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EFFECT OF CONCEPTION PARAMETERS OF NON-WOVEN ALFA FIBRES ON THEIR ADHESION IN UNSATURATED POLYESTER MATRIX

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ABSTRACT

The adhesion of non-woven Alfa fibres in 'NORSODYNE H13372TAE' unsaturated polyester matrix was studied using a dielectric spectrometer and Scanning Electron Microscope (SEM) observations. Non-woven Alfa fibres were obtained either by a reinforcement sheet of Alfa and wool fibres in a ratio of a relative volume fraction 4:1 or by a reinforcement sheet of Alfa, wool and PET-PE thermo-binder fibres in a ratio of a relative volume fraction 17:2:1. The needle punching passages were varied in the reinforcement comprising Alfa and wool fibres, whereas the Shirley analyzer passages were varied in the reinforcement made up of Alfa, wool and thermo-binder fibres. The dielectric analysis revealed the absence of the water dipoles polarisation when increasing the number of passages of these two parameters. Such results were explained by a less hydrophilic character of Alfa fibres. As for the analysis of dielectric relaxations at high temperatures using the Havriliak-Negami model, it allowed the investigation of the interfacial region by means of the interfacial polarisation effect. Less passages of the needle punching has evidenced a better fibres/matrix adhesion. Nevertheless, in the case of increasing the number of Shirley analyzer passages passages, the conductivity effect overlapped with the interfacial polarization effect, which was explained by the change in the adhesion mechanism. SEM observations confirmed these dielectric analyses.

KEYWORDS

Adhesion; Alfa fibres; non-woven fibres; Dielectric properties; SEM

1. INTRODUCTION

Natural fibres have been used as reinforcements in composite materials in order to promote environmental friendly products [Krawczak, 2008]. These fibres are likely to compete with the traditional synthetic fibres thanks to their interesting specific mechanical properties and and replace them in some interesting applications [Wambua et al., 2003]. Indeed, these natural fibre composites have shown their great potential as structural materials due to their low density and low abrasiveness [Pinto et al., 2014]. However, natural fibres are known by their high moisture absorption that can limit their use in high performance structural

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composite applications such as aerospace, but they can serve some other needs such as interior parts of automobiles, furniture's, partitions etc. [Hasan, Rayyaan, 2014]. The hydrophilic character of natural fibres makes them incompatible with hydrophobic polymeric matrices [Oksman, 2003] leading to poor fibres/matrix adhesion [Kaddami et al., 2006]. Taking into account the chemical and physical structures of natural fibres, different chemical and physical surface treatments, such as alkali treatment, permanganate, peroxide, silane oxidization [Kalia et al., 2009] and physical corona discharge treatment [ragoubi et al., 2010], respectively, were applied so as to improve the fibres/matrix adhesion. Furthermore, the incorporation of several and diverse types of fibres into a single matrix has led to the development of hybrid composites. The hybrid effect has been used to describe the phenomenon of an apparent synergistic improvement in the properties of a composite containing two or more types of fibres [Jones, 1994]. Many techniques have been used to give evidence for the effect of these treatments on the fibres/matrix interfacial adhesion [Triki et al., 2011, Triki et al., 2014], among which the dynamic mechanical analysis and scanning electron microscope (SEM) observation are the most used. Nonetheless, the recourse to other methods, able to explore the interface, still draws the attention of many researchers and should complement the analyses done by the other techniques mentioned previously.
Alfa plants grow in North Africa. In Tunisia, they are located at the centre. They are mostly used in the production of high quality papers used for decoration, cigarettes and dielectric applications for condensers [ben Brahim, Ben Cheikh, 2007]. A great deal of recent research has been carried out to promote Alfa fibres in reinforced polymeric systems [Bessadok et al., 2009, Ben Abderrahmane, Ben Cheikh, 2008, Ghallabi, 2010], which can be applied in automotive industry. In fibre reinforced composites, fibres can be reinforced in polymer matrix in different forms and structures [Hasan, Rayyaan, 2014]. Textile Performs are the most economical technique used to make structures from fibre strands using different traditional textile techniques and machinery [Hasan, Rayyaan, 2014].This is the most effective way of handling fibres without any distortions before impregnation in resin [Zoghi, 2013]. Many researchers have studied studied the effect of the number of needle punching passages on the physical and mechanical properties of non woven Alfa-wool [Ghali et al, 2014].

