

Study the effect of kaolin on the electrical properties of polyvinyl acetate-polyol composite

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دراسة تأثير الكاولين على الخصائص الكهربائية لمكون الأسيتيت متعدد الفيناييل -
بولي اول

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المستخلص

تم تحضير نماذج من مزج اسيتيت متعدد الفيناييل والبولي اول بنسبة وزنية ثابتة 3:1 و خلط المزيج مع نسب وزن مختلفة للكاولين للقياسات الكهربائية. تم تشخيص الكاولين بواسطة مطياف تحويلات فورير للأشعة تحت الحمراء. قياسات عامل النوعية, عامل الفقد, مقاومة التوازي, مقاومة التوالي, الممانعة, سعة التوازي, سعة التوالي وزاوية الطور. تم حساب التوصيل الكهربائي ووجدت أعلى قيمة لها 6794 سيمنز.م⁻¹ للنموذج (1) 3% لوزن الكاولين وأوطأ قيمة لها 10x10⁻⁵ سيمنز.م⁻¹ للنموذج (2) 5% لوزن الكاولين. تم دراسة و حساب ثابت العزل وفقد العزل. تم دراسة وحساب التوصيل المتناوب المعتمد على التردد والتوصيل المستمر غير المعتمد على التردد. تم حساب أوطأ وأعلى قيم التوصيل المتناوب المعتمد على التردد 10x3.2⁻⁵ سيمنز.م⁻¹ للنموذج (3) 6% من وزن الكاولين 0.45 سيمنز م⁻¹ للنموذج (2) 5% لوزن الكاولين. أوطأ واعالى قيم للتوصيل المستمر غير معتمدا على التردد -0.3472 سيمنز م⁻¹ للنموذج (2) 5% لوزن الكاولين و 1.1672 سيمنز م⁻¹ للنموذج (1) 3% لوزن الكاولين.

Abstract

Polyvinyl acetate and polyol (PVA-Polyol) has been blended in percentage by weight 3:1 and a mixture has been done by mixing different percentages by weight of kaolin for electrical measurements. Diagnosis of kaolin was measured by FTIR spectrophotometer. Quality factor (Q), dissipation factor (D), parallel resistance (R_p), series resistance (R_s), impedance (Z), series capacitance (C_s) and parallel capacitance (C_p) and Phase angle (Φ) were measured. The maximum electrical conductivity is found 6794 S.m⁻¹ for samples 1, 3wt% kaolin and the minimum electrical conductivity is 1x10⁻⁵ S.m⁻¹ for sample (2) 5wt% kaolin. Dielectric constants (ϵ'), dielectric loss (ϵ''), have been studied and calculated. The frequency dependent ac conductivity (σ_{ac}) and frequency independent dc conductivity (σ_{dc}) has studied and investigated. The calculated minimum and maximum ac conductivity (σ_{ac}) is 3.2x10⁻⁵ S.m⁻¹ for samples (3) 6wt % kaolin and 0.45 S.m⁻¹ for sample (2) 5wt % kaolin. The minimum and maximum calculated independent frequency dc conductivity (σ_{dc}) is -

0.3472 S.m⁻¹ for sample (2) 5wt % kaolin and 1.1671 S.m⁻¹ for sample (1) 3wt % kaolin.

Keywords: Composites; FTIR spectroscopy; electrical properties; conductivity.

Introduction

Polyvinyl acetate (PVA) is one of the most important commercial polymers, and has many advantages, such as safe operation, low cost and room temperature cure. Therefore, it is suitable for population in birch process. It has other applications such as adhesives, membrane, paper and many other applications (1). PVA is a synthetic resin polymer and due to its non-polar nature, is insoluble in water, oil, fats or gasoline. On the other hand, it is soluble in alcohols, ketones and esters. Kaolinite is one of the abundant aluminosilicate minerals, occurring primarily as clay sized particles with high surface-area to volume ratios. Hence kaolinite weathering may play an important role in controlling the chemical characteristics such as degree of crystalline, concentration of impurities and particles size distribution. The dispersion of the particles differs with particle size as the content is the same. The average inter-particle distance reduces with decrease of particle size afford more connected with the composites (2). Attempts of spectroscopic characterization are still in criterion of study. Clay is widely utilized for different industrial applications and as such any of occurrences is worth proper chemical, mineralogical and technological investigations (3). It is well known that composites can be

