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Electrical Dielectric Authorship of Polyvinyl-Acetate and Toluene Diisocyanate (PVA-TDI) with Manufactured Sulfonated Phenol-formaldehyde (SPF) Viscous Mass Material Composite

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Abstract:

The electrical insulation of the manufacture sulfonated phenol-formaldehyde viscous material (product) has been studied with Polyvinyl-acetate (PVA) and toluene diisocyanate (TDI) blend has been prepared by fixing percentage by weight 3:1 and mixed with different percentages by weight of the product sulfonated phenol formaldehyde viscous mass (SPF). The Fourier transform infrared (FTIR) spectroscopy is done on (SPF) resin powder and prepared film of PVA-TDI-SPF viscous mass. The quality factor (Q), dissipation factor (D), parallel resistance (R_p), series resistance (R_s), parallel capacitance (C_p), series capacitance (C_s) and phase shift (ϕ) are measured. The calculated maximum dielectric constant (ε') is $3.49x10^7$ at sample (1) wt.1% SPF viscous mass to the weight of (PVA-TDI), the minimum dielectric constant is $1.12x10^6$ at sample (3) wt.3% of SPF viscous mass to PVA-TDI weight. The maximum dielectric loss factor (ε'') is $3.68x10^7$ at sample (1) and the minimum dielectric loss is $2.04x10^6$ for sample (3). The maximum conductance is $1.06x10^{-4}$ S at sample (1) and minimum conductance is $6.64x10^{-6}$ at sample (3). The maximum frequency dependent ac. conductivity (σ_{ac}) is 2.048 S m^{-1} for sample (1) and the minimum is 0.113 S m^{-1} at sample (3). The maximum total conductivity (σ_t) is 126.2 S m^{-1} for sample (1) and minimum (σ_t) is 1.129 S m^{-1} for sample (3). The maximum independent conductivity (σ_{dc}) is 124 S m^{-1} for sample (1) and minimum value is 1.015 S m^{-1} for sample (3). The maximum capacitive reactance (X_s) is 0.83 MΩ at sample (5) wt.5% SPF viscous mass to PVA-TDI weight and the minimum is 0.14 MΩ for sample (3).

Key words: Mixture, Electrical Insulators, Conductivity, Capacitive Reactance.

Introduction:

Dielectrics are class of materials that are highly resistive to electrical current. A few simplified definitions of dielectric own qualities are important for the meaningful discussion of their measurements and applications. They have been defined previously in terms of electrical circuit concepts and electromagnetic field concepts. Permittivity (ε) can be represented by a complex quantity relative to the permittivity of free space ε_o. The real

part ε′ is the dielectric constant that is a direct measure of amount of energy can be stored in a material in the form of electric field in the material. The imaginary part ε″ is the loss factor that is associated with ability of the material to absorb or dissipate energy that converts electric energy into heat energy[1]. The distinct qualities of polymers depend upon several factors like molecular weight, chemical nature of the units composing the polymer and morphology in solid state. Generally the dielectric characterization of polymers is studied as a function of the degree of polymerization, frequency, temperature and pressure[2]. The physical structure of polymer composites in the solid or viscoelectric state is of great importance in determining the dielectric behavior. The dielectric own quality of polymer composite materials have been studied with a view to modify the own quality of polymer system for practical applications. The inorganic insulators and dielectrics have been largely replaced by polymers on account of their quality and ability for specific needs. Epoxides, and polyesters have been used in electronics as insulators, dielectrics substrates, potting compounds[3]. In order to present unambiguous picture of the dielectric behavior of a such a system, a careful analysis and evaluation of the two major dielectric parameters namely dielectric constant (representing polarization) and tangent of dielectric loss angle (representing relaxation phenomena) is desirable [4]. A material is classified as "dielectric" if it has the ability to store energy when an external electric field is applied. It is well known that composites can be produced exhibiting enhanced behaviors that the constituent materials may not exhibit. 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 nature in the use of capacitors[5]. Phenol formaldehyde (PF) adhesive is widely utilized in various industrial fields for its advantages such as high bonding strength, good chemical stability and resistance of water, heat and wear. With the development of economy, further improvement of its quality and performance is required. Therefore many researchers have carried out the modifications of PF with different methods[6]. Fourier transform infrared spectrometry (FTIR) spectroscopy has been employed widely to examine microscopic areas in polymers for the last twenty years. The technique has attained high precision and accuracy in measurements in researches and development purposes[7]. FTIR spectroscopy could be a possible option is used to obtain functional group information of SPF and functional group of PVA-TDI-SPF. The investigation represents the dielectric measurement carried out on PVA-TDI-SPF mixture at frequency 1 kHz, and laboratory temperature, and to rationalize the dielectric own quality of PVA-TDI-SPF. The effect of different percentage by weight of SPF viscous mass on the electrical dielectric of PVA-TDI gives a significant results of Gs, ε′, ε″, σ_{ac}, σ_{dc}, σ_t and capacitive reactance.

