that as thickness of the sample increases the activation energy decreases.

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