produced exhibiting enhanced properties that the constituent material may not exhibit. For instance, from the combination of different fibres or fillers with polymer matrices one can produce polymer-matrix composites, a material important to the electronic industry for its dielectric properties in use of capacitors. 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 polymer matrix. The electrical response of a normal dielectric can be described by its conductance, dielectric constant and loss factor (4). The electric measurements of composites with reinforced fiber were considered the effect of frequency, fiber content and fiber length. The dielectric constant increased steadily with increasing fiber content for all frequencies in the range of 1 to 10⁷ Hz. Authors also noted that the dielectric constant was decreased with increase of fiber length and frequency. The composite with 1mm fibers and 30% fiber content had the highest value of dielectric constant (5). Conducting polymeric materials possess great design flexibility together with a number of characteristics that are desirable for a number of specific applications in the

field of catalyst, conversion and storage of energy, chemical and biochemical sensing, microelectronics and optoelectronics. However, experiments have also indicated some general limitations of these materials with respect to the fabrication of devices. Several methods have been proposed to prepare polymer composites, such as sol-gel reaction, interactive polymerization and polymerization via melt processing, depend upon the nature of nanoparticles and types of polymeric matrix. The final properties of these composites depend upon various parameters like size of particles, method of preparation of composites and dispersion of particles into the polymeric matrix (6). The design of polymer composites requires materials that can improve their electrical performance. Polymer filled with non-sized conductive filler can make functional polymer composites. In addition, the attached functional groups may enhance the interfacial interaction between the polymer matrix and the filler (6).

Experimental work

FT-IR test

10 mg of Kaolin was Grinded by ceramic mortar to a fine powder with Kbr, this undergoes FT-IR test by JASCO FTIR 4200 spectrophotometer, Japan. The spectra were indicated at the wave number 400-4000 cm^{-1} as shown in Figure (1). The infrared spectra of kaolin minerals consist of a sheet of corner-sharing tetrahedral, sharing a plane of oxygen and hydroxyls (inner hydroxyls) with a sheet octahedral with every third site

An I-V measurements is a task to obtain the current vis voltage or resistance characteristics by providing a voltage/current stimulus and measuring current/voltage reaction. It is a basic electric measurement and fundamental way to discover behavior and characterize the devices such as semiconductors (ICs, memory, MOS FETs, bipolar transistors, etc.), components (LEDs, sensors, resistors, etc.), new materials (carbon nanotube, Graphene, Nanowire, GMR, organic devices, etc.) and other electronic devices (photovoltaic cell, electric circuit, etc.). According to the trends in electronics strongly demanding more advanced features, lower power consumption and lower cost for next generation devices. It is becoming increasingly important to perform more accurate and precise low-current, low-voltage or low-resistance measurements quicker than ever for the research and development of next-generation devices and their timely deployment (7).

vacant (dioctohedral) (8). A typical spectrum of kaolin at 3437.49 cm^{-1} , this is a characteristics band H-O-H stretching absorbed water. The band observed at around 3437.49 cm^{-1} with band 1650.77 cm^{-1} could be assigned to the OH vibrationally mode of the hydroxyl molecule, this is observed in almost the natural hydrous silicate. The H-O-H bending of water is observed at 1650.77 cm^{-1} . C-H stretching 1434.78 cm^{-1} region (9). Main functional

groups were Si-O and Al-OH libration t 935 cm^{-1} . Si-O out of plane stretch 1109 cm^{-1} (10). Si-O stretching could be observed at 1017.03 cm^{-1} . 799.108 cm^{-1} and 780 cm^{-1} is due to Si-O-Si

inter tetrahedral bridging bonds in SiO_2 and OH. A compression between the obtained results and previous studied as show in Table (1).