Materials and Methods:

A-Manufacture of Sulfonated Phenol-Formaldehyde Resin (SPF) 42.5 moles of phenol, Thomas Baker, India, has been put in a clean bottom round flask 500 ml. in capacity and emplaced in Isomental heater sort LabHeat BAECO, Germany. A stop-fit thermometer and a condenser are connected at both sides of the bottom round flask, and stirrer sort Heidolph, Germany, are used with rubber to close the middle of the bottom round flask and operating the system. Set up of the system is shown in Figure (1). The

system is operated and the phenol is heated to appropriate temperature and mixing to dissolve any solid bodies. The system is stopped and 4 moles of sulfuric acid 97% in concentration Thomas Baker India, are added slowly to the dissolved phenol, from the stop-fit thermometer side by using pipettes and the bottom round flask is closed again, the system is operated, the stirrer adjusted to an appropriate speed on scaling 2 measure 6 (800 1/min.rpm) and the temperature is raised, and maintained between 100-120 ^{o}C and the reflux has been done for two hours. The system is stopped and the temperature is left to cool slowly, then the bottom round flask is emplaced in an ice path and 12 moles of Formaldehyde, Thomas Baker, India; has been added by using pipettes, a fizzing and bubbling is occurred, the temperature is raised and stirred by hand using clean glass then the temperature is cooled to 35 ^{o}C then below 22 ^{o}C. Continuous stirring is done until a viscose solid mass is obtained; the product is left over night. The PH is examined by using indicator paper which is colored red, low PH. NaOH solution has been prepared in a separate flask and drops are added until over saturation is reached by the high PH, a few drops of H_2SO_4 have been added for equilibrium until PH=7 is reached. The solution has been removed into clean plastic container this is a viscous mass of SPF and the precipitate (SPF) resin is put in a glass plate to be dried at room temperature that is collected in plastic container.

B-Fourier Transform Infrared (FTIR) Spectroscopy:

FTIR Spectroscopy of Sulfonated Phenol-Formaldehyde resin (SPF):

A sample of manufactured sulfonated phenol-formaldehyde resin, is crushed by stainless steel mortar, is examined with kBr disc by Fourier transform Infrared (FTIR) JASCO FTIR 4200 spectrophotometer serial No. C081761018, Japan as in Figure (2). The peaks at 1128.39 cm^{-1}and 1175cm^{-1} correspond to C-C-O asymmetric stretch and C-H in plane formulation respectively while the 1000 cm^{-1} and 748.8 cm^{-1} peak belong to the C-H out of plane vibration. The peak at 1506.37 cm^{-1} correspond to the C=C aromatic ring vibration. The above mentioned peaks diminish with increasing reaction time while the absorbance band of hydroxyl groups increase. Peak at 1034.14 cm^{-1} is the stretching vibration of C-O[8] and band at 3503.3 cm^{-1} is stretching vibrations of OH, Guo, et al[6]. Table (1) indicates the comparison between the obtained results with previous study.

FTIR Spectroscopy of PVA-TDI-SPF:

A sample of polyvinyl acetate with Toluene diisocyanate is blended by percentages 3:1 by weight and mixed with wt.1% of the manufactured sulfonated phenol-formaldehyde viscous mass is prepared on clean glass substrate and left to dry in room temperature for two weeks. The sample is crushed by ceramic mortar and examined with kBr by Fourier Transform Infrared Instrument JASCO FTIR 4200 spectrophotometer serial No. C081761018, Japan as in Figure (3). The spectra are taken from 400 to 4000 cm^{-1}. The peak at 843.222 cm^{-1} and wave number 946.395 cm^{-1} is C-H out of plane phenol. The wave number at 1116.1 cm^{-1} is C-H in plane. The wave number at 1252.06 cm^{-1} is C-C-O phenol, Poljansek, [8]. The band at 3308.29 cm^{-1} is the hydroxyl group[9]. Band at 1739.48 cm^{-1} is assigned to free carbonyl, while a lower frequency at 1651.73 cm^{-1}[10].

