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Correlation between mechanical and dielectric properties of Alfa/Wool/Polymeric hybrid fibres reinforced polyester composites

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Abstract Dielectric measurements and tensile testing of polyester/natural fibres (Alfa/wool) and thermo binder fibres (Pe/Pet) composites were investigated in order to study the adhesion of the fibres in the polyester matrix. Two composites #1 and #2 having 17:1:2 and 17:2:1 as a relative fraction of alfa/wool and thermo binder (Pe/Pet), respectively, have been characterized in this study. The obtained results revealed that the fibres adhesion in the matrix was better in the composite #1 than in the composite #2. Indeed, the analysis of the interfacial or Maxwell-Wagner-Sillars (MWS) polarization intensity, using the Havriliak–Negami model, has shown a lower intensity and the tensile testing exhibited a higher Young modulus in the composite #1. So the thermo binder fibres improve this adhesion.

1. Introduction
Over the last two decades, natural fibres reinforced polymer composites have been the focus of academic and industrial research interest (e.g. automotive, sport, construction and packaging) due to several advantages, such as low cost, high strength-to-weight ratios, low densities and recyclability compared to the synthetic fibre composites. Mechanical properties of fibre reinforced composites depend mainly upon the nature of fibre, matrix and fibre/matrix adhesion. Natural fibres such as alfa, hemp, sisal, flax, kenaf and jute are highly hydrophilic owing to the presence of hydroxyl groups (OH). This character is a potential cause for poor interfacial adhesion with hydrophobic polymer matrices. This makes natural fibre reinforced polymer composites vulnerable to environmental attacks, which may cause weakness and thus reduce its life span. In other words, insufficient adhesion between the hydrophobic polymers and hydrophilic fibres results in poor mechanical properties in these composites. Interfacial adhesion and resistance to moisture absorption of natural fibre composites can be improved by the suitable modification of fibres surface, or by using coupling agent.

In addition, the amount of moisture sorption can be reduced significantly through the replacement of natural fibres with a small amount of synthetic fibres, such as glass or carbon. Many techniques have been used to give evidence for the effect of these treatments on the fibres/matrix interfacial adhesion. The most frequently ones used are dynamic mechanical analysis, scanning electron microscope (SEM) observation, dielectric spectroscopy and tensile testing analysis.

Composites having two or more fillers contained in the same matrix are called hybrid composites. Recently there has been a growing interest in hybridizing different natural fibres in order to produce high performance composite materials. Hybrid biocomposites can be designed by the combination of a synthetic fibre and natural fibre (biofibre) in a matrix, or a combination of two natural fibre / biofibre in a matrix. Hybridization with glass fibre provides a method to improve the mechanical properties of...
natural fibre composites and the effect on different modes of stress depends on the design and construction of the composites [1]. For example, the hybridization of oil palm fibres with glass fibres increases the tensile strength, Young’s modulus, and elongation at break of the hybrid composites [2]. Moreover, synergistic strengthening of fibres has been observed in the case of banana/sisal hybrid fibre composites, where tensile and flexural properties show a positive hybrid effect [3, 4].

This research work aims at investigating the effect of wool and thermo binder (Pe/Pet) fibres on the fibres/matrix interfacial adhesion of the composites of polyester/natural fibres, where the natural fibres consist of a mixture of Alfa and wool, and the polymeric fibres composed of PET and PE as a cover. To do this the study compares two composites differing in the relative volume fractions of the wool and thermo binder fibres. These composites were characterized by the dielectric spectroscopy and the tensile testing analysis. The dielectric properties of these composites were analyzed and the intensity of the interfacial dielectric relaxation was determined, using the Havriliak-Negami model, in order to examine the interfacial region between the fibres and the matrix. A correlative study between dielectric and tensile properties of these composites was realized to confirm this dielectric study.