In this study, composite materials using different non-woven Alfa matt have been used to analyse their adhesion in the unsaturated polyester resin. In this study, two sets of composites were elaborated. In one of the two sets, non-woven Alfa matts were obtained by Alfa-wool blend in a ratio of a relative volume fraction 4: 1, and in each obtained composite, the reinforcement differs by the number of needle punching passages. Yet, in the other set, non-woven Alfa matts were obtained by Alfa-wool and -PET-PE thermo-binder blend in a ratio of a relative volume fraction 17:2:1, and in each obtained composite, the reinforcement differs by the number of Shirley analyzer passages. The focus of this study is to determine the effect of these conception parameters on fibres/matrix adhesion. For this purpose, dielectric measurements were performed on these composites in the temperature range of 40-150°C and frequency range of 0.1-10^6 Hz. The dielectric relaxations at high temperatures were analyzed using the Havriliak-Negami model allowing the examination of the interfacial polarization in order to investigate the interfacial region between fibres and matrix. This analysis was achieved by a thorough study of the fibres/matrix interfacial adhesion aspect, by means of scanning electron microscope observations.

2. MATERIALS AND METHODS

2.1 Materials

The matrix used in this study is the same one as that used in our previous studies [Triki et al., 2011]. To elaborate non-woven Alfa fibres, four different reinforcements were manufactured depending on the hybrid character of the reinforcement on the one hand and on the conception parameters of the reinforcement on the other.. For the reinforcements (1) and (2), Alfa fibres were mixed with wool fibres in a ratio of a relative volume fraction 4:1. To prepare these sheets of non-woven fibres, different steps were followed as described with details in a previous research [Triki et al., 2011]. However, these reinforcements vary according to the number of the needle punching passages. Indeed, while two passages were used for the reinforcement (1), four passages were used in the reinforcement (2). In the case of the reinforcements (3) and (4), Alfa fibres were mixed with wool fibres and thermo-binder fibres (PET-PE) in a ratio of a relative volume fraction of Alfa to wool to PET-PE of 17:1:2. Different steps were followed to manufacture these reinforcements as described in a previous work [Triki et al., 2013]. These reinforcements differ in the number of the Shirley analyzer passages. In fact, only one passage was needed for the reinforcement (3), whereas two passages were needed for the reinforcement (4). All composites were manufactured using the classical 'contact mould method' [Kaynak, Akgul, 2001]. The fibres volume fraction of each composite was calculated using the following formula [Krawczak, 1997]:

$$\frac{1}{\varphi_V} = \frac{\rho_r}{\rho_m}\left(\frac{1}{\varphi_P} - 1\right) + 1 \quad (1)$$

where ρ_r is the volume weight of the reinforcement, ρ_m is the volume weight of the matrix and φ_p is the weight fraction of the composite. The obtained fibres volume fractions were 7.4%, 6.2%, 5.2% and 6% for the composites # 1, #2, #3 and #4, respectively. These latter were reinforced by the reinforcements (1), (2), (3) and (4), respectively. The low volume fractions allow manufacturing composites with low thicknesses as they will serve as surface coatings for the inner part of buses. These composites are illustrated in Figure 1.

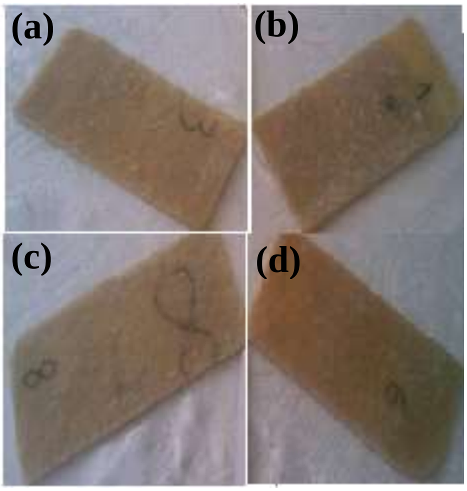

Figure 1: Photos of the composites #1 (a), #2 (b), #3 (c) and #4 (d).

