ISSN: 0974-5823 Vol. 6 No. 3 December, 2021

International Journal of Mechanical Engineering

Behavior of Dielectric Constant, Resistivity and Dissipation Factor of Foamed Epoxy

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***Abstract* - Dielectric materials are used in many applications as insulators to separate electrical conductors. These materials are usually thermoplastic materials. The current study try to use a thermosetting material which is the epoxy and foaming the resin, using a blowing agent of siloxane, in order to determine the dielectric characteristics such as dielectric constant, resistivity, and dissipation factor. The work has been conducted experimentally by manufacturing several specimens with different siloxane content as: 0, 5, 10, 15 and 20 wt%. The dielectric characteristics of the specimens have tested under suitable conditions using the impedance analyzer 6377 LCR that has high accuracy and speed. The results revealed that the quantity and sizes of pores as well as foam morphologies have a direct influence on the on the resultant bulk density thus the dielectric characteristics. The obtained results have shown an overall decrease in the dielectric constant from 3.7 to 2.1 for each 15% addition of siloxane. Furthermore, the increasing in the resistivity was from 5→90x1010 Ω.cm, and the decrease in the dissipation factor from was 0.0025 to 0.005.**

Index Terms - dielectric constant, resistivity, dissipation factor, epoxy, and siloxane

INTRODUCTION

A dielectric is a material whose internal electric charges do not flow freely, and therefore make it nearly impossible to conduct an electric current under the influence of an electric field. This contrasts with other materials which conduct electric current more easily. Dielectric materials are used to separate electrical conductors in the devices beside the role of waterproofing and support the strength of the tools [1]. Many materials are considered as good dielectric materials such as polymers and ceramics. Dielectric materials vary with respect to many bulk properties such as: thickness, cell size, temperature, frequency. However, general characteristics of the dielectric materials are [2-5]:

- Dielectric constant: is defined as the material's ability to store an amount of charge when used as a capacitor dielectric. It is usually greater than 1 since it is measured as a ratio to the permittivity of vacuum.
- Volume resistivity: is defined as the measured electrical resistance through a material when a certain voltage is applied for a specific amount of time. For an insulator material, it is generally greater than 10^{10} Ω.m at 25°C.
- Dissipation factor: also called loss factor or dielectric loss, is defined as the power dissipated by a dielectric, generally less than or equal to 0.03 at 1 kHz and less than or equal to 0.05 at 1 MHz, according to ASTM D150.
- Dielectric strength: also referred to as breakdown voltage, is the maximum electric field a material can withstand before breaking down. It is an important property for applications that run at high current. Dielectric strength for polymeric insulators is between 5-500 kV/cm.

Epoxy is a solid thermoset polymer has a strong structure due to the crosslinking bonds that gives the material many preferable mechanical and physical properties, such as high strength, and good thermal and chemical resistance. Epoxy is a good electrical insulator as well. The dielectric characteristics of pure epoxy are listed in Table 1.

Table 1. The dielectric characteristics of pure epoxy [2-5]

Dielectric property	Value
Dielectric constant	3-4
Resistivity	>1010 Ω.cm
Dissipation factor	0.01-0.05
Dielectric strength	>5 kV/cm

Many investigations have studied the dielectric properties of epoxy resin for certain cases. Most of these studies are mainly looking for improving physical, mechanical and chemical properties of epoxy composites by reinforcement methods. These methods include mixing the neat epoxy with different materials such as: particles, fillers, fibers or solutions, as referred by [6-11]. Some researchers have studied light weight epoxy obtained by involving nano-materials in the composite, as referred by [12-18], or by foaming the epoxy using blowing agents, as referred by [19-22]. The current work investigates the properties of foamed epoxy, as a light weight epoxy using a blowing agent of siloxane, regarding their dielectric characteristics such as dielectric constant, resistivity, and dissipation factor.

Materials and Methods

The study proposes manufacturing several specimens of epoxies incorporated with different contents of blowing agent, and evaluating the dielectric characteristics of the produced foamed epoxy as a light weight composite. The epoxy used in this study was SIKADUR-52 which is produced by Swiss company (Sika). The product is a 2-component: resin and hardener, moisture-tolerant, low-viscosity and high strength adhesive. The blowing agent used was 1,1,3,3-tetramethydisiloxane which is produced by Chinese company (Jinan) .The specifications of the materials used are shown in Table 2.