C- Samples Preparation

Glass substrates are cleaned by rinsing for several times with distilled water then by acetone until they dried, again with distilled water for several times. The glass substrates are dried in furnace under vacuum for one hour. Percentage 3:1 of PVA: TDI by weight is prepared using sensitive electric balance sort Sartorius, Germany and blended on the clean glass substrates. The prepared sulfonated phenol-formaldehyde (SPF) viscous mass is added in percentages by weight (wt.1%, wt.2%, wt.3%, wt.4% and wt.5%) to the blend and the compound is mixed by hand using thin glass rod and spatula until homogeneous mixtures are obtained and to ensure uniform thicknesses. Two copper wires are connected at both ends of bulk samples, which are left to dry overnight. The thickness and electrode diameters of the samples are measured using Vernier Certificate made in China, as shown in Table (3). The samples undergo dielectric measurement.

D-Dielectric Measurement

Dielectric measurements have been done by using LCR meter; sort FLUKE RCL Meter, Automatic 6303 MP, No. Lo 781003, Germany. The measurements include the electric parameter, the quality factor (Q), dissipation factor (D), impedance (Z), (Rp), (Rs), (Cp), (Cs) and (ϕ). The calculations of dielectric constant (έ), dielectric loss (ε̋) ac conductivity (σ_{ac}) and dc conductivity (σ_{dc}) have been calculated from the formulae below, Table (4) shows the measurements and calculated values. The dielectric constant (έ) and dielectric loss (ε̋) of samples are calculated using the following relations. The dielectric constant is given by the relation [11]:

$$\varepsilon' = \frac{Cs\ d}{\varepsilon o\ A} \ldots.(1)$$

Where C_s the series capacitance, ε_o is the permittivity of free space and equal to $8.83x10^{-12}$ F and d is the sample thickness in m. The dielectric loss is given by $\varepsilon'' = \frac{\varepsilon'}{Rp\ Cp\ W}$ … (2)

Where Rp is the parallel resistance in MΩ, C_p is the parallel capacitance in Pf and w is the angular frequency is equal to 2πf, where f is the frequency in Hertz. The capacitance of free space C_o is given by the equation

$$Co = \frac{\varepsilon o\ A}{d} \ldots(3)$$

Where the dimensions have been measured by micrometer, the diameter of the electrode is $0.07x10^{-3}$ m, $A = \frac{\pi D^2}{4}$ is the area of the electrode is equal to $3.846x10^{-9}$ m^2. and d is the thickness of the electrode $0.07x10^{-3}$ m. the calculated Co is equal to $4.863x10^{-4}$ pF

The frequency dependent conductivity can be calculated by the formula[12]:

$$\sigma_{ac} = \varepsilon''\varepsilon o\ w \ldots (4)$$

The conductance is given by the formula:

$$Gs = \varepsilon_o\ C_o\ w \ \ldots(5)$$

The total conductivity is given by the formula;

$$\sigma = Gs\frac{d}{A} \ \ldots (6)$$

The total conductivity is also given by the relation:

$$\sigma_t = \sigma_{ac} + \sigma_{dc} \ldots (7)$$

Where σ_{dc} is independent frequency dc conductivity

The complex impedance Z is given by the relation:

$$Z = Rs + Xs \ \ \ldots (8)$$

Where Rs is the equivalent series resistance and Xs is the capacitive reactance is given by

$$Xs = \frac{1}{2\pi fCs} \ \ \ldots (9)$$

Results and Discussion:

Table 1 Comparison between the Obtained Results with Previous Study of SPF Resin

Literature Data cm^{-1}Ref. No.5	Functional Group Obtained	Wave number cm^{-1}	Functional Group. Ref.	Observed cm^{-1}
1240	C-C-O	1200	C-C-O phenol	1224 and 1262
1180	C-H in plane	1175	C-H aromatic phenol	1170
835	C-H out of plane phenol	1000 and 748.8	C-H out of plane, para	826
760		750	C-H out of plane, ortho	756.6
1153	C-O	1034.14	C-O stretch	1154 and
----------	C-H unsaturated	3024.43	C-H unsaturated stretched phenol	3026
1610 and 1517	C=C	1506.37	C=C	1610 and 1552
3400	OH	3530.3	OH	3389 and 3466

Table 2 Comparison between Obtained Results and Reference Results of PVA-TDI-SPF

Functional Group Obtained	Wave number cm^{-1}	Functional Group Reference No.5 and 9	Wave number cm^{-1}
C-H out of plane phenol	843.222 and 943.365	C-H out of plane para	826
C-H in plane	1116.1	C-H aromatic phenol	1170
C-C-O phenol	1252.06	C-C-O phenol	1224
Free Carbonyl	1739.48	Free Carbonyl	1727
Low frequency Carbonyl	1651.73	Low frequency Carbonyl	1689
OH	3308.29	OH	3350

Table 3 Samples Preparations and Dimensions

Samples No.	PVA Weight gm.	TDI Weight gm.	Percentages SPF viscous mass	Sample Length mm	Samples Width mm.	Samples Thickness mm.	Effective Area of Electrode mm^2.
1	0.06	0.02	1%	12.2	5.7	1.8	$1.519x10^{-3}$
2	0.06	0.02	2%	14	9	0.9	$1.519x10^{-3}$
3	0.06	0.02	3%	12.06	6.4	0.875	$5.15x10^{-3}$
4	0.06	0.02	4%	12.2	5.3	1.9	$4.654x10^{-3}$
5	0.06	0.02	5%	14.2	7.5	0.9	$4.654x10^{-3}$

Table 4 Dielectric Measurements and Calculations

Sample No.	Q	D	Rp MΩ	Rs MΩ	Z MΩ	Cp Pf	Cs Pf	ϕ Deg.	ε′
1	0.689	1.63	2.276	1.034	2.075	66.3	261	-44.4	$3.49x10^{7}$
2	0.847	1.09	2.11	16.70	38.2	62.4	168	-41.5	$1.128x10^{7}$
3	0.691	1.71	1.995	1.355	1.40	85	113	-53.2	$2.17x10^{6}$
4	0.726	1.21	1.876	1.238	1.604	62.2	145	-48	$6.68x10^{6}$
5	0.931	1.01	1.695	1.232	1.182	90.2	191	-43.3	$8.828x10^{6}$
Sample No.	ε″	Gs S	σac S m^{-1}	σt S m^{-1}	σdc S m^{-1}	Xs MΩ			
1	$3.68x10^{7}$	$1.06x10^{-4}$	2.048	126.2	124	0.61			
2	$1.36x10^{7}$	$3.43x10^{-5}$	0.755	20.35	19.6	0.94			
3	$2.04x10^{6}$	$6.64x10^{-6}$	0.113	1.129	1.015	0.14			
4	$9.12x10^{6}$	2.0410^{-5}	0.507	8.336	7.829	1.09			
5	$9.38x10^{6}$	$2.69x10^{-5}$	0.521	10.99	10.47	0.83			