2.2 Methods

Alpha Dielectric/impedance Analyser (Novocontrol) was used to carry out the dielectric measurements of each sample in the temperature range from 40 to 150°C and the frequency interval from 10^{-1} to 10^6 Hz. These experiments were conducted in isothermal runs with fixed temperatures and scanning frequencies from 10^{-1} to 10^6 Hz.

The shapes of dielectric relaxation processes of polymers could be represented by the following expression given by Havriliak and Negami [Havriliak Jr, Watts, 1986]:

$$\frac{\varepsilon^*(\omega) - \varepsilon_\infty}{\varepsilon_S - \varepsilon_\infty} = [1 + (j\,\omega\,\tau)^\alpha)]^{-\beta} \quad (2)$$

In this expression $\varepsilon^*(\omega)$ is the complex dielectric constant measured at the radian frequency $\omega = 2\pi f$, where f is the oscillator frequency in Hz. The quantities ε_∞ and ε_S represent the instantaneous and equilibrium dielectric constants, respectively. The parameters α and β are formally related to the distribution of relaxation times, [Havriliak Jr, Watts, 1986] while τ is the relaxation time. This expression has the significance that no new parameters were needed to represent the data: when α = 1 the Cole-Davidson [Davidson, Cole, 1951] expression is obtained, and when β = 1, the the Cole-Cole [Cole, Cole, 1941] expression is obtained. But, when α and β = 1, this equation reduces to the Debye equation.
Given a data set consisting of the real and imaginary values at various frequencies, the parameters were estimated graphically coupled with a objective evaluation of the fit in the complex plane (Argand representation).

The observations of the cross section surface of each composite conducted using the scanning electron microscope PHILIPS XL30 so that the interfacial region fibres/matrix of each composite could be analyzed.

3. RESULTS AND DISCUSSION

3.1 Dielectric analysis

Effect of the needle punching:

The isothermal runs of the real part of the permittivity ε' are depicted in Figures 2-(a) and (b) for the composites #1 and #2, respectively, for different temperatures varying from 40 to 150 °C in increments of 10°C.

It can be noted that the permittivity is steadily constant and equal to that of the matrix [Triki et al., 2011] at low temperature and low frequencies for the composite #2. However, it increases with the decrease in frequency for the composite #1 at low temperature. So these results indicate the presence of water dipoles polarization for the composite #1. Similar results were obtained on the palm trees fibres reinforced polyester matrix [Ben Amor et al., 2009]. As temperature increases, ε' increases due to the enhanced conductivity of the composites at higher temperatures. As a result, the increase in the number of the needle punching passages, which decreases the thickness of the obtained sheet can decrease the hydrophilic character of Alfa fibres. This result can be explained by the enhancement of hydrogen bonds which can occur between hydroxyl groups on adjacent cellulose surfaces [Fornué et al., 2011]. Similar results were obtained in our previous work [Karray et al., 2014]. Accordingly, this adhesion can be enhanced by the mechanical interlocking caused by irregular surfaces and intermolecular diffusion caused, in turn, by molecular chains interacting between the cellulosic surfaces [Gardner et al., 2008]. Such adhesion can decrease the hydrophilic character of Alfa fibres reinforcement.

