Characterization of El-Fawakhir Serpentine Fibers and Their Use in the Reinforcement of Unsaturated Polyester

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Abstract: Serpentine fibers from the El–Fawakhir area in the Eastern Egyptian desert were obtained from the parent rock and characterized using XRD, XRF, IR and thermal analysis. They were then incorporated into unsaturated polyester (UP) matrix to from slabs. These slabs were tested for thermal conductivity and thermal expansion where their insulation behavior was much better than UP samples containing E type glass fibers. Both composite matrices (UP + Serpentine and glass fibers) exhibited similar values of thermal expansion, decreasing with increased fiber level. Both matrices showed comparable tensile and flexural strengths both increasing with increased fiber fraction; while the elongation was much lower in case of serpentine fibers. AC characteristics (AC resistivity, dielectric constant, dielectric loss and dissipation factor) were also determined for both types of UP matrices at different frequencies ranging from 40 Hz to 1 MHz and temperatures up to 120^{o}C. The results showed similar behavior in both types of matrices although those reinforced with mineral fibers showed lower dissipation losses.
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1. Introduction:

The advantage of composite materials over conventional materials stems largely from their higher specific strength, stiffness and fatigue characteristics, which enables structural design to be more versatile. By definition, composite materials consist of two or more constituents with physically separable phases [(1)]. However, only when the composite phase materials have notably different physical properties it is recognized as being a composite material. The most common advanced composites are polymer matrix composites. These composites consist of a polymer thermoplastic or thermosetting reinforced by fiber (natural fiber or glass). These materials can be fashioned into a variety of shapes and sizes. They provide great strength and stiffness along with resistance to corrosion. The reason for these being most common is their low cost, high strength and simple manufacturing principles. Due to the low density of the constituents the polymer composites often show excellent specific properties. In composites where the continuous phase consists of unsaturated polyester this latter transfers external loads to the reinforcement, and protects the reinforcement from the environment. If individual fibers are fractured, the matrix will redistribute the load to the surrounding fibers, thus preventing the complete failure of the material. Such composites exhibit a broad range of mechanical, chemical, thermal and physical properties, depending on the composition of the unsaturated polyester [(2)]. The commercial use of fiberglass–reinforced unsaturated polyester resins began in 1942, when polyester resin was combined with glass–fiber reinforcement to produce protective housings for radar equipment. Nowadays unsaturated polyester resins fiber composites are used in a wide variety of markets, including construction, marine transportation, industrial, electrical, and sanitary ware.

The use of reinforcing fibers to produce unsaturated polyester composite dramatically improved both tensile and flexural characteristics. The properties that can be obtained depend on the amount and type of reinforcement used. For example, adding 40% (by weight) E – glass fibers to orthopthalic unsaturated polyester raises the tensile strength from 55 MPa to 152 MPa, while the flexural strength increases from 85 MPa to 221 MPa [(3)].

As for electrical properties, polyester resins are nonconductors, having relatively low dipolar characteristics, and providing high dielectric strength and surface resistivity. At high voltage or high current, however, the cross–linked plastics fail due to carbon arcing or tracking caused by the charring of the polymer surface into a conductive carbonaceous residue [(4)]. The AC resistivity of isopthalic polyester reinforced with E type glass fibers ranges from 10^{11}

to 10^{13} Ω.m, while the dielectric constant decreases from about 7 to 5 as the frequency increases from 60 Hz to 1 MHz [3]. These composites find extensive use in the insulation of motor windings, encapsulation of electrical components, fabrication of printed circuit boards, high voltage standoff insulators, switch boxes, and miscellaneous equipment used on high–voltage transmission lines [5]. Large electrical equipment, such as high–voltage motors or generators, often operates at elevated temperatures. In such applications, the electrical property of greatest concern is the dissipation factor, especially the dissipation factor versus temperature. Polyester resins can be formulated for a low dissipation factor at elevated temperatures. They can be used as electrical varnishes at continuous use temperatures up to 180°C [3].

2. Experimental

2.1 Raw materials:

The following raw materials were used in the present study:

- Polyester resin of type "Siropol 8339". This is an orthophtalic unsaturated polymer resin manufactured by "Saudi Industrial Resins Limited".
- Mat E – glass fibers "M 706" of surface density 300 gm/cm^2, manufactured by "European Owens–Coring Glass fiber S.A." company.
- Mat mineral fibers from El–Fawakhir area, supplied by "Central Metallurgical Research Institute (CMRDI)", this was characterized using X – ray diffraction, X – ray fluorescence and Scan electron Microscopy.