2. Materials and experimental techniques

2.1. Materials

The unsaturated polyester matrix used in this research work was supplied by Cray Valley/Total and is the same as that used in our previous study [5]. The matrix was mixed with the initiators methyl ethyl ketone peroxide and cobalt octanone at a concentration of 1.5% w/w before the introduction of the alfa fibres. The latter which were extracted from the plant were treated chemically by an NaOH solution and bleached in an NaClO solution. Then, they were separated mechanically with a Shirley analyzer. As combining nonwoven fibres with only alfa fibres was not possible because of the non-cohesion between them, the alfa fibres were mixed with wool fibres to ensure cohesion. Moreover, since natural fibres on their own cannot be thermoformed and require the addition of polymeric fibres to act as binders, the added thermo binder fibres were composed of PET and PE as a cover. The obtained sheet of nonwoven fibres was made up of the three kinds of fibres under study. The diameters of the alfa and wool fibres were 204.86 and 37.21 μm, respectively. Besides, the length of the thermo binder fibres was 51 mm; the fibres’ count was 4 deniers, and their melting temperatures were 260°C and 110°C. The relative volume fractions of these fibres in dry preforms used in the composites #1 and #2 had a ratio of alfa to wool to PET–PE of 17:1:2 and 17:2:1, respectively. Regarding the preparation steps of the nonwoven fibres (alfa + wool + PET-PE) fabric, they were given in details in our previous work [6]. The obtained composites #1 and #2 manufactured with the classical contact mould again as previous described [6], had 4.8 and 4.9 wt % fibre fractions, respectively.

2.2. Experimental techniques

Dielectric measurements were carried out using an Alpha dielectric–impedance analyzer (Novocontrol), with the measurements of the studied samples taken over the temperature range from the ambient to 150°C and in a frequency interval from 10⁻¹ to 10⁶ Hz. A circular gold electrode (2 cm in diameter) was sputtered on both surfaces of the sample to ensure good electrical contact with the gold-plated measuring electrodes. A sinusoidal voltage was applied in the sample in order to create an alternating electric field that produced polarization which oscillated at the same frequency as the electric field but had a phase angle shift (δ). Such phase angle shift was measured by the comparison of the applied voltage with the measured current, which was divided into capacitive and conductive components. The dielectric parameters were calculated with the following equations:

\[ \varepsilon' = \varepsilon'' - j \varepsilon'' \]  
\[ \varepsilon' = \frac{C_p(sample) \omega}{\varepsilon_0 A} \]
\[
\varepsilon'' = \frac{G_{(\text{Sample})}d}{\varepsilon_0 \varepsilon A}
\]

where \(\varepsilon'\) and \(\varepsilon''\) are the real and imaginary parts of the complex permittivity \(\varepsilon^*\). \(\tan \delta = \varepsilon''/\varepsilon'\) is the dissipation factor. \(A\) and \(d\) are the area and thickness of the sample, respectively. \(C_p\) is the capacitance, \(G\) is the conductance, \(\varepsilon_0 = 8.854 \times 10^{-12} \text{ F m}^{-1}\) is the permittivity of the free space.

The dielectric experiments were carried out with fixed temperatures and scanning frequencies from \(10^{-1}\) to \(10^6\) Hz. The tensile testing of the composites #1 and #2, it was carried out with a Lloyds Dynamometer universal testing machine as per NF T 57-301 at a crosshead speed of 5 mm/min and a gripping length of 100 mm [7]. The specimen was cut out in the direction of nonwoven production. All of the results were calculated at the average of 10 samples for each test.

3. Results and discussion

3.1. Dielectric properties

Comparative plots of the frequency dependence of the dielectric permittivity \(\varepsilon'\) and those of the dissipation factor, \(\tan \delta\), in the composites #1 and #2 for different temperatures varying from 40°C to 150°C in increments of 10°C are depicted in Figure 1-(a-d), respectively. For low frequencies, the dielectric permittivity \(\varepsilon'\) reaches high values with the increase in temperatures. It is worthy to note that at low temperatures, \(\varepsilon'\) is steadily constant and approximately equal to that of the polyester resin in the composite #1. However, in the case of the composite #2, the dielectric permittivity \(\varepsilon'\) is superior to that of the resin. This indicates the presence of water dipoles polarization in the composite #2. Indeed, the dissipation factor, \(\tan \delta\), proves the presence of a dielectric relaxation at low temperatures as illustrated in Figure 1-d. As temperature increases, the dissipation factor curves of the composite #1 have revealed the presence of a dielectric relaxation attributed to interfacial polarization field effect-Maxwell- Wagner- Sillars (MWS) polarization effect added to the one associated with dc conductivity effect above glass transition temperature (\(T_g\)). The interfacial dielectric relaxation was the result of the charge accumulation between the fibres and the matrix having different conductivities and permittivities. Therefore, the enhancement of these relaxations above \(T_g\) temperature amplifies \(\varepsilon'\) intensity as temperature increases. In the case of the composite #2, the isothermal runs of the dissipation factor show the presence of only one dielectric relaxation at high temperatures associated with the MWS polarization effect. This results in a lower rise of the permittivity \(\varepsilon\) at low frequencies and high temperatures in comparison to that of the composite #1.