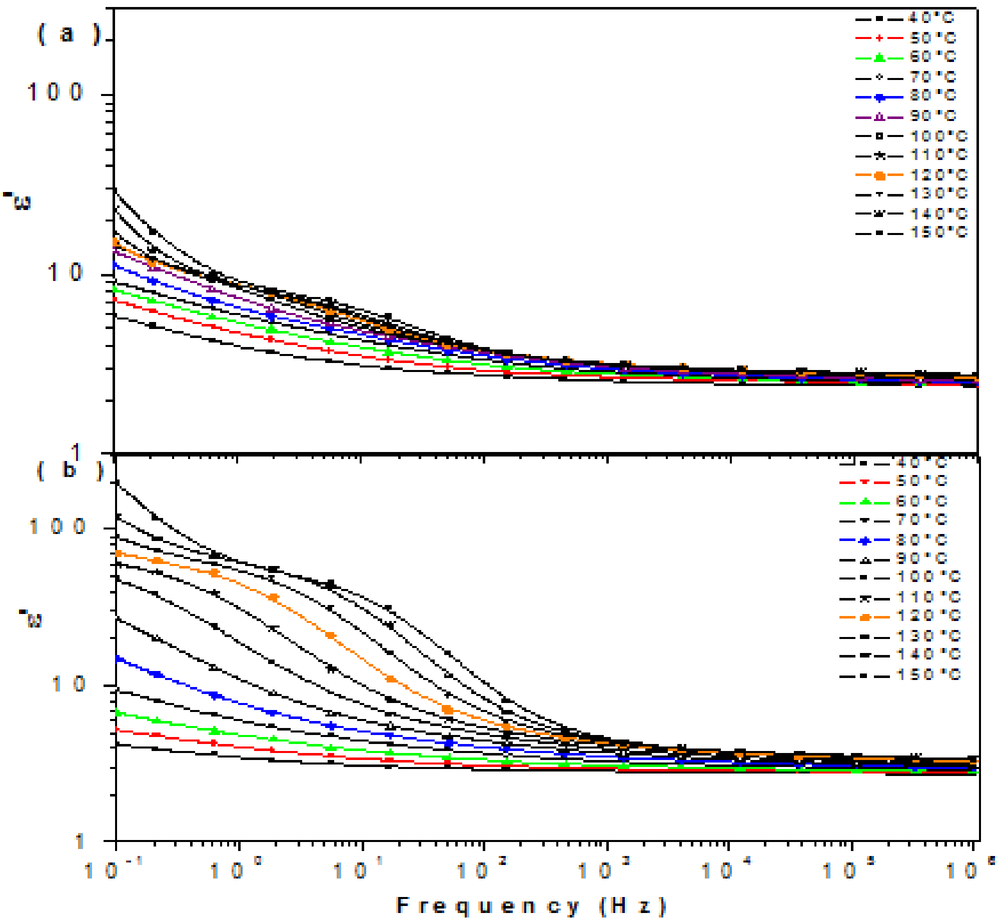

Figure 2: Isothermal runs of the dielectric permittivity ε' versus frequency of the composites #1 (a) and #2 (b).

Effect of the Shirley analyzer:

Figures 3-(a) and (b) illustrate the isothermal runs of the permittivity ε' for the composites #3 and #4 for different temperatures varying from 40 to 150 °C in the increments of 10°C. At low temperature, the permittivity increases when lowering the frequency for the composite #3. However, it is steadily constant in the whole frequency range for the composite # 4. Hence, water dipoles polarization is present in the composite #3. As temperature increases, ε' increases due to the enhanced conductivity of the composites at higher temperatures and low frequencies. The means diameters of the obtained Alfa fibres were about 204.86 μm and 89.95 μm for the reinforcements (3) and (4), respectively. So, the decrease of the Alfa fibres diameter may decrease its hydrophilic character. Indeed, Raman spectroscopy results performed on these Alfa fibres have shown a decrease of the vibrations intensities associated with the lignin [Omri et al., 2013, Omri et al., 2015].