2.2 Characterization of as – received mineral fibers:

a. X – ray diffraction analysis:

The parent rock hosting mineral fibers from El–Fawakhir area were analyzed by using a "PW 1170 Philips Diffractometer" with Co K_α radiation on randomly oriented specimens. This way, the phases constituting these fibers were disclosed.

b. X – ray fluorescence analysis:

Philips X–ray flouresences (XRF) Spectrometer was applied for determining the chemical composition.

c. IR spectroscopy:

The IR spectra were obtained using FTIR spectrometer (Bruker). For each sample 128 scans were recorded in the 4000–400 cm^{-1} spectral range in the transmittance mode with a resolution of 4 cm^{-1}. The KBr pressed disk technique was used.

d. Thermal analysis:

The thermal analysis for mineral fibers was carried out by using Shimadzu TGA–50H apparatus that displays DTA and TGA behavior at heating rate = 10^{o}C/min in nitrogen atmosphere.

2.3 Preparation of reinforced UP slabs:

1. A coat of mold release wax was applied with a brush or soft cloth to the entire mold surface and its lid; it was allowed to dry thoroughly then was lightly buffed.
2. Glass and mineral fibers were cut with a sharp cutter into rectangular pieces of dimensions (300 × 300) mm.
3. Polyester resin was stirred at room temperature, as mixed with a hardener (1% by weight petanox) as advised by the producer.
4. The entire mold surface was covered with the resin mixture using a brush.
5. A layer of glass fibers or mineral fibers was laid out.
6. Air bubbles were evacuated by pressing with an iron roller, and then both glass fibers and mineral fibers layers were saturated with a resin mixture.
7. Steps 4, to 6 were repeated, with varying percent fibers to produce a sheet of 7 mm thickness.
8. The mold was covered with its lid in order to obtain a regular, smooth sheet surface, and the composite was let to cure at room temperature.

2.4 Determination of fibers ratio in composite samples:

The value of the glass or mineral content in test samples was determined by the burn off test (ash test), which is carried out according to ASTM D 2584 / 08 [6].

2.5 Determination of linear expansion:

The linear expansion was measured by using Shimadzu TMA–50 apparatus. The test samples were heated from room temperature to 250°C in nitrogen atmosphere (Nitrogen flow rate = 30 ml/min), and the change in length ΔL was recorded.

The linear expansion coefficient (α) was calculated according to the equation:

$$\alpha = \frac{1}{L_o} . \frac{\Delta L}{\Delta T} \qquad (1)$$

2.6 Determination of thermal conductivity:

It was not possible to determine the thermal conductivity of mineral fibers because of their fluffy state. It was possible, however, to determine the thermal conductivity of fibers compacted to different bulk densities using the heat flow meter apparatus of type (Stanford Research Systems Model SR630) according to ASTM C 518 / 10 [(7)].

On the other hand, the thermal conductivity of composite reinforced UP slabs was measured by using (comparative thermal conductivity instrument) according to ASTM C 177 / 10 [(8)].

2.7 Determination of mechanical properties:

a. Tensile strength:

The tensile strength of reinforced slabs was determined according to ASTM D 638 / 10 [(9)] using Universal test machine jaws (model 4206, Instron.). Its measuring range is up to 150 KN, and its accuracy is about 0.05% max load. The loading rate was adjusted at 2.5 mm/min.

b. Flexural strength:

The flexural strength of reinforced slabs was determined according to ASTM D 790 / 10 [(10)] using the same universal testing machine mentioned above with a loading rate of 7 mm/min.

2.8 Determination of electrical properties:

The AC electrical resistance and dielectric properties (dielectric constant, dielectric loss and dissipation factor) were determined for specimens in the form of disks of diameter 10 mm and thickness of 7 mm. These were measured on the temperature range 30 – 120°C and on a frequency range 40 Hz – 1 MHz by a computerized RLC Bridge Model Hioki 3531Z Hitester.

3. Results and Discussion

3.1 Assessment of mineral fibers:

a. Mineralogical composition:

As stated earlier this was accomplished using XRD. Figure (1) shows the pattern obtained. It shows that these fibers are composed of antigorite and actinolite amphiboles [$Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$] as well as the related tremolite phase [$Ca_2(Mg)_5Si_8O_{22}(OH)_2$]. Besides are present the following phases: quartz, magnetite and potash feldspar.