Table 2. Specifications of the materials used in this study*

Material	Properties
Epoxy resin (SIKADUR-52)	Two-component liquid, viscocity:500 mpa.s at 20 oC, compressive strength: 53 MPa after 10 days, Modulus:1000 MPa
Blowing agent (1,1,3,3-tetramethydisiloxane)	Clear colorless liquid, CAS: 3277-26-7, MW:134.32, Boiling at 70 oC, Flash at -10 oC.

* Supplied by the product data sheets.

The experimental works have done in the Department of Materials, Mustansiriyah University to prepare specimens of different siloxane contents as: 0, 5, 10, 15 and 20 wt%. Bulk epoxy samples were prepared by mixing the solution (resin and hardener) in a suitable container set on a precised weight scale in order to get the desired quantity. The epoxy resin was mixed with the amine hardener by 2/1 ratio (epoxy/hardener). The blowing agent (1,1,3,3-tetramethydisiloxane) then added with blending. The solution then poured in the mold and remained for 24 h at room temperature for curing, followed by post-curing at 70 C for 4 h. Due to the long gel time of the epoxy resin at room temperature and in order to avoid the early reaction of the blowing agent with the epoxy, the siloxane was added to the mixture after a pre-curing period of 2 h, where the viscosity was still low. Note that ignoring the last procedure exposes the resin to fast thermal curing, and induce an abrupt decrease of viscosity of the resin due to the high temperature rise, causing the coalescence and escape of most of the bubbles generated [23]. Each specimen has a disc shape of 4 cm diameter and 0.5 cm height, as shown in Figure 1, to satisfy the requirements of measurement of dielectric characteristics according to ASTM D150.

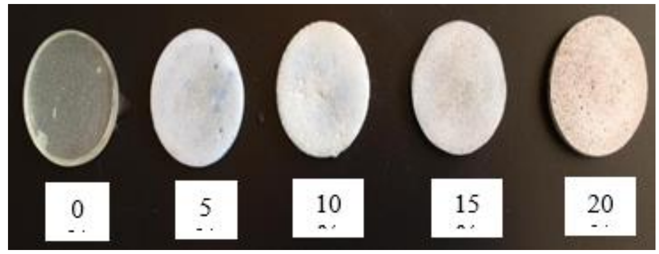

Figure 1. Specimens manufactured in this study

The measurements were done at the Test Lab, University of Technology, where the dielectric characteristics of the specimens have tested under suitable conditions using the impedance analyzer MICROTEST 6377 LCR that has high accuracy and speed, as shown in Figure 2.

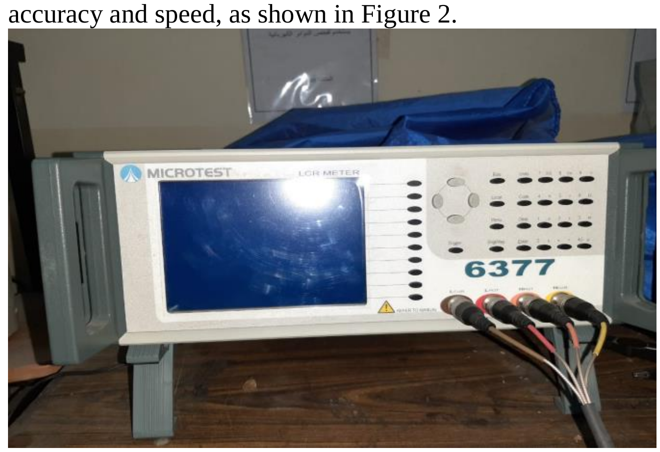

Figure 2. MICROTEST impedance analyzer 6377

Results and Discussion

Many factors influence the foaming reaction in the epoxy such as type and concentration of blowing agent [23], type and concentration of amine curing agent [24], temperature and heat transfer of the mixed resin [25, 26], water absorption [27], as well as the viscosity of the resin [28]. The viscosity is critical for the proper foaming process, and the curing kinetics can abruptly change the process of forming the gel, posing a relevant processing difficulty. A controlled pore size ensures consistent foam quality and improved dielectric properties. An early foaming gas release will lead to coalescence and escape of the bubbles due to the low viscosity of the resin; a delayed release will result in an insufficient or inhomogeneous foaming [28, 29].

Bulk density of the manufactured samples of the foamed epoxy due to the addition of siloxane content, from 5 to 20 wt % are obtained as a function of siloxane content, as shown in Figure 3. As expected due to the expansion happened by foaming, an important decrease in the bulk density has achieved, from 1.25 g/cm^3 for the neat epoxy down to 0.64 g/cm^3 for the sample synthesized with the addition of 20 wt % of blowing agent.

