MECHANICAL PROPERTIES ANALYSIS OF SHORT HEMP-FIBRE/POLYPROPYLENE COMPOSITES: INFLUENCE OF FIBRE CONTENT AND HYGROTHERMAL AGEING

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Abstract

This paper aims to investigate the effect of hygrothermal ageing on the mechanical properties of short Hemp Fibre Reinforced Polymers (HFRP) composites with different fibre contents (from 10% to 40%). Injected specimens were subjected to hygrothermal ageing with a relative humidity (RH) of 80% and a temperature of 25 °C. The water absorption and its effect on tensile properties of HFRP composites are investigated. The Acoustic Emission (AE) technique combined with electronic microscope observations were carried out to discriminate some damage mechanisms at the microscopic scale. For the proposed hygrothermal ageing of HFRP composites, it is shown that the moisture absorption shows a quasi linear evolution according to the hemp-fibre content. Besides, no significant effects on the mechanical characteristics of the studied composites were observed.

1 Introduction

Today, the request of markets for products more environment-friendly is in strong growth resulting from ecological concern, environmental attentiveness and new rules and regulations. The development of composite materials with vegetal fibres presents numerous advantages because of their biodegradability and of their low density. Besides, vegetal fibres, used as reinforcement of composite materials, constitute an interesting alternative to glass fibres because of their competitiveness in both mechanical performance and price. Vegetal fibres, which originate from renewable resources, are a new perspective to mineral and organic fibres [1]. Hemp fibre is one of the inexpensive and readily available vegetal fibres and hemp-fibre reinforced thermoplastic composites products have gained considerable attraction for automotive products. Even if they are low in strength performance, they have the advantage of design flexibility and exhibit interesting recyclability [2]. However, they present some disadvantages such as their incompatibility with the polymer matrices and their low resistance to moisture.

Many researches have been carried out to investigate the performance of vegetal fibres composites, in particular in a wet environment because of the hydrophilic nature of the fibre. These problems of environmental ageing are essential for the development of eco-composites. Research investigations have shown that the exposure of vegetal fibre composites to a wet environment leads to a decrease of their mechanical properties when water spreads in the material. This loss of properties was noticed on composites with fibres of sisal [3], bamboo [4], flax [5, 6] or hemp [7]. Dhakal et al. [8] showed, in the case of hemp fibre reinforced polyester composites, that the moisture uptake increased with the fibre volume fraction. These authors also showed that moisture induced a significant degradation of their composites mechanical properties at elevated temperature. The mechanical, water absorption, and thermal properties of short hemp/glass fibres reinforced polypropylene hybrid composites was investigated in [9]. The authors observed that thermal properties and resistance to water absorption properties of the hemp fibre composites were improved by hybridization with glass fibres.

The main objective of this work is to assess the mechanical properties of short Hemp-Fibre Reinforced Polymer (HFRP) composites, with different fibre contents (from 10% to 40%), and subjected to an hygrothermal ageing, with a relative humidity of 80% and a temperature of 25 °C. The Acoustic Emission (AE) technique combined with electron microscope observations were used to examine how hygrothermal ageing affects damage mechanisms of composites.

2 Materials and testing methods

2.1 Materials

The tested materials are Hemp-Fibre Reinforced Polymer (HFRP) composites based on short fibres of hemp and polypropylene (PP). They are distinguished by the percentage of fibres weight in the composite. Four materials were used:

- PP-hemp 90/10: PP/hemp composite with 10 wt% hemp fibre
- PP-hemp 80/20: PP/hemp composite with 20 wt% hemp fibre
- PP-hemp 70/30: PP/hemp composite with 30 wt% hemp fibre
- PP-hemp 60/40: PP/hemp composite with 40 wt% hemp fibre

All samples were obtained from AFT Plasturgie® Company (Fontaine-Les-Dijon – France). They are elaborated using the injection moulding process.