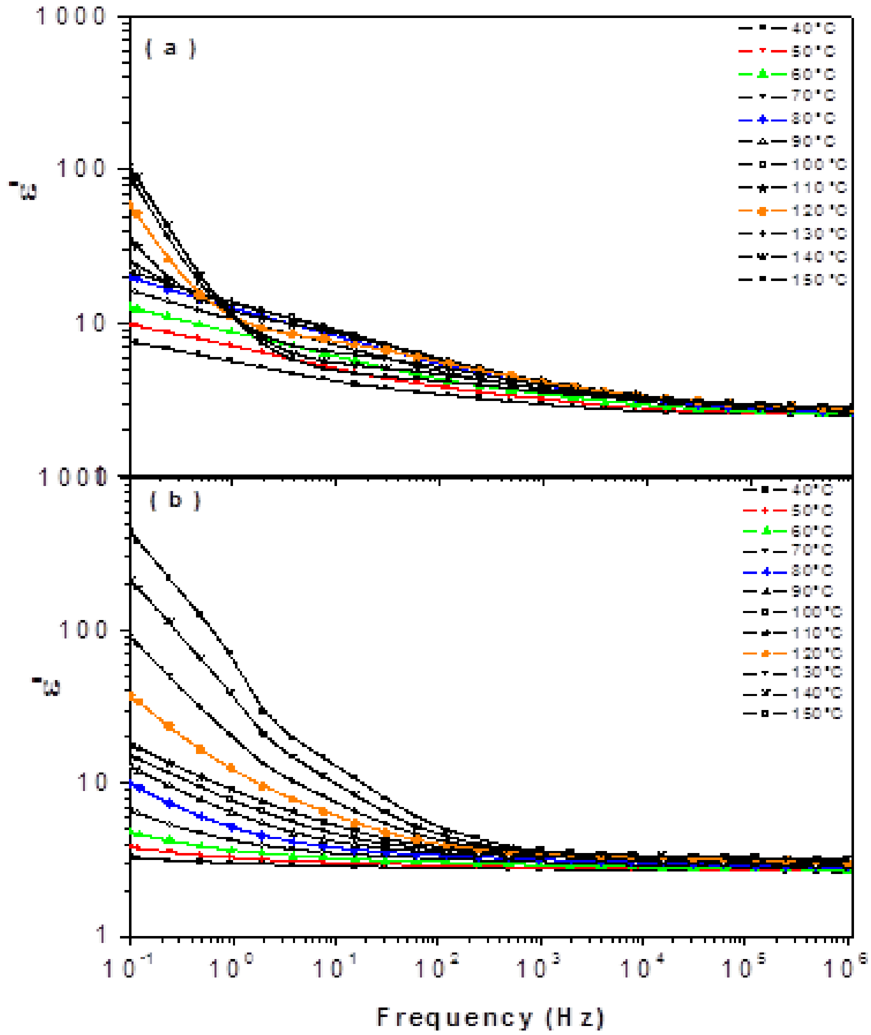

Figure 3 : Isothermal runs of the dielectric permittivity ε' versus frequency of the composites #3 (a) and #4 (b).

Analysis of the reinforcement adhesion in the matrix:

In order to study the effect of the hydrophilic character on the adhesion of the reinforcement in the matrix, the electric formalism was used. This latter is defined by the following Eq. 3 [Howard, 1992]:

$$M^* = \frac{1}{\varepsilon^*} = \frac{1}{\varepsilon' - j\varepsilon''} = \frac{\varepsilon'}{\varepsilon'^2 + \varepsilon''^2} + j\frac{\varepsilon''}{\varepsilon'^2 + \varepsilon''^2} = M' + j\,M'' \qquad (3)$$

where ε' and ε'' are the real and imaginary parts of the complex dielectric constant,respectively. M' and M'' are the real and the imaginary parts of the electric modulus, respectively. The use of the Argand representation (Cole-Cole diagram) provides interesting knowledge regarding the nature of relaxation. Cole-Cole plots of the composites at 150 °C are depicted in Figures 4-(a) and (b) and 5-(a) and (b). Havriliak–Negami model fits correctly the data found in the literature [Havriliak, Negami, 1966, Wagner, H. Richert, R. 1999]. In the electric modulus formalism, the Havriliak–Negami equations (4, 5) are as follow [Havriliak, Negami, 1966]:

$$M' = M_\infty \frac{[\,M_S\, A^{\beta} + (\,M_\infty - M_S)\cos\beta\varphi\,]\, A^{\beta}}{M_S^2\, A^{2\beta}\,(M_\infty - M_S) M_S \cos\beta\varphi + (\,M_\infty - M_S\,)^2} \quad (4)$$