The incorporation of natural fibres in the matrix has also led to a decrease in the intensity of the dissipation factor in both composites compared to that of the matrix [6]. This decrease is more important in the composite #1, which can be explained by a better thermal insulation in this composite. Indeed, it is recognized that the measurement of dissipation factor of insulating material is important
since the loss tangent is a measure of the alternating current electrical energy which is converted to heat in an insulator. Such heat raises the insulator temperature and accelerates its deterioration.

To minimize the effects of the dc conductivity, the formalism of “electric modulus” or “inverse complex permittivity” is introduced. This electric modulus has recently been adapted for the investigation of dielectric processes occurring in composite polymeric systems, as well as proposed for the description of systems with ionic conductivity. The electric modulus, $M^*$, is defined by the following Eq. 4:

$$M^* = \frac{1}{\varepsilon'} - \frac{1}{\varepsilon' - j \varepsilon''} = \frac{1}{\varepsilon' + j \varepsilon''} + j \varepsilon'' = M' + j M''$$

Figure 1: Isothermal runs of the dielectric permittivity ($\varepsilon'$) and of the dissipation factor ($\tan \delta$) versus frequency for the composite #1 (a), (c) and composite #2 (b) and (d).

Adopting this electric modulus formalism, Figure 2-(a, b) shows the behaviour of $M^*$ as a function of frequency for the composites #1 and #2 when they were heated over the temperature range from 40°C to 150°C.
40 °C up to 150 °C. A series of two distinct relaxations can be considered in the composite #1, the first of which is related to the α relaxation associated with the glass rubbery transition of the polymer and the second one, which appears at high temperatures, is attributed to ionic conduction effect. Yet, in the composite #2, a series of three distinct relaxations can be identified. Indeed, the α relaxation of the polymer appeared at temperatures above the glass transition temperature. The one which appeared at low temperatures is associated with the water dipoles polarization and the last one which appeared at high temperatures was assigned to the interfacial polarization effect. To further support these assignments, the activation energy relative to the different relaxation was evaluated using the Arrhenius relation:

\[ f_{\text{max}} = f_0 \exp\left(-\frac{E_a}{k_B T}\right) \]  

where \( k_B \), \( T \), \( E_a \) and \( f_0 \) are Boltzmann constant, temperature, activation energy of the process and frequency at high temperature. Figure 3 shows the evolution of \( \log(f_{\text{max}}) \) versus \( 1/T \) for each one of the different observed relaxations; i.e., α and conductivity for the composite #1 and α and interfacial polarization effect for the composite #2. The mean values of the activation energy \( E_a \) and the relaxation time \( \tau_\alpha \) relative to α relaxation are 136.02 KJ mol\(^{-1}\), 10\(^{-20.96}\)s and 132.694 KJ mol\(^{-1}\), 10\(^{-20.73}\)s for the composites #1 and #2, respectively as mentioned in Table 1. Nevertheless, it can be noted that the incorporation of fibres decreases the apparent activation energy \( E_a \) for the two composites in comparison with that for the matrix determined in the previous study and this could be ascribed to the interaction between the fibres and matrix [5]. This interaction will determine the nature of the interfacial adhesion region as well as the interfacial polarization effect for the composite #2 i.e., 82.711 KJ mol\(^{-1}\) for \( E_a \) and 10\(^{-13.65}\)s for \( \tau_\alpha \). These values seem to be a typical thermoactivated phenomenon that is not related to an elementary relaxation of dipoles as the characteristic time \( \tau_\alpha \) is slightly different from that of Debye (10\(^{-13}\) s). In the case of composite #1, the average values of activation energy and time relaxation relative to conduction effect and are 96.76 kJ/mol and 10\(^{-15.52}\)s, respectively. These values are in agreement with those reported in other research works [9].
Figure 3 Arrhenius plots of the frequency of M' versus the reciprocal temperature for the composites #1 (α relaxation (*), dc conduction (●)) and #2 (α relaxation (▲), MWS polarisation effect (×)).