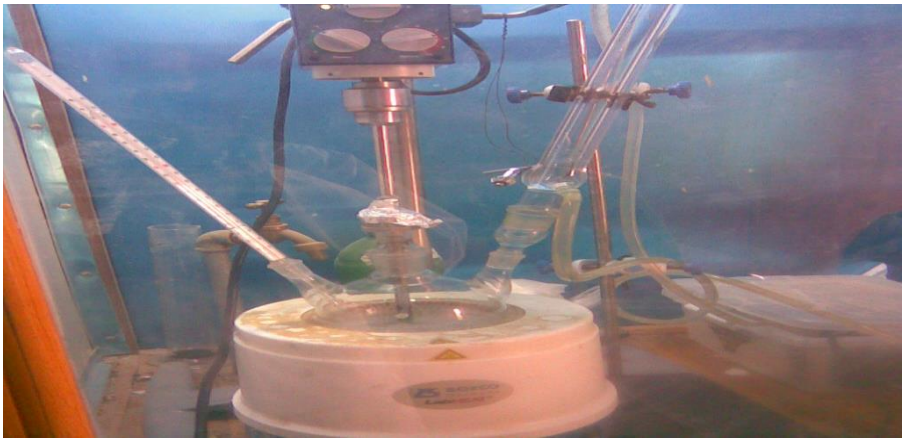

Fig.(1). The Setup of Instruments Used in Manufacture of SPF

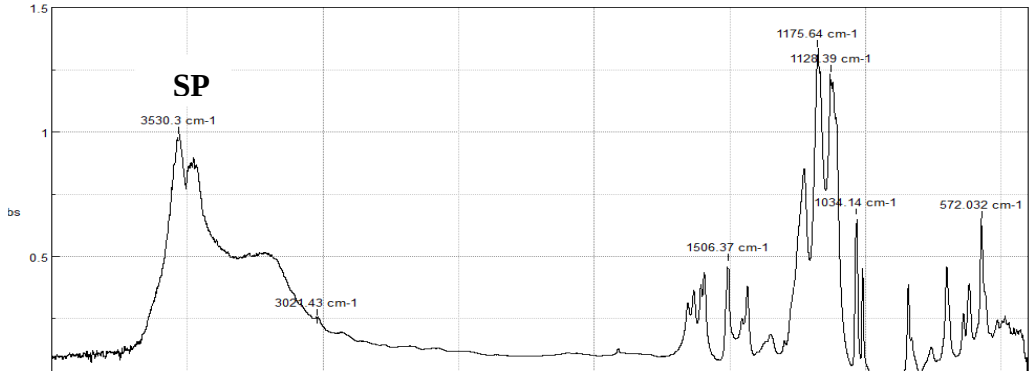

Fig.(2). The FTIR Spectroscopy of SPF.