2.2 Hygrothermal ageing

Specimens from each batch of tensile samples were subjected to hygrothermal ageing. After being dried during 24 hours, they were put into an environmental test chamber (Binder) with a relative humidity (RH) of 80 % at a temperature of 25 °C. During the wet time and at certain periods of time, specimens were periodically taken out of the chamber. To assess the weight change, they were wiped dry with tissue paper and weighed using an analytical balance (Ohaus Pioneer) having a precision of ±1 mg. The specimens were weighed regularly up to 1200 h exposure. After various periods of time, the water absorption characteristics of the composites were evaluated by the relative uptake of weight defined by M_t according to:

$$\mathrm{M_t} = \frac{W_t - W_0}{W_0} \times 100\ (\%) \tag{1}$$

where W_0 is the weight of dry specimen and W_t is the weight of wet specimen at time *t*.

2.3 Tensile tests

Using a universal mechanical testing machine Instron, the tensile tests were conducted at room temperature, with a cross-head speed of 2 mm.min^{-1}. A clip-on extensometer with 50 mm gauge length was used to measure the longitudinal strain. Five specimens were tested for each set of samples according to ASTM D638M standard and only the mean values are taken into account. To minimize loss of moisture, the specimens were immediately taken out from the environmental test chamber before the tensile testing.

2.4 Acoustic emission

Acoustic emission (AE) is an efficient technique for structural health monitoring which has been frequently used during mechanical tests on traditional polymer composites. AE signal types can be correlated with specific damage phenomena occurring in composites, such as matrix cracking, fibre–matrix debonding, fibre failure, delamination, etc. In this study, two channels data acquisition system from Mistras Group with a sampling rate of 5 MHz and a 40 dB pre-amplification was used to record AE data. AE measurements were achieved by using two piezoelectric sensors with a frequency range 100 kHz–1 MHz, coupled on one of the sides of the specimens with a silicon grease, as described in [9]. In order to better understand the damage, AE signals will be treated in the subsection 3.3 through analyses based on cumulative number of hits and amplitude.

3 Results and discussion

3.1 Water absorption

Figure 1a shows the percentage of water absorption for all by the samples after 80% RH ageing at 25°C, as a function of the ageing time. The water uptake increases in a quasi-linear way with increasing the hemp content as illustrated in Figure 1b. At the end of the 1200 h exposure, the water uptake of each composite doesn't reach an equilibrium plateau. This water uptake is lower than 1 % for the four composites, which shows that few water spreads in the materials. A hygrothermal ageing in higher temperature could induce an increase of diffusivity and moisture content [7, 8].

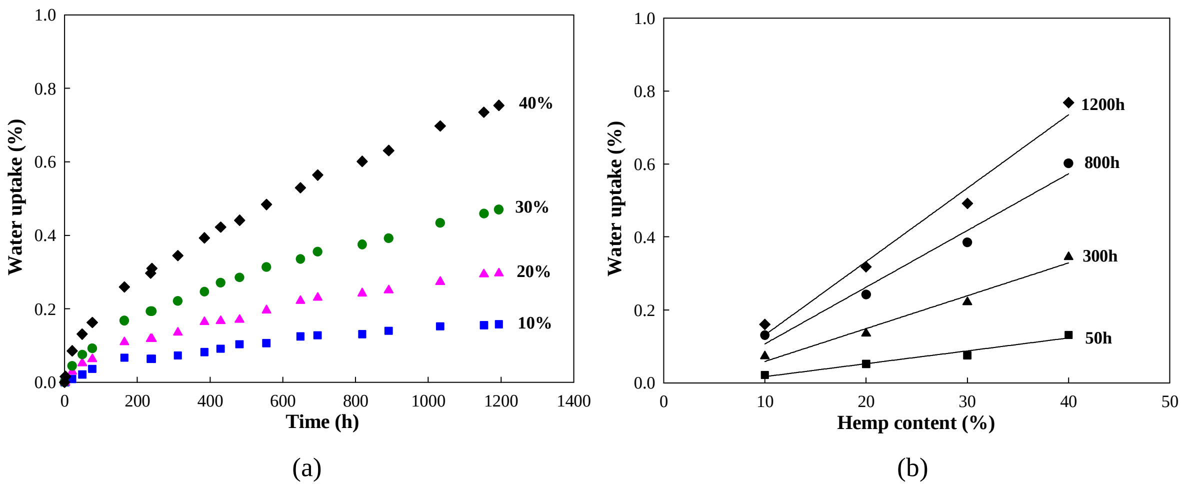


Figure 1. Water uptake of aged HFRP composites according to: a) the ageing time, b) the hemp fibre content .