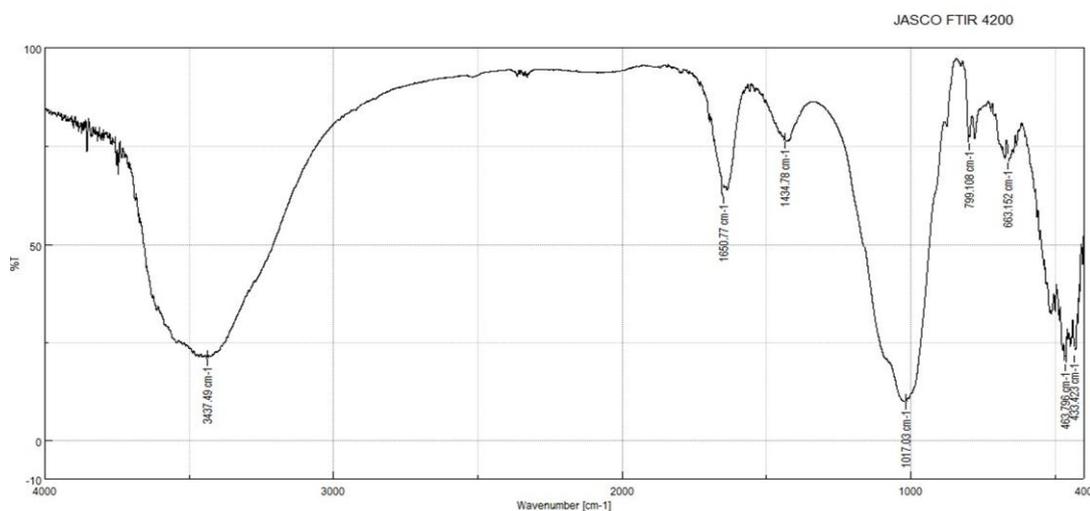


Figure (2): FT-IR test of Kaolin

Table (1): FTIR spectroscopy of kaolin compared with previous study.

Obtained results wavenumber cm^{-1}	Functional group	Previous study wavenumber cm^{-1}	Functional group observed
3437.49	H-O-H stretching, Absorbed water	3440-3432	H-O-H Stretching Absorbed water
660.152	Si-O Quartz	693-696	Si-O quartz
1650.77	H-O-H bending of water	1642-1632	H-O-H bending of water
1434.78	C-H stretching	1470-1475	C-H stretching
1017.03	Si-O out of plane stretch	1109	Si-O out of plane stretch
799.108-780	Si-O quartz	788-799	Si-O quartz
463.796-433.423	Si-O-Si bending	470-467	Si-O-Si bending
935	Al-OH libration	915 and 935	Al-OH libration

Sample preparation

Glass substrates have been put in flask and have been cleaned with distilled water and rinsed with ethanol for five minutes, and washed again with distilled water, the glass substrates have been put in oven for half hour to be dried. The oven switched off to room temperature and the glass substrates have been taken out of the oven to be ready for use. Kaolin (Available in local market) has been grinded by glass mortar and has been sieved for different particles size. Percentages by weight 3:1 of polyvinyl acetate and polyol were blended on the clean glass substrates by using spatula

until the blend become homogeneous. Percentages by weight of prepared kaolin have been dropped on the blend for each sample. The mixtures have been prepared to suitable shapes by spatula to obtain significant dimensions bulk samples as shown in Table (2). Two copper wires were connected at each side of the sample and left over night to be dried (11). The samples were undergoing electrical measurements.

Electrical measurements

The current-voltage was measured at 10 volt dc. Figure (2) shows a schematic diagram of the electrical circuit. The samples have been put inside enclosure of wood box with front slide window. The samples

$$\rho = \frac{RA}{\tau} \quad (1)$$

$$\sigma = \frac{\tau}{RA} \quad (2)$$

Where: ρ is the electrical resistivity of the samples, R is the electrical resistance of the sample, τ is the thickness of the samples,

dimensions were measured by Vernier Capilar made in China as shown in Table (2). The resistivity and the conductivity have been calculated by the following expressions (1).