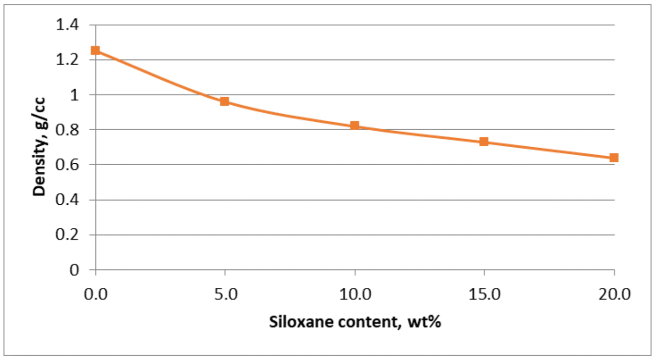

Figure 3. Density of the epoxy as a function of siloxane content

Images of scanning electron microscope (SEM) for internal partitions of the samples have been captured, by VEGA-II TESCAN device, to analyze the structure morphology of the epoxy before and after the addition of siloxane. Figure 4 shows the images for two selected samples, 0% as a neat epoxy and 15% as a foamed epoxy. In the first image (neat epoxy), the cells are too small, packed and gathered in a continuous clusters of amorphous groups. By looking to the second image (foamed epoxy), the structure was less uniform and showed the presence of many pores with different sizes and distributed randomly. The difference in cell size between the two images is most apparent in the side-by-side comparisons. It is clearly recognized how the pores are mainly closed, fairly spherical with an average pore size in the order of the tens of micrometers.

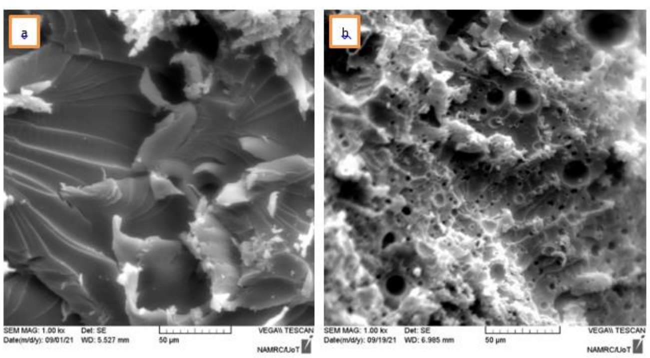

Figure 4. SEM images for (a) 0% and (b) 15 % specimens

The average diameters and distribution of pores in 1 mm^3 volume for the selected samples according to the amount of siloxane added has been calculated from the SEM images, as presented in Table 3. In general, the quantity and the size of the pores have increased by increasing of the blowing agent content. This can be attributed to coalescence phenomena, which are favored by an increase on blowing agent concentration. At the end, this leads to a lower apparent density.

Table 3. Diameters and distribution of pores in 1 mm^3 volume

Sample	Distribution	Diameter of pore (μm)			
		<10	10-25	25-50	>50
0%	Quantity	-	-	-	-
	Percentage	-	-	-	-
5%	Quantity	12500	4500	700	200
	Percentage	70%	25%	4%	1%
10%	Quantity	15000	6000	1800	500
	Percentage	64%	26%	8%	2%
15%	Quantity	16000	8000	3000	1000
	Percentage	57%	28%	11%	4%
20%	Quantity	17000	9500	4000	1500
	Percentage	53%	30%	12.5%	4.5%

During the measurements of the dielectric characteristics of the foamed epoxy, special attention is paid to the following dielectric parameters: dielectric constant, resistivity and dissipation factor through a range of frequencies between 100-1000 kHz. Figures 5-7 show the variation of dielectric characteristics with respect to the bulk density. The results show generally that the presence of the foam as a result of siloxane content has a decreasing effect on both dielectric constant and dissipation factor due to less permittivity value for air or vacuum comparing to the polymer, but there is an increasing effect regarding the volume resistivity due to the role of pores in repelling the electrical current.

The dielectric constant of all samples was clearly dependent on foam density as a proportional relationship with no significant differences regarding the frequency, only for the 20% sample where there is a slight reduction at 1000 kHz. The dielectric constant has decreased from 3.7 to 2.1, or 5-15% for each 5% addition of siloxane. The volume resistivities showed less dependence on foam density at high frequencies (>400 kHz) but quit more variation and higher values at low frequencies. The overall increasing in the resistivity was from $5 \rightarrow 90 \times 10^{10}$ Ω.cm. The results indicated a reduction in the values of dissipation factor for the foamed epoxy samples. There was again a clear dependence on foam density and a strong correlation with the frequency where the peak values appeared at 500 kHz. The overall decrease in the dissipation factor was from 0.0025 to 0.005.