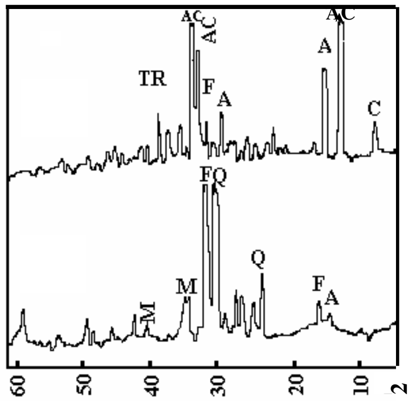

Fig. (1): XRD patterns of parent rock

Where:

AC = Actinolite [$Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$] F = Feldspar

TR = Tremolite [$Ca_2Mg_5Si_8O_{22}(OH)_2$] Q = Quartz

M = Magnetite A = Antigorite

b. Chemical composition:

As subjected to XRF analysis, the chemical composition of fibers was as shown in the following table.

Table 1: Chemical composition of mineral fibers

Component	Composition (%)
SiO_2	46.4
Al_2O_3	2.64
Fe_2O_3	6.3
$Ti\ O_2$	0.56
MgO	42.4
CaO	0.63
K_2O, Na_2O	0.090

c. Thermal analysis:

Figure (2) shows the TGA – DTA pattern obtained on heating samples of these fibers in nitrogen atmosphere. It is clear that no appreciable weight loss could be detected at temperatures as high as 580°C.

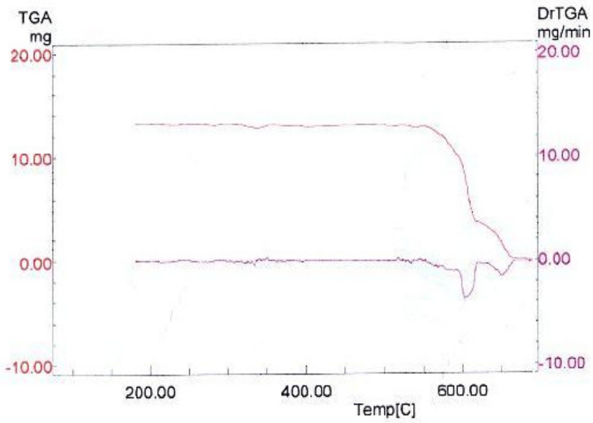

Fig. (2): TGA – DTA pattern of mineral fibers

3.2 Thermal properties:

a. Thermal expansion coefficient:

Figure (3) shows the variation of thermal expansion coefficient for both types of slabs: UP + fiber glass and UP + mineral fibers with increasing volume content. This curve displays the following features: First, the values of α for all samples investigated showed a decrease with increased fiber content. This is probably due to the impeding action of fibers on the natural expansion of the polymer. In this respect the effect of adding mineral fibers is more pronounced than in case of glass fibers, particularly at temperatures above 100°C where adding 10% (by volume) mineral fibers decreases the thermal expansion coefficient to 10% of its original value. This result is of importance in using reinforced UP in applications involving relatively elevated temperatures (such as high–voltage motors or generators).

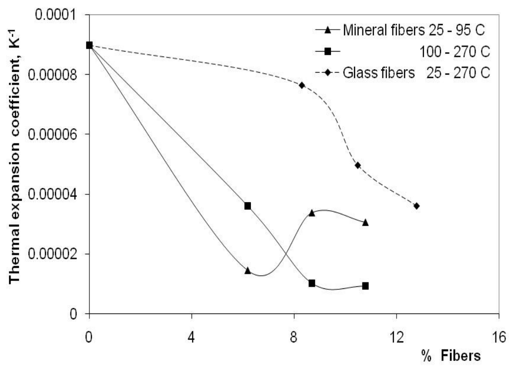

Fig. (3): Effect of volume percent fibers on the thermal expansion coefficient

b. Thermal conductivity:

As can be seen from Figure (4), while the addition of glass fibers favors higher thermal conduction, adding mineral fibers, on the opposite, yields lower values of thermal conductivity. For example, adding about 10% by volume mineral fibers decreases the thermal conductivity to less than half its original value. This means that polymer lattices reinforced with such fibers will have better thermal insulating properties than the parent matrix.