A is the effective cross sectional area of the sample this is equal to $\pi D^2/4$,

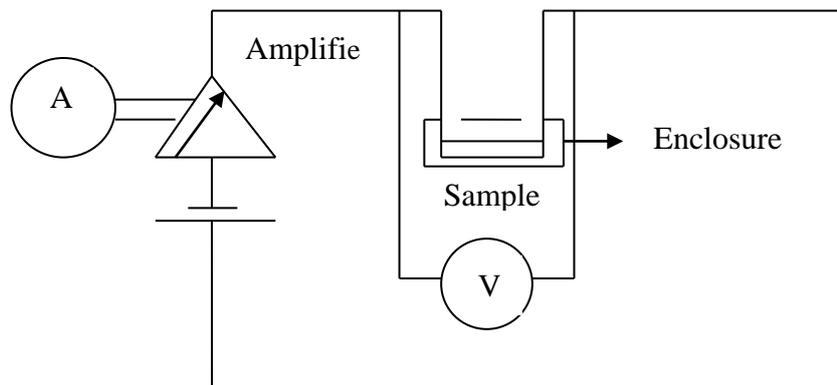


Figure (1): Schematic diagram of the electrical

The dielectric measurements have been done by LCR meter; sort FLUKE RCL Meter, Automatic 6303 MP, Germany. The measurements include the quality factor (Q), dissipation factor (D), impedance (Z), parallel resistance (R_p), series resistance (R_s), parallel capacitance (C_p), series resistance (C_s)

$$\epsilon' = C_s d / \epsilon_0 A \quad (3)$$

Where: C_s the series capacitance, ϵ_0 is the permittivity of free space equal to 8.85×10^{-12} Farad(meter)⁻¹. d is the thickness and A is the effective cross sectional of the electrode = $\pi D^2/4$.
The loss factor is:

$$\epsilon'' = \epsilon' / R_p C_p w \quad (4)$$

Where R_p is the parallel resistance

$$R_p = 1/D C_p w, \quad (5)$$

C_p is the parallel capacitance and w is the angular frequency equal to $(2\pi f)$.

D is the dissipation factor (12).

$$G_s = \epsilon' C_0 w \quad (6)$$

$$C_0 = \epsilon_0 A/d \quad (13)$$

and phase shift (Φ). Calculations have been done of dielectric constant (ϵ'), dielectric loss (ϵ'') ac conductivity (σ_{ac}) and dc conductivity (σ_{dc}). As shown in Table (3). The dielectric constant (ϵ') is calculated from the following formula:

thickness and A is the effective cross sectional of the electrode = $\pi D^2/4$.

σ_{ac} is the frequency dependent ac conductivity of the sample that arises from the motion of charge carrier through the polymer.

The conductance G_s is calculated by following expression;

Where is the free space capacitance $C_0 = 4.863 \times 10^{-4}$ pF without dielectric, the permittivity of free space ϵ_0 , the cross sectional area of the electrode of free space (A) is 3.846×10^{-6} mm² and the thickness (d) is 0.07 mm the separation between the electrode is 5 mm.

$$\sigma' = \sigma_{dc} + \sigma_{ac} \quad (7)$$

Where the ac conductivity σ_{ac}

$$\sigma_{ac} = \epsilon' \epsilon_0 \omega \quad (8)$$

σ_{dc} is dc conductivity, this is frequency independent conductivity from (7) is equal to

$$\sigma_{dc} = \sigma' - \sigma_{ac}$$

The real part of the conductivity can be calculated σ'

$$\sigma' = (d/A) G_s \quad (9)$$

The calculation of the conductivity is as shown in Table 4.

The ideal resistance R_s and an ideal

$$Z = R_s + jX_s \quad (10)$$

At that frequency, the impedance behaves like a series of an ideal resistance and an ideal reactance X_s . If X_s is negative, the impedance is

$$X_s = -1/2\pi f C_s \quad (11)$$

Where f is the frequency equal 1 KHz.

In general the complex impedance $\sigma^* = \sigma'(w) + i\sigma''(w)$ where σ' and σ'' are the real and imaginary parts of the conductivity. The real part of the conductivity σ' has been often analyzed separated in two different components (14).

reactance X_s . The real part and imaginary part:

capacitive and the reactance can be replaced with capacitance as shown in equation (11). (15).