$$M'' = M_\infty\, M_S \frac{[\,(\,M_\infty - M_S)\sin\beta\varphi\,]\, A^{\beta}}{M_S^2\, A^{2\beta}\,(M_\infty - M_S) M_S \cos\beta\varphi + (\,M_\infty - M_S\,)^2} \quad (5)$$

where $$M_S = \frac{1}{\varepsilon_S} \quad (6)$$

$$M_\infty = \frac{1}{\varepsilon_\infty} \quad (7)$$

$$A = [\,1 + 2\,(\omega\,\tau)^{1-\alpha} \sin\frac{\alpha\,\pi}{2} + \,(\,\omega\tau)^{2\,(\,1-\,\alpha)}]^{1/2} \quad (8)$$

$$\varphi = \text{arctg}\ \left[\frac{(\omega\,\tau)^{1-\alpha}\cos\frac{\alpha\,\pi}{2}}{1 + (\omega\tau)^{1-\alpha}\, sin\frac{\alpha\,\pi}{2}}\right] \quad (9)$$

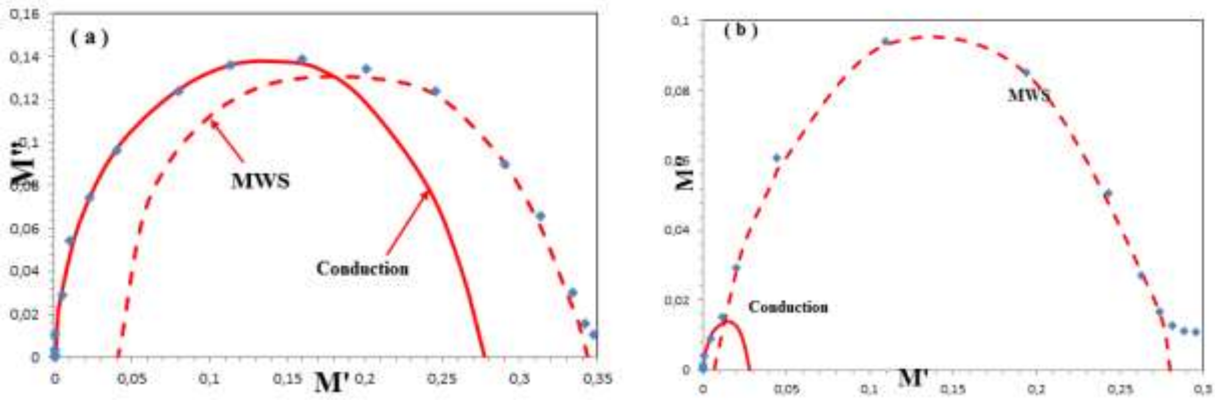

Figure 4: Argand's plots of the electric modulus, M*, of the composites #1 (a) and # 2 (b) at 150°C.

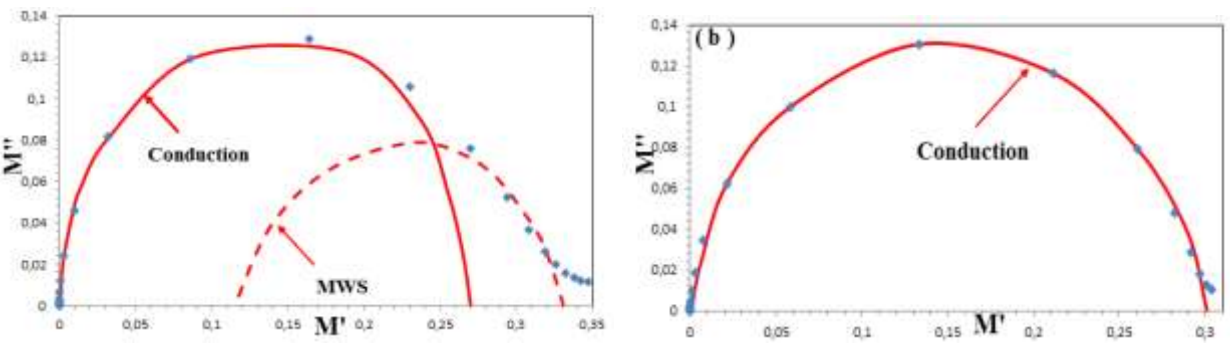

Figure 5: Argand's plots of the electric modulus, M*, of the composites #3 (a) and #4 (b) at 150°C.