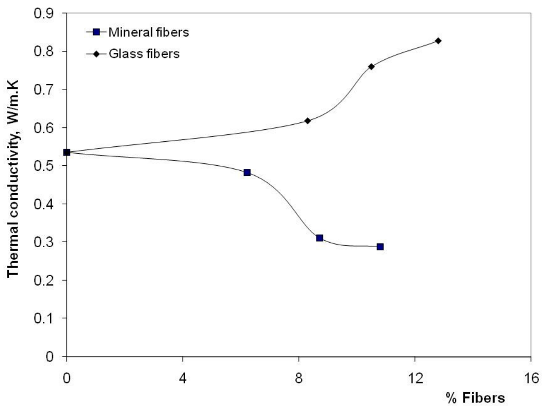

Fig. (4): Effect of volume percent fibers on thermal conductivity

3.3 Mechanical properties:

a. Tensile strength:

The effect of adding either type of fibers on the tensile strength of reinforced UP slabs is shown in Figure (5). As expected an increase in fibers percent will have for effect to increase the tensile strength. This increase however is not considerable and is comparable when either type of fibers is used. For example, adding about 6% (by volume) mineral fibers or about 12% glass fibers will only increase the tensile strength by about 10%.

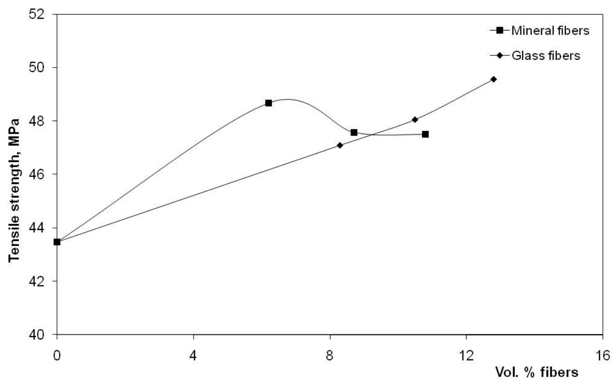

Fig. (5): Effect of volume percent fibers on tensile strength

b. Flexural strength:

The same situation can be followed up in Figure (6) which shows the variation of flexural strength as function of volume percent fibers added. Here also, the increase in strength is not appreciable, reaching a maximum of 12% when 10.8% mineral fibers were added and 19% in case of adding 12.8% glass fibers.

To conclude: Although mechanical strength increases with fiber addition, this increase is limited.

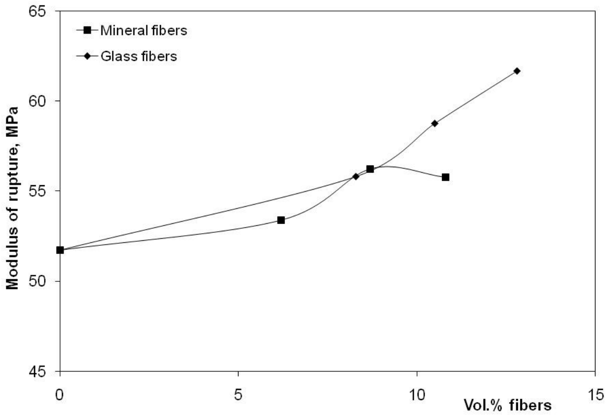

Fig. (6): Effect of volume percent fibers on flexural strength

3.4 Electrical properties:

a. AC resistivity:

Figure (7) shows the effect of adding either type of fibers on the AC resistivity of samples at low frequency (30 Hz) and at 30°C. As can be seen from this figure, there is a slight increase in resistivity following the addition of more than 8% of either type of fibers. At higher temperatures, the effect of adding fibers on increasing resistivity is more pronounced in case of adding glass fibers. The addition of mineral fibers, on the other hand has little effect on resistivity regardless of temperature.

At higher frequencies, the resistivity naturally decreases as can be seen from Figure (8) displaying data at 1 MHz. However, the effect of adding reinforcing fibers is much more pronounced, particularly at higher temperatures. For example at 100°C, adding 8.7% mineral fibers increases resistivity by more than 300%, while it increases on adding 10.5% glass fibers about 12 times.

b. Dielectric constant:

As expected, the dielectric loss decreases with applied frequency until it stabilizes at about 1 kHz. This is due to the fact that at high frequency, the polymeric dipoles don't get enough time to align because of the rapid inversion of the polarity of the electric applied field. Actually, the values of dielectric constant don't vary appreciably over 1 kHz. That is why; the results reported here are those obtained at a frequency of 1 MHz.