Table (2): Sample preparation

No. Weight of PVA gm.	Weight of polyol gm.	Percentage of added kaolin gm.	Total weight gm.	Thickness of sample mm.	Length of sample mm.	Width of sample mm.	Electrode effective area mm ² .
1-0.18	0.06	3%	0.2472	10	22	12.5	7.85×10^{-3}
2- 0.18	0.06	5%	0.252	11	21.5	12.4	1.96×10^{-1}
3-0.18	0.06	6%	0.2544	10	22	12.5	7.85×10^{-1}
4-0.18	0.06	8%	0.2592	8	21.4	13	7.06×10^{-2}
5-0.18	0.06	10%	0.264	15	19	11.2	3.84×10^{-1}

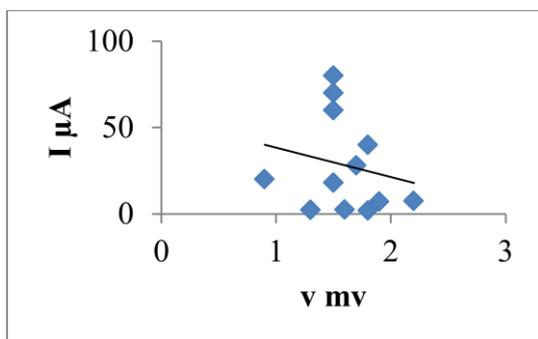


Figure (3): I-V characteristics of PVA-Polyol with 3wt% kaolin.

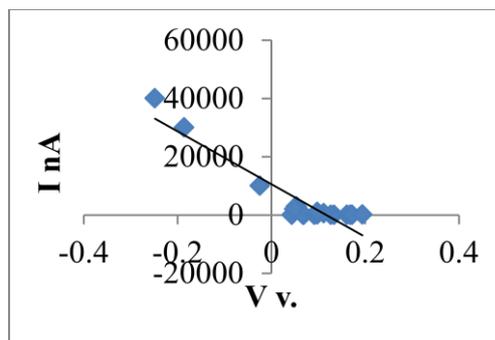


Figure (4): I-V characteristics of PVA-Polyol with 5 wt% kaolin

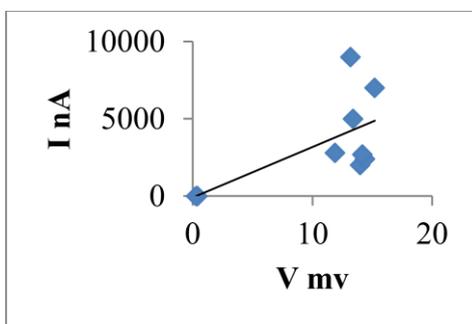


Figure (5):I-V Characteristics of PVA-Polyol with 6wt% kaolin.

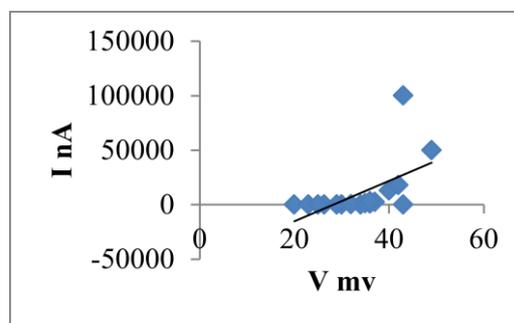


Figure (6): I-V characteristics of PVA-Polyol with 8wt% kaolin.

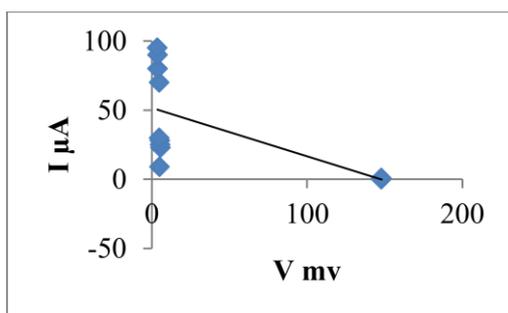


Figure (7): I-V characteristics of PVA-polyol with 10wt% kaolin.