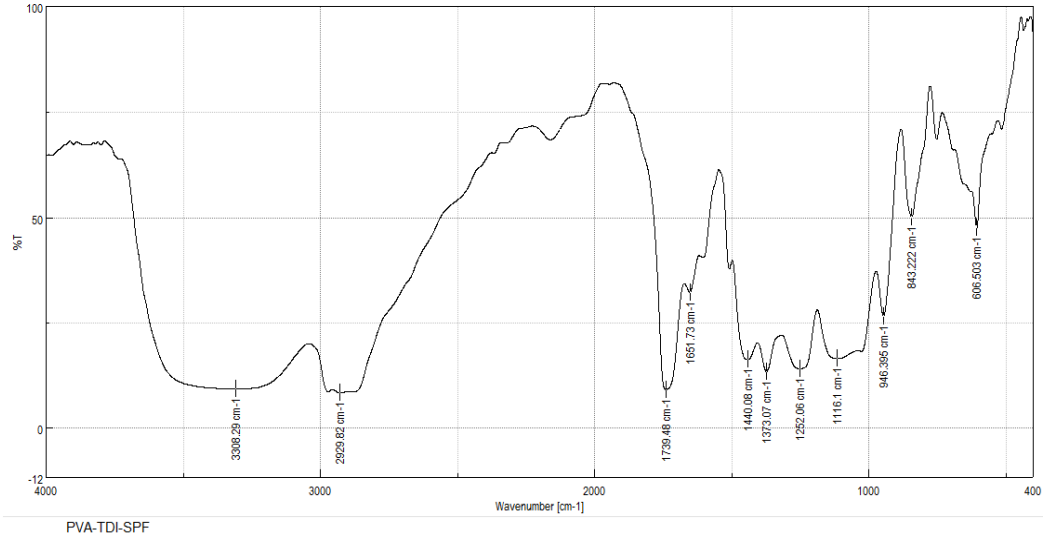

Fig.(3). FTIR Spectroscopy of PVA-TDI-SPF Mixture

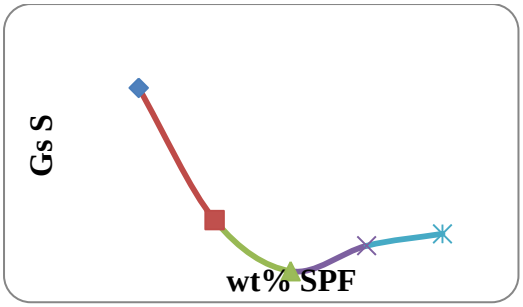

Fig.(4). Conductance Variation with wt.% SPF

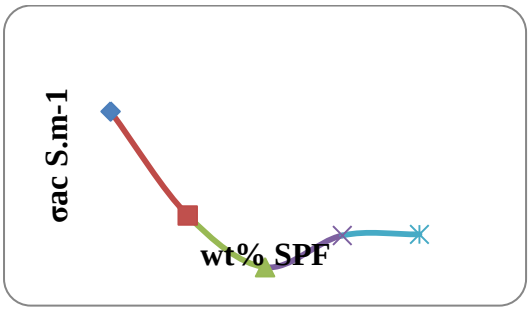

Fig.(5). TheDependence of ac Conductivity on wt.% SPF

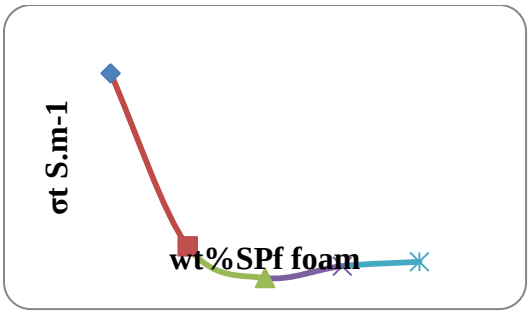

Fig.(6). The Dependence of Total Conductivity on wt.% SPF

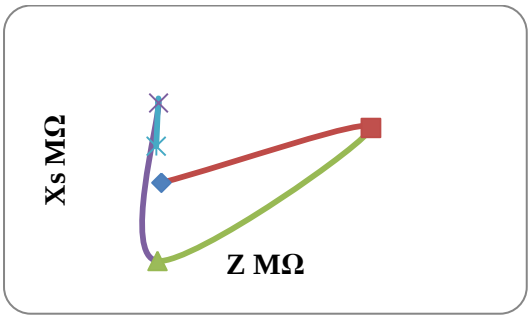

Fig.(7). Impedance vis Reactive Capacitance

FTIR spectroscopy of SPF resin is shown in Figure (2) that indicating functional groups are obtained, such as the C-C-O asymmetric and C-H in plane formulation and out of the plane vibration. Table (1) shows the comparison between the obtained result and the results in literature. Figure (3) indicates the functional groups of PVA-SPF mixture and Table (2) indicates the comparison of the obtained results with previous studies, for example, the peaks at 843.222 cm^{-1} and 946.395 cm^{-1} are out of plane phenol. The peak at 1116.1 cm^{-1} is C-H in plane. Table (3) indicates the samples preparations used in dielectric measurements and calculations. From Table (4), Figure (4) indicates the variation of conductance with wt.% of SPF viscous mass to PVA-TDI weight, the conductance decrease with increasing wt.% SPF viscous mass to PVA-TDI weight, the minimum decrease is at wt.3% SPF viscous mass to PVA-TDI weight to the value $6.64x10^{-6}$ S, sample (3). This can be explained by a specific conductance mechanism of these materials, where even a certain number of conducting paths are present, due to the drop in voltage, more electrons are activated causing an increasing in current. [13]. The maximum value of conductance is $1.06x10^{-4}$ at sample (1) wt.1% SPF viscous mass to PVA-TDI weight. Figure (5), the dependence of the frequency dependent ac conductivity (σ_{ac}) on wt.% SPF viscous mass the maximum value of (σ_{ac}) is 2.048 S m^{-1} at sample (1) and the minimum value is 0.113 S m^{-1} at sample (3). The frequency dependent of PVA-TDI-SPF samples at 1 kHz it is distinctly observed that the dielectric constant ε' and dielectric loss ε'' are decreased. Figure (6), the dependence of the total conductivity (σ_t) on wt% SPF viscous mass to PVA-TDI weight. The maximum value of (σ_t) is (126.2) S m^{-1} at sample (1) and minimum value is (1.129) S m^{-1} at sample (3). At sample (2) wt.2% SPF viscous mass to PVA-TDI weight the value is (20.35) S m^{-1}. The maximum value of frequency independent dc conductivity is 124 S m^{-}

^{1} for sample (1) and the minimum is 1.129 S m^{-1} for sample (3). Figure (7), the impedance vis capacitive reactance, the curve is rather alternative of the capacitive reactance with impedance of samples according to their wt% SPF viscous mass to PVA-TDI weight. There are two major factors responsible for the contact resistance magnitude: geometry and insulating layer (potential barriers) between the contact surfaces. The resistance of the contact is inversely proportional to its area and is independent on the surface stiffness, force holding the two surfaces together. [14].