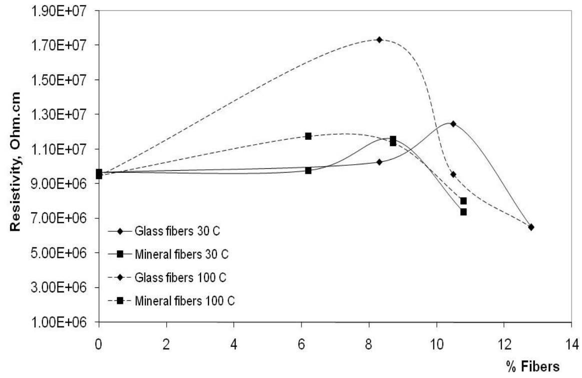

Fig. (7): Effect of volume percent fibers on Resistivity at 60 Hz

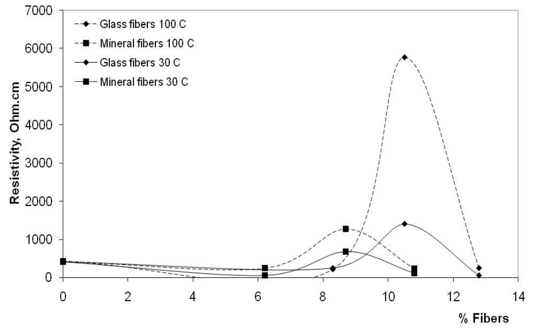

Fig. (8): Effect of volume percent fibers on AC resistivity of samples at 1 MHz and 30°C

As can be seen from Figure (9), depicting the variation of dielectric constant with the addition of fibers, the addition of glass fibers generally increases the values of dielectric constant since fiber glass possesses a higher dielectric constant than UP [11,12] (4.84 for E type fiber glass against 2.56 for UP at 1 MHz). The same trend is followed by mineral fibers although the effect is less pronounced probably because of mineral fibers having a lower dielectric constant than glass fibers. Although the dielectric constant of such fibers could not be determined, it has been reported by Johnson [12] that the dielectric constant of most ceramic raw materials falls in the range 3.3 – 3.8. This figure is higher than that of UP but lower than that of glass fibers. However, following a certain peak value, the values of dielectric constants fall once more for both types of fibers and at all temperatures investigated.

It is also clear from Figure (9) that raising temperature results in a decrease in dielectric constant. In principle, in a polar polymer, an increase in temperature should cause rapid alignment of dipoles resulting in an increase in dielectric constant [13]. It seems therefore that the presence of fibers impedes the alignment of dipoles at higher temperatures resulting in a decrease in the values of dielectric constant.

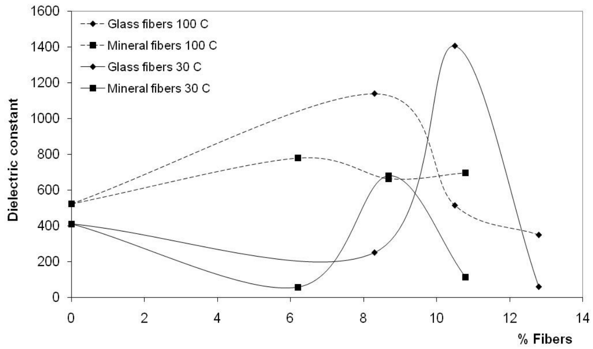

Fig. (9): Effect of volume percent fibers on the dielectric constant at 1 MHz

c. Dielectric loss:

As expected, the dielectric loss decreases with applied frequency since the rapid alternation of electric field polarity decreases dipole alignment thereby decreasing thermal losses associated with the movement of dipoles. As fibers of either type were added to the UP matrix, values of dielectric loss displayed a maximum value as can be seen from Figure (10).

This figure shows that as the percent fibers is raised, the dielectric loss in both types of samples passes through a maximum value at to drop at higher volume fractions. The effect is more observed in case of samples containing glass fibers. The probable interpretation lies in the fact that the fibers present absorb part of the heat generated by losses particularly above the glass transition temperature (About 105°C for UP) [14]. Under such conditions the increased ease in fiber movement absorbs a great part of the energy losses that is transformed in kinetic energy particularly since the specific heat of glass fibers, in particular, is much higher than that of UP: 0.8 – 1 kJ/kg.K, compared to 0.2 – 0.3 kJ/kg.K [15]. This accounts for the decrease in dielectric loss at higher temperatures. On the other hand, the fact that the maximum dielectric loss is much higher in case of samples containing glass fibers than those containing mineral fibers can be also related to the much higher specific heat of the former compared to the latter which will increase its capacity for heat absorption. (Specific heat of serpentine fibers = 0.26 kJ/kg.K).