Table (3): The dielectric and electrical properties of the samples

Sampl	Q As	Cp P F	CsP F	RpMΩ	Rs MΩ	Z MΩ	Dissipati on factor	Φ deg
1	8.57	3	2	8	8	55	0.11	83
2	0.73	65	10 ²	2	1.3	1	13.6	39
3	8.03	2.9	2.9	318*	6.7	54	0.145	82
4	2.35	4	3	135	13.8	51	0.366	6
5	0.81	2.9	2.8	2	7.5	55	0.137	82
	ε	ε'	Gs S	σac Sm ⁻¹	σ' Sm ⁻¹	σdc Sm ⁻¹	σ** Sm ⁻¹	Xs Ω
1	3x10 ⁵	3.3x10 ⁴	5x10 ⁻⁴	1.8x10 ⁻³	1.1672	1.1671	3522.4	-7.6x10 ⁷
2	6x10 ⁵	8.1x10 ⁶	1.8x10 ⁻⁶	4.5x10 ⁻¹	0.1028	-0.3472	1.044	-1.5x10 ⁶
3	4x10 ³	5.8x10 ²	1.2x10 ⁻⁸	3.2x10 ⁻⁵	1.5x10 ⁻⁴	1.5x10 ⁻⁴	27.229	-5.4x10 ⁷
4	3.8x10 ⁴	1.4x10 ⁴	6.2x10 ⁻⁵	7.7x10 ⁻⁴	0.0122	0.0121	20.58	-5.3x10 ⁷
5	1.2x10 ⁴	1.6x10 ³	3.6x10 ⁻⁸	3.8x10 ⁻⁵	1.4x10 ⁻³	0.00136	467.248	-5.6x10 ⁷

*calculated from $R_p = 1/DwC_p$.

** The average values of electrical conductivity are calculated from Table

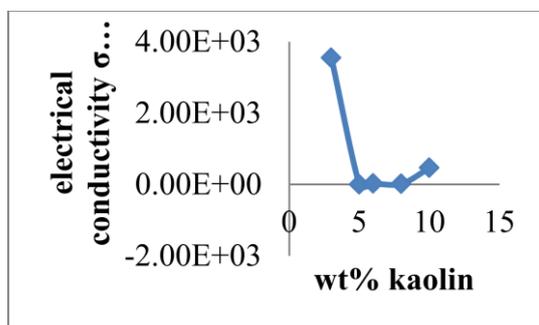


Figure (8): Effect of wt% kaolin on the electrical conductivity (σ).

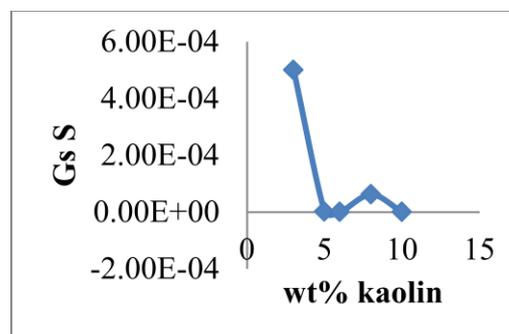


Figure (9): the dependence of conductance on the wt% kaolin.

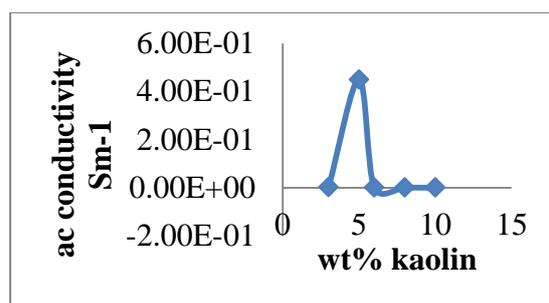


Figure (10): Effect of wt% kaolin on ac conductivity (σ_{ac})