The Argand representation is used to analyse the nature of relaxation at high temperatures. Cole–Cole plots of the composites #1 and #2 at 150 °C are depicted in Figure 4-(a, b). It is well established that the response of every relaxation mechanism can be represented very precisely by a model function with four parameters at the most. Among others, the function:

\[ \varepsilon^* = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{(1 + (\omega \tau)^\alpha)^\beta} \]

introduced by Havriliak–Negami is widely used thanks to its suitability for mathematical processing. \( \varepsilon_s \) and \( \varepsilon_\infty \) are the dielectric constants on the low and high frequency sides of the relaxation, \( \tau \) is the central relaxation time, \( \omega \) is the radial frequency, \( \alpha \) and \( \beta \) are fractional shape parameters describing the skewing and broadening of the dielectric function, respectively. Both \( \alpha \) and \( \beta \) range between 0 and 1. These coefficients act for the deviation from the Debye equation. In fact, when \( \alpha \) and \( \beta = 1 \), this equation reduces to the Debye equation. In the electric modulus formalism, the Havriliak–Negami equations (7, 8) have the following form:

Table 1 Activation Energies \( E_a \) and Relaxation Times \( \tau_0 \) for the Composites #1 and #2.

| Samples     | Relaxation      | \( E_a \)(KJ mol\(^{-1}\)) | \( \tau_0 \)(s) |
|-------------|-----------------|----------------------------|-----------------|
| Composite#1 | α relaxation     | 136.02                     | 10\(^{-20.96}\) |
|             | Conduction      | 96.33                      | 10\(^{-15.47}\) |
| Composite#2 | α relaxation     | 132.694                    | 10\(^{-20.73}\) |
|             | MWS             | 82.711                     | 10\(^{-13.65}\) |
\[ M’ = M_o \varepsilon \frac{[ M_o A^\beta + (M_c - M_o) \cos \beta \varepsilon] A^\beta}{M_o^2 A^\beta (M_o - M_c) M_o \cos \beta \varepsilon + (M_c - M_o)^2} \]  
(7)

\[ M^* = M_o \varepsilon \frac{[ (M_c - M_o) \sin \beta \varepsilon] A^\beta}{M_o^2 A^\beta (M_c - M_o) M_o \cos \beta \varepsilon + (M_c - M_o)^2} \]  
(8)

Where

\[ M_o = \frac{1}{\varepsilon_o} \]  
(9)

\[ M_c = \frac{1}{\varepsilon_c} \]  
(10)

\[ A = \left[ 1 + 2 (\omega \tau)^{1-\alpha} \sin \frac{\alpha \pi}{2} + (\omega \tau)^2 (1-\alpha) \right]^\frac{1}{2} \]  
(11)

\[ \varphi = \arctg \left[ \frac{(\omega \tau)^{1-\alpha} \cos \frac{\alpha \pi}{2}}{1 + (\omega \tau)^{1-\alpha} \sin \frac{\alpha \pi}{2}} \right] \]  
(12)

Accordingly, the dotted curves are produced by the best fitting experimental points using the Havriliak–Negami equations (7, 8). In Figure 4-(a, b), it is illustrated that it is impossible to fit the Havriliak–Negami model to all the experimental points for these composites. So, two semicircles are obtained in every examined temperature. This analysis confirms the presence of the MWS relaxation which is overlapped by dc conductivity effect in composite #1. The parameters evaluated by fitting data are listed in Table 2. To determine the parameters characteristics of the Havriliak and Negami model (\( \alpha, \beta, M_s, M_c \)), the experimental \( M’ \) and \( M^* \) data are smoothed through a numerical simulation in the complex plane. The purpose of such simulation is to find the theoretical values (\( M’_{th}, M^*_{th} \)). Concerning the values of \( \alpha, \beta, M_s \) and \( M_c \) which smoothed best the Havriliak–Negami data, they are