The analysis of the results reveals that dielectric characteristics were clearly dependent on bulk density in all samples as a result of presence of the foam in the resin. Since the quantity and sizes of pores as well as foam morphologies have a significant effect on the resultant density thus the dielectric characteristics are affected by these factors as well. References [30, 31] mentioned also to the fact that the dielectric constant decreases with an increase in the porosity and that the dielectric constant is associated also with pore shape and pore anisotropy.

Table 4 shows a comparison between the results obtained from the current study with average results extracted from comparative studies for a range of foamed epoxy samples

Table 4. Comparison between different results for a range of foamed epoxy samples

Property	Current study	Ref. [19]	Ref. [21]	Ref. [22]
Density (g/cc)	1.25→0.64	0.8→0.2	→0.3	→0.13
Dielectric constant	3.7→2.1	2.6→1.5	-	3.5→1.7
Resistivity (Ω.cm)	5→90x1010	3→20x1015	1→1000x1010	-
Dissipation factor	0.025→0.005	0.015→0.004	-	<0.1

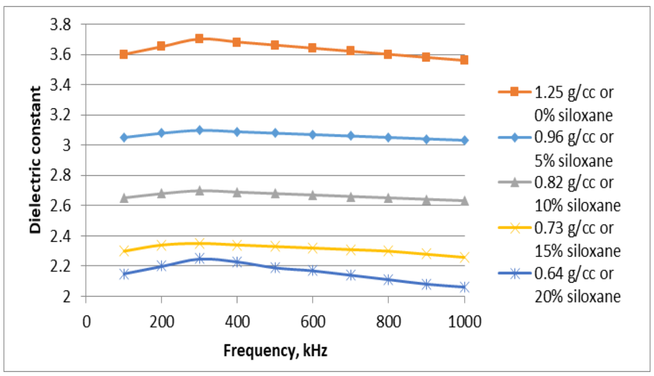

Figure 5. Variation of dielectric constant values for selected specimens

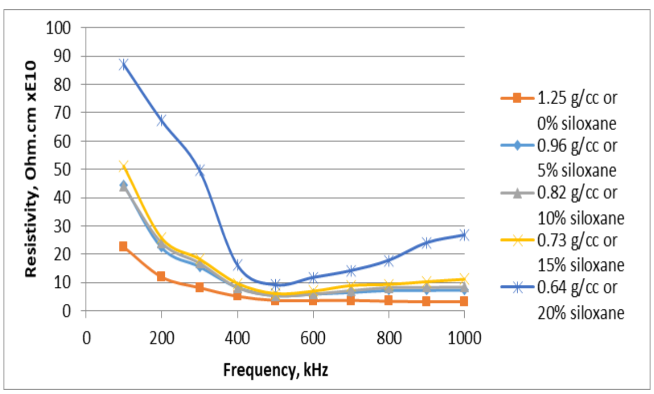

Figure 6. Variation of resistivity values for selected specimens

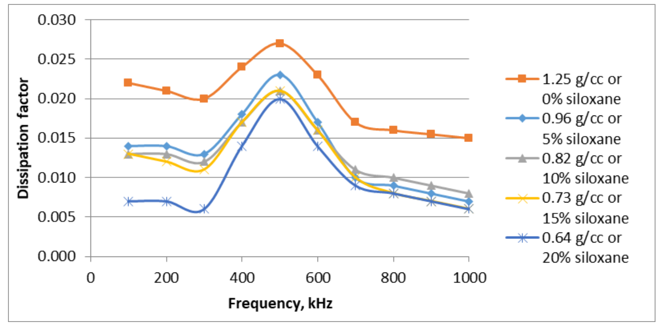

Figure 7. Variation of dissipation factor values for selected specimens

Conclusions

The effect of foaming the neat epoxy resin by siloxane blowing agent has a direct influence on the dielectric characteristics such as dielectric constant, resistivity, and dissipation factor, as following:

- Overall decrease in the dielectric constant from 3.7 to 2.1, or 5-15% for each 5% addition of siloxane.
- Overall increasing in the resistivity from 5→90x10^{10} Ω.cm.
- Overall decrease in the dissipation factor from 0.0025 to 0.005.
- The dielectric characteristics for all samples were clearly dependent on bulk density as a proportional relationship with low significant differences regarding the frequency.
- The quantity and sizes of pores as well as foam morphologies have a direct influence on the on the resultant bulk density thus the dielectric characteristics.

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Acknowledgments
Authors are grateful to the support given by College of Engineering, Mustansiriyah University for academically aid and facilities.