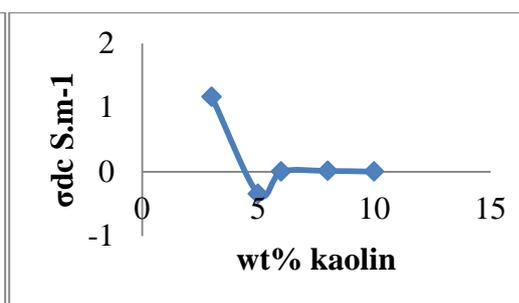


Figure (11): the effect of wt% kaolin on dc conductivity (σ_{dc})

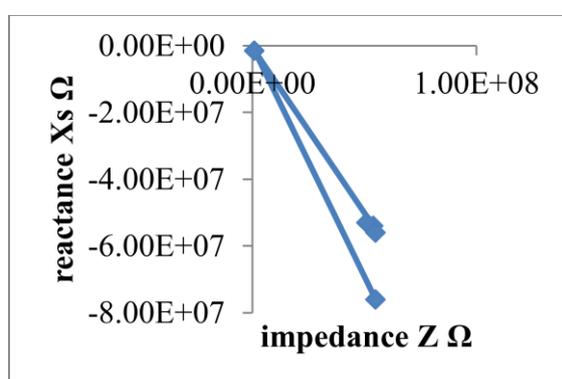


Figure (12): The impedance Vis reactance at different percentage of kaolin.

Results and discussion

FTIR test of kaolin as shown in Figure (1). Table (1) indicates the improvement of chemical composition of kaolin because the functional groups become evident with respect to the wave numbers. Investigates OH vibrations whose transitions band appears at different frequencies depending on the cations directly linked to the hydroxyls. These are permit the determination of cation distribution around hydroxyls and thus allows assessing short-range cation ordering. The structure of kaolin mineral consist of tetrahedral sharing a

plane of oxygen and hydroxyls (inner hydroxyl), with a sheet of edge-sharing octahedral and every third site vacant(dioctahedral)(8). I-V curve measurements by using the electric circuit in Figure (1) and the calculated resistivity and electrical conductivity of the samples. These are depending on the variations of current as a function of time (16). According to these variations the conductivity is varied for several orders of magnitude (17). The sizes of particles of small aspect ratio of kaolin have great effect on the conductivity (18). For example, sample

1 at 3% wt kaolin the variation is nearly the same there is no change in orders of magnitude as shown in Figure (3). For sample (3), 6wt% kaolin the conductivity raised by three orders of magnitude and sample (4), 8wt% kaolin the change is five orders of magnitude. The I-V curve measurements of PVA-polyol for 3wt% kaolin is shown in Figure (3) the variation of current with voltage is rather unstable in time interval 2760 seconds, because the straight line either to obey Ohm law (16). In Figure (4) the percentage is 5wt% kaolin there is a reverse bias in voltage this makes the current in same direction in time interval 3300 seconds. At PVA-polyol with 6wt% kaolin as shown in Figure (5), the direction is also unstable as in Figure (3) and the measurement is done in time interval 2280 seconds. In Figure (6), PVA-polyol with 8wt% kaolin, The I-V curve measurement is rather stable and Ohm law can be applied in time interval 3480 seconds. For 10wt% kaolin the I-V curve measurement as shown in Figure (7) the change in current values to μA rather than in direction, this is let the current flow steep directions in time interval 2040 seconds. The size effect of electrical conductivity originates from the electrical conductivity decrease as the film thickness increased at constant wire diameter. The experimental values indicate that bulk electrical conductivity reduces along with increasing grain size. The calculated maximum electrical conductivity is found (6794, 2.287, 131.89, 263.52 and 1284.4) Sm^{-1} . In Figure (8) shows the effect of wt(3, 5, 6, 8 and 10)% kaolin of the bulk