Accordingly, the dotted curves are produced by the best fitting experimental points using the Havriliak–Negami equations (4, 5). It is to be noted that it is impossible to fit to the Havriliak–Negami model with all the experimental points for the composites #1, #2 and #3. Similarly, it can be noted that at low frequencies domain (lower values of M' and M''), the experimental points reach the origin of the graph, which is a typical behavior of dc conduction effect. Therefore, two semicircles are obtained in every examined temperature. The first one is related to the conduction effect and the second is attributed to the interfacial polarisation effect (MWS). The parameters evaluated by fitting data were listed inTable 1. To determine the parameters characteristic of the Havriliak and Negami model (α, β, M_s, M_∞), 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}). The values of α, β, M_s and M_∞, which adjust the best to the Havriliak–Negami data are obtained by a successive approach method where the following expressions (10, 11) are minimized:

$$\chi^2_{M'} = \sum_i \left(M'_{th} - M'_{exp}\right)^2 \quad (10)$$

$$\chi^2_{M''} = \sum_i \left(M''_{th} - M''_{exp}\right)^2 \quad (11)$$

It has been proven that only one-quadruplet value is able to tone with these conditions.

The values of α and β obtained for the conductive effect are in harmony with a pure Debye-type for all composites. While, the values of α and β obtained for the interfacial relaxation go in the same line with the Havriliak-Negami response. However, in the case of composite #4, all experimental values are fitted according to Havriliak-Negami model. So, the obtained semicircle is attributed to the dc conductivity effect. The values of α and β are in harmony with a pure Debye-type. Hence, the interfacial polarization effect is hidden by the conductivity effect of the composite #4.

Table 1: Parameters evaluated by fitting data according to the Havriliak–Negami equation for the composites

Composires	T(°C)	Relaxation	α	β	M_s	M_∞
#1	130	Conduction	0.99912	0.97499	0.00086	0.2549796
		MWS	0.90	0.913	0,1085	0.3362
	140	Conduction	0.999988	0.99999	0.00153	0.2465
		MWS	0.912	0.951	0.05	0.3389
	150	Conduction	0.99956	0.999	0.00091	0.2772
		MWS	0.994	0.8975	0.041	0.3437
#2	130	Conduction	0.984	0.992	0.000391	0.035
		MWS	0.86	0.88	0.012	0.26
	140	Conduction	0.982	0.98	0.00028	0.023
		MWS	0.86	0.874	0.0077	0.27
	150	Conduction	0.988	0.994	0.00028	0.028
		MWS	0.865	0.88	0.0068	0.28
#3	130	Conduction	0.9641	0.97	0.00066	0.2285
		MWS	0.74	0.891	0.14	0.33
	140	Conduction	0.9925	0.99	0.0005	0.23
		MWS	0.9	0.826	0.11	0.32415
	150	Conduction	0.999	0.994	0.0005	0.27
		MWS	0.87	0.931	0.115	0.331
#4	130	Conduction	0.992	0.907	0.000899	0.291
	140	Conduction	0.9919	0.94	0.00089	0.301
	150	Conduction	0.9917	0.91	0.00089	0.301

The interfacial relaxation strength was calculated according to the equation $\Delta\varepsilon = \varepsilon_s - \varepsilon_\infty$ [Puertolas, 1999] for all composites, #1, #2 and #3 at different temperatures and presented in Table 2. The analysis of these values has demonstrated that the interfacial relaxation strength increases with temperature according to each composite. This can be due to the increase in free charges, which was found to be blocked in greater numbers at the interfaces, thus increasing the aptitude of the dipoles to be polarized. In addition, the comparison of of the different composites has shown that the adhesion of the reinforcement in the matrix was better for composite #1 than for composite #2. Hence, wool fibres contribute to the adhesion of Alfa fibres in the unsaturated polyester matrix for the composite #1[Omri et al., 2015].