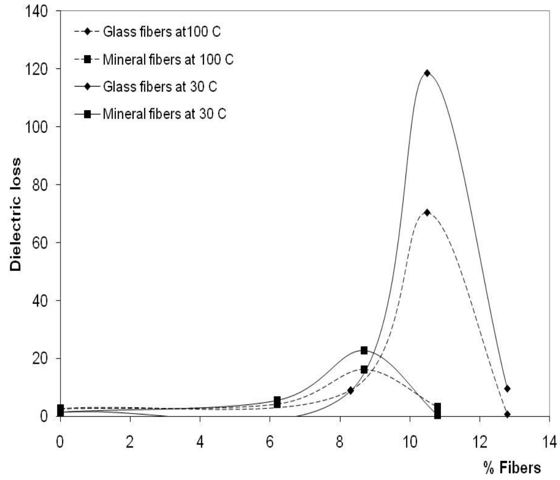

Fig. (10): Effect of volume percent fibers on dielectric loss at 1 MHz

4. Conclusions

Natural mineral fibers from El–Fawakhir region of the serpentine type were used to reinforce slab samples consisting of unsaturated polyester. Samples reinforced by E – glass fibers were also prepared for comparison. Different volume percent of fibers were used in each case. The results were as follows:

(a) Thermal properties:

Samples reinforced with natural fibers showed better dimensional stability on heating than those containing glass fibers by exhibiting much lower expansion coefficients. The mineral fibers samples also displayed much better insulating properties, showing values of thermal conductivity much lower than those of samples reinforce with glass fibers.

(b) Mechanical properties:

No appreciable difference between the two types of reinforcing fibers was noticed as tensile and flexural strengths were investigated. In both cases the mechanical strength steadily increased with increased volume percent of fibers.

(c) Electrical properties:

- At room temperature, there is no much difference in low frequency AC resistivity between both types of samples. At high frequency, however, the resistivity of samples containing glass fibers is much higher than that of samples containing mineral fibers at all temperatures investigated. Nevertheless, the addition of mineral fibers at volume percent of 8.7% raises considerably the AC resistivity of UP samples. It is therefore possible to use UP doped with about 9% mineral fibers in applications involving high frequency electrical insulation at moderate temperatures.

- At frequencies above 1 kHz, the dielectric constant of samples containing glass fibers is higher than that of samples containing mineral fibers at all temperatures. On the average, for all temperatures investigated, the maximum values of dielectric constant in case of samples containing glass fibers was about 50% higher than the corresponding values for samples containing mineral fibers. This means that UP containing glass fibers is a better capacitor than if containing mineral fibers.
- In general, the dielectric loss, at frequencies above 1 kHz, was higher in case of samples containing glass fibers than those containing mineral fibers at all temperatures. The potential use of any UP composite as dielectric rather depends on a compromise between having a high dielectric constant and a low dissipation factor. This latter is defined as the ratio between dielectric loss and dielectric constant. In this respect, it was found that the dissipation factor of samples containing mineral fibers is much lower than that of samples containing glass fibers (Figure 11).

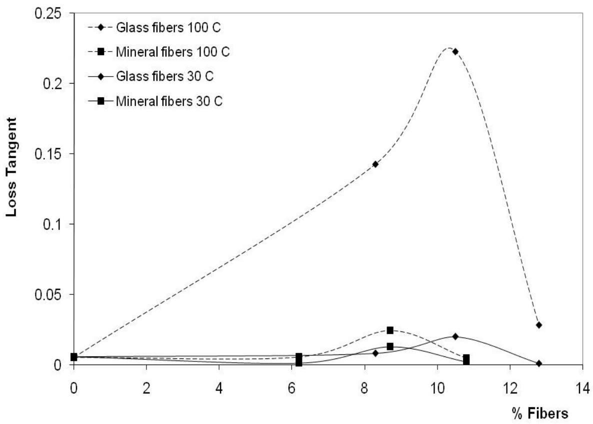

Fig. (11): Effect of volume percent fibers on the dissipation factor at 1 MHz

- UP samples with about 8.7% volume fraction mineral fibers display better dielectric constants than un–reinforced UP and relatively low dielectric loss at frequencies higher than 1 kHz. They can therefore be used as capacitors in moderate to high frequency applications.

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