![Figure 4](image-url)

**Figure 4** Argand’s plots of \( M^* \) of the composites #1(a) and #2 (b) at 150°C. obtained by a successive approach method where the following expressions (13, 14) are minimized:

\[ \chi^2_{M’} = \sum (M’_{th} - M’_{exp})^2 \]  
(13)

\[ \chi^2_{M^*} = \sum (M^*_{th} - M^*_{exp})^2 \]  
(14)

It has been proven that only one-quadruplet value is able to tone with these conditions. While the values of \( \alpha \) and \( \beta \) obtained for the conductive effect fit with a pure Debye-type, the values of \( \alpha \) and \( \beta \) obtained for interfacial polarization are in accordance with the Havriliak–Negami response.
Table 2. Parameters evaluated by data fitting according to the Havriliak–Negami equation for the composites #1 and #2.

| Samples    | T(°C) | relaxation | α     | β     | M₀    | M∞    |
|------------|-------|------------|-------|-------|-------|-------|
| Composite #1 | 130   | cond       | 0.9915 | 0.90  | 0.002 | 0.295 |
|            |       | MWS        | 0.901  | 0.88  | 0.185 | 0.3191|
|            | 140   | cond       | 0.995  | 0.93  | 0.002 | 0.298 |
|            |       | MWS        | 0.999  | 0.92  | 0.21  | 0.316 |
|            | 150   | cond       | 0.999  | 0.904 | 0.0033| 0.301 |
|            |       | MWS        | 0.99   | 0.863 | 0.153 | 0.3182|
| Composite #2| 130   | cond       | 0.995  | 0.83  | 0.005 | 0.32  |
|            |       | MWS        | 0.92   | 0.892 | 0.199 | 0.315 |
|            | 140   | cond       | 0.999  | 0.819 | 0.00399| 0.3269|
|            |       | MWS        | 0.92   | 0.892 | 0.199 | 0.315 |
|            | 150   | cond       | 0.945  | 0.879 | 0.003 | 0.33  |
|            |       | MWS        | 0.92   | 0.892 | 0.199 | 0.315 |

In order to probe the interface between the matrix and the fibres, we have calculated the strength of the MWS relaxation in permittivity values defined by ($\Delta \varepsilon = \varepsilon_s - \varepsilon_\infty$) [10]. The obtained values for both composites are presented in Table 3. We can notice that the relaxation strength of MWS relaxation increases with the increase in temperature in both composites. This can be ascribed to the increase of free charges that come to be blocked in greater numbers at the interfaces, thus increasing the aptitude of the dipoles to be polarized. Furthermore, the strength of this relaxation is lower in the composite #1 compared to that of the composite #2 at each temperature. A similar trend is seen after a chemical treatment of alfa fibres indicating a rigidification of fibre-matrix interfacial region which in turn reduce the ability of dipole to relax [11] and then enhance the compatibility and the adhesion between epoxy matrix and alfa fibres. As in the composite #1, the relative volume fraction of the Pe-Pet thermo binder fibres is twice the one of wool fibres contrary to the composite #2 in which the relative volume fraction of wool fibres is twice the one of thermo binder Pe-Pet fibres. The latter can enhance the adhesion of the fibres in the matrix. Contrary to the wool fibres which can reduce this interaction and this can be attributed to its hydrophilic character. Indeed, while the dielectric study has revealed the presence of the polarization of the water dipoles at low temperatures in the composite #2, this dielectric relaxation is absent in the composite #1.
Table 3. Interfacial relaxation strength of the relaxation MWS

| Samples       | T(°C) | Δε  |
|---------------|-------|-----|
| Composite #1  | 120   | 1.85|
|               | 130   | 2.27|
|               | 140   | 1.59|
|               | 150   | 3.39|
| Composite #2  | 120   | 2.69|
|               | 130   | 4.11|
|               | 140   | 6.00|
|               | 150   | 5.67|