samples on the average electrical conductivity (σ), there is decreased in the electrical conductivity with increasing at wt5% kaolin and the electrical conductivity is increased again at wt10% kaolin. In Table (3), the dielectric behavior of polymer determined by the charge distribution and also by statistical motion of its polar group. The dielectric measurements were done in frequency 1 KHz. The effect of kaolin content on the dielectric constant (ϵ') (is the ability of a material to store an electric charge) and dielectric loss (ϵ'') of PVA-Polyol as composite films there is increased at 5wt% kaolin to the value 6×10^5 in sample (2) and 3wt% kaolin to the value 3×10^4 as in sample (1), these are for (ϵ'). For (ϵ'') there is increased at 5wt% kaolin to the value 8.1×10^6 sample (2) and 3wt% kaolin to the value 3.3×10^4 for sample (1). These are due to the weak magnetization at low percentage by weight of kaolin (19). The investigations of the dielectric properties of the composite films are depend on the percentages by weight of kaolin. The dissipation factor (D) (is the degree of the dielectric loss) is increase slightly with increasing percentages by weight of kaolin accept at 5wt% kaolin there is increased to 13.6. The dc conductivity is frequency independent has been calculated different percentages by weight of kaolin in Table (3). The samples were exhibited variation of dc conductivity range using formula (7) and (8). Figure (9) shows the dependence of the conductance on the applied wt% kaolin there is increased in the conductance 5×10^{-4} S at wt3% kaolin sample (1) and the conductance is decreased to

1.2×10^{-8} S at 6 wt% kaolin. Figure (10) shows the effect of (3, 5, 6, 8 and 10) wt% kaolin of the bulk samples on the frequency dependant ac conductivity (σ_{ac}), is increased the ac conductivity with increase 5wt% kaolin to 0.45 S.m^{-1} at sample (2), and is decreased at wt6% kaolin sample (3) to $3.2 \times 10^{-5} \text{ S.m}^{-1}$, 8 wt % kaolin to the value $7.7 \times 10^{-4} \text{ S.m}^{-1}$ in sample (4) and 10 wt % kaolin to the value $3.8 \times 10^{-5} \text{ S.m}^{-1}$ in sample (5). Figure (11) shows the effect of wt (3, 5, 6, 8 and 10) wt% kaolin on the frequency independent (σ_{dc}), that is increased first at 3 wt % kaolin to the value 1.1671 S.m^{-1} sample (1) and is decreased at 5wt% kaolin to -0.347 S.m^{-1} in sample (2), at

6 wt % kaolin to $1.5 \times 10^{-3} \text{ S.m}^{-1}$, at 8wt% kaolin to 0.0121 S.m^{-1} in sample (4) and is decreased at wt10% kaolin to 0.00139 S.m^{-1} in sample (5). The frequency dependent conductivity (σ_{ac}) representation is a most prominent representation to relate the macroscopic measurements to the microscopic movement of the ions (20). Ac conductivity has been calculated from expression (7), at constant frequency 1 KHz as shown in Table 3. Figure (13) shows the variation on impedance in ohm vis the calculated reactance X_s in ohm.

Conclusion

1- The electrical characterization and the dielectric properties depend on the functional groups of the kaolin.

2- The electrical characterization and dielectric properties depend on particles size and percentage by weight of kaolin.

3- Frequency dependent (σ_{ac}) and frequency independent (σ_{dc}), dielectric constant (ϵ') and dielectric loss (ϵ'') in PVA-Polyol and kaolin samples composite has been studied at frequency 1 KHz.

4- Kaolin mineral has properties that make it a functional Ingredient in industrial applications such as paper industry, paint, rubber and plastics.

5- The dielectric Loss is increased with increasing the percentages by

weight of kaolin at sample (2) and is decreased at sample (3) and sample (5)

6- The dielectric constant (ϵ') is increased at 5wt% of kaolin and 10wt% of kaolin. 7- The dc conductivity (σ_{dc}) decrease with increasing the percentages by weight of kaolin accept at sample (1) is increased to 1.167 S.m^{-1} .

8- The ac conductivity (σ_{ac}) depends on the calculated loss factor increase at a certain percentage by weight of kaolin. This is 5 wt % of kaolin to 0.45 S.m^{-1} .

9- The minimum calculated conductance is at 6 wt% kaolin $1.2 \times 10^{-8} \text{ S}$ and maximum at 3wt% kaolin $5 \times 10^{-4} \text{ S}$.

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