Table 2: Interfacial relaxation strength

Composites	T(°C)	Δε
#1	130 140 150	6.24 17 21.48
#2	130 140 150	79 126 143
#3	130 140 150	4 6 5.7

Nevertheless, in the case of composites #3 and #4, the adhesion mechanism was different as the interfacial polarization effect was detected only for composite #3. Indeed, the vibrational study performed on these composites has revealed that the adhesion mechanism was based on covalent bonds and hydrogen bridges for composite #3 [Triki et al., 2014] but only on chemical bonds formed by secondary bonding for composite # 4 [Omri et al., 2013]. Furthermore, the composite # 3 presented the lowest values of the interfacial relaxation strength. Hence, the addition of the PET-PE thermo-binder fibres in the reinforcement proved to increasingly improve this adhesion.

3.2 SEM observation

To further support our dielectric analysis, SEM micrographs of all composites are shown in Figure 6-(a, b, c, d).

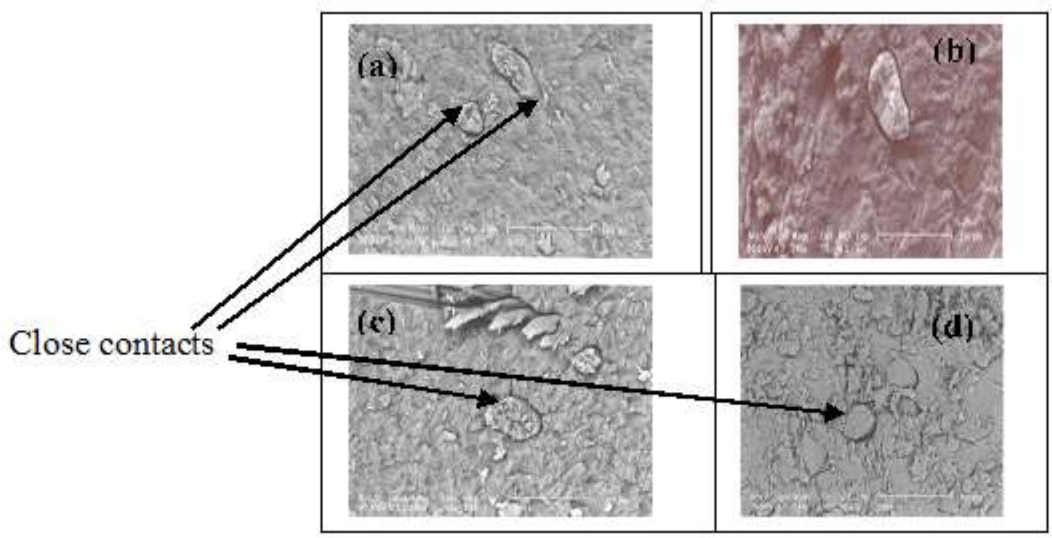

Figure 6: SEM micrographs of the polished cross-sections for the composite #1 (a), #2 (b), #3 (c) and #4 (d).

This figure reveals the existence of close contacts between the fibres and matrix for composites #1, #3 and #4. However these close contacts are fewer for composite #2. These observations confirm the highest values of the interfacial relaxation strength of MWS relaxation obtained for this composite.

4. CONCLUSION

The adhesion of different non-woven Alfa fibres in unsaturated polyester has been analyzed using the dielectric spectrometer and SEM observations. Taking into account of the needle punching passages, the hydrophilic character of the reinforcement may decrease by the increase of its passages numbers. But, the latter would diminish the contribution of wool fibres in the adhesion of Alfa fibres in the matrix. The addition of the PET-PE thermo-binder fibres to the reinforcement improved the fibres/matrix adhesion. Nevertheless, the change of the Shirley analyzer passages numbers gave rise to different adhesion mechanisms. These dielectric analyses were confirmed by the SEM observations.

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