Conclusions:

The conductance of the samples shows little decrease with increasing wt.% SPF viscous mass of PVA-TDI weight except at sample wt.3% SPF viscous mass of PVA-TDI weight.

The frequency dependent conductivity (σ_{ac}) shows increase with increasing wt.% SPF foam of PVA-TDI weight. The independent frequency dc conductivity (σ_{dc}) reaches the maximum values compared with the frequency dependent ac conductivity (σ_{ac}). The maximum value of total conductivity is obtained at sample wt.1% SPF viscous mass of PVA-TDI weight to the value 126.2 S m^{-1}.

The increase of SPF viscous mass, first conducting contact between the fibres are formed compared with composites, in this time a low conductivity at wt.3% SPF viscous mass of PVA-TDI weight to the value 1.129 S m^{-1}, this increase only slowly further, indicates only a small number of contacts between fibres in the composites at a content of several percent of SPF viscous mass.

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ايجاد العزلية الكهربائية لمكون الأسيتيت متعدد الفاينيل و تلوين داي أيسوسيانيت مع تصنيع كتلة لزجة لمادة الفينول-فورماليهايد المسلفن

أ.م ثامر سلمان بجاري

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الخلاصة:

تم ايجاد العزلية الكهربائية لمادة لزجة من الفينول فورماليهايد المسلفن التي تم تصنيعها (المنتج)، مع نسبة مثبتة 1:3 من الأسيتيت متعدد الفاينيل و التلوين داي أيسوسيانيت التي تم خلطها مع نسب مختلفة من المنتج. تم دراسة تحويلات فورير للأشعة تحت الحمراء لكل من مسحوق راتنج الفينول الفورماليهايد المسلفن ومزيج الأسيتيت متعدد الفاينيل مع التلوين داي أيسوسيانيت و المادة اللزجة من الفينول-فورماليهايد المسلفن. ودراسة العزلية الكهربائية للأستيت متعدد الفاينيل و تلوين داي ايسوسيانيت والمنتج من مادة لزجة للفينول-فورماليهايد المسلفن. تم قياس عامل النوعية، عامل الفقد، مقاومة التوازي و مقاومة التوالي، الممانعة، سعة التوازي، سعة التوالي و أنحراف الطور. تم حساب ودراسة ثابت العزل ووجد اعلى قيمة له $3.68x10^7$، للنموذج (1) 1% من وزن المنتج لوزن الأسيتيت متعدد الفاينيل و تلوين داي ايسوسيابنيت و أوطأ قيمة $1.12x10^6$ للنموذج (3) 3% من وزن المنتج لوزن الأسيتيت متعدد الفاينيل و تلوين داي أيسوسيانيت، أعلى قيمة لفقد العزل $3.68*10^7$ للنموذج (1) و أوطا قيمة $2.04x10^6$ للنموذج (3). أعلى مواصلة $1.06x10^{-4}$ سيمنز، للنموذج (1) و أوطأ مواصلة $6.64x10^{-6}$ سيمنز، للنموذج (3). أعلى توصيل معتمد على التردد 2.048 سيمنز م$^{-1}$ للنموذج1ج (1) و أوطأ قيمة 0.113 سيمنز م$^{-1}$، للنموذج (3) 3. أعلى توصيل كلي 126.2 سيمنز م$^{-1}$ للنموذج (1) و أوطأ قيمة 1.129 سيمنز م$^{-1}$ للنموذج (3). اما أعلى توصيل غير معتمد على التردد 124 سيمنز م$^{-1}$ للنموذج (1) و أوطأ قيمة 1.015 سيمنز م$^{-1}$ للنموذج (3). أعلى مفاعلة سعوية 0.83 ميكا أوم للنموذج (5) 5% من وزن المنتج لوزن الأسيتيت متعدد الفاينيل و التلوين داي أيسوسيانيت و أوطأ قيمة 0.14 ميكا أوم للنموذج (3).

الكلمات المفتاحية: مزيج، العزل الكهربائي، التوصيل، المفاعلة السعوية