3.2. Tensile properties

The interface between reinforcing fibre and the matrix plays a critical role in the mechanical performance of composites. A weak interface generally results in low strength and stiffness but high resistance to fracture, whereas a strong interface produces high strength and stiffness, but often low resistance to fracture. Figure 5-(a-c) shows the Young’s modulus, strength, and stress at break of the composites #1 and #2, respectively. From these histograms, we can see that the Young’s modulus of the composite #1 is superior to that of the composite #2. This shows a good cohesion of the materials to transfer stress from the matrix to the fibres [12]. However, the strength and stress at break of the composite #2 is superior to that of the composite #1. In a hybrid composite, the properties of the composite are mainly dependent on the modulus and the percentage elongation at break of the individual reinforcing fibres. The modulus of the Pe-Pet thermo binder fibre is comparatively higher than that of the alfa and the wool fibres [13], whereas the extensiveness of alfa fibre is low compared to the Pe-Pet thermo binder fibre and the wool fibres [13]. This results in early Alfa fibre failure which transfers stress to the stronger thermo binder and wool fibres. As the Pe-Pet thermo binder fibre is less extensive than that of wool fibre, it leads to its failure and it transfers high stress to the less strong wool fibres. This leads to the failure of the wool fibres resulting in the failure of the composite.

![Figure 5](image)

Figure 5 (a) Young’s modulus, (b) tensile strength at break, and (c) stress at break of the composites #1 and #2.

Enhancement of the tensile strength of the composite #2, i.e. at low volume fraction of the Pe-Pet thermo binder fibre loadings, will be due to the fact that at this particular composition, the wool fibre can effectively transfer the load from the Pe-Pet thermo binder fibre. Hence, comparatively less
volume faction of Pe-Pet thermo binder fibres along with the wool fibres reinforcement give rise to a composite with high tensile strength, which in turn can result in an enhancement of the stress at break. A similar trend was obtained in the tensile strength of the sisal/glass hybrid polyester composite [14]. Accordingly Misha et al. have proven that the improvement in tensile strengths of the composites at low wt.% of glass fibre loadings will be due to the fact that at these particular compositions, the sisal fibre can effectively transfer the load from the glass fibre. The determination of the percentage elongation at break of the composites #1 and #2, as mentioned in Table 4, reveals that the elongation of the composite #1 is inferior to that of the composite #2 and this can be explained by a strong interface fibres/matrix [15, 16]. Indeed, a better adhesion of fibres in the matrix prevents sliding effect of the reinforcement in the matrix under the effect of stress. The tensile performance of the composite #1 becomes more marked than that of the composite #2 when specific properties are compared, as illustrated in Table 4. The difference between the specific stresses of both composites is lowered whereas that of the specific Young’s modulus is increased in favour of the composite #1. So the adhesion of the reinforcement in the matrix is improved by the increase in the volume fraction of Pe-Pet thermo binder fibres.

| Samples | ε (%) | σt(MPa) | σt/ρ (MPa cm³/g) | Ei(GPa) | Ei/ρ (GPa cm³/g) |
|---------|-------|---------|------------------|---------|------------------|
| #1      | 4.70±0.23 | 12.19±0.61 | 12.0±0.6 | 0.936±0.046 | 0.92±0.05 |
| #2      | 11.15±0.55 | 19.05±0.95 | 15.8±0.8 | 0.707±0.035 | 0.58±0.03 |

In table 4, σt, ρ and Ei are the stress at break, the density and the Young’s modulus of the composites, respectively.

4. Conclusion
The dielectric study of the composite #1 substantiates the presence of three dielectric relaxations, which were attributed to the α relaxation of the polymer, the ionic conduction which occurred above the glass transition and at low frequencies and the interfacial polarization effect, respectively. The dielectric study of the composite #2 have revealed, in addition, the presence of water dipoles polarisation which could diminish the adhesion of the reinforcement in the matrix as demonstrated by the interfacial polarisation analysis. The correlation of these dielectric properties to the tensile ones has confirmed that the increase of relative volume fraction of Pe-Pet thermo binder fibres to that of wool fibres could enhance the adhesion of the reinforcement in the polyester matrix.

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