3.2 Mechanical properties

In order to study the influence of the hemp content on the mechanical behaviour of unaged HFRP composites, figure 2 shows the stress-strain curves in static tensile tests. After a linear behaviour, all curves reveal a substantial non linear response, which is attributed to the matrix plastic deformation. This non linear portion decreases proportionally with increasing the hemp content. For each hemp fibre reinforced PP composite, we remark that the mechanical stress remains approximately constant after reaching the maximum stress while the displacement increases until failure.

To study the influence of the hygrothermal ageing on the mechanical behaviour of aged HFRP composites, figure 3 shows the evolution of the mechanical properties (Young's modulus, maximal stress and maximal strain) for unaged and aged HFRP composites (1200h). As expected, we remark that the Young's modulus increases linearly with the hemp content respecting the law of mixture, while the maximal strain decreases. Moreover, the hemp fibre content has no significant effect on the maximal tensile stress as it remains approximately constant for all composites. Besides, it is interesting to note that, the maximal stress of aged HFRP composites is found to be higher than that of unaged ones except for those with 10% of hemp fibre content. This could be related to the swelling of hemp fibres (due to humidity) which could fill the hemp fibre/matrix gaps and hence results in a moderate increase of the maximal stress.

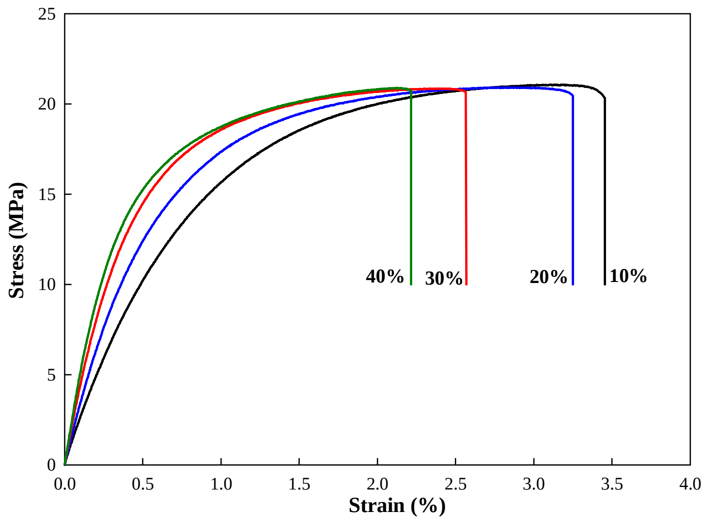
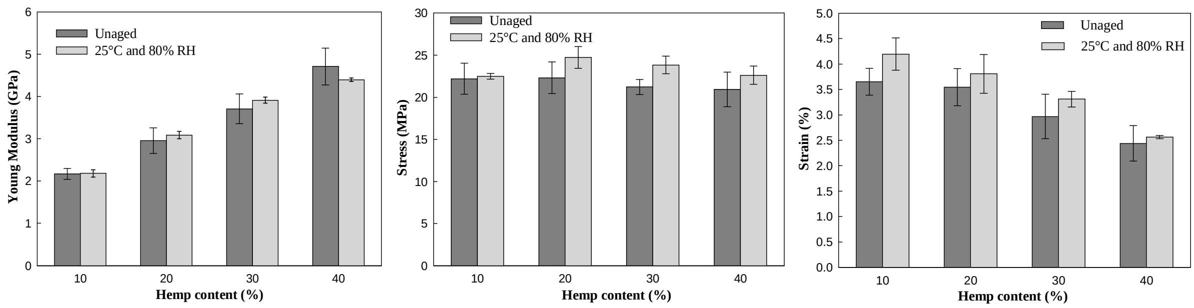


Figure 2. Stress-strain response of unaged HFRP composites under tensile loading.



(a) (b) (c)

Figure 3. Evolution of the mechanical properties of unaged and aged HFRP composites (1200h), according to the hemp content: a) Young's modulus, b) maximal stress, c) maximal strain.

3.3 Damage mechanisms analysis

3.3.1 Acoustic Emission

During the ageing of HFRP composites, several damage mechanisms at the microscopic scale occur. The acoustic signals analysis allows discriminating these different types of damage. In order to get information about these mechanisms, the signals recorded during tensile tests were analysed according to the hits number, the tensile test time and the signal amplitude. Figure 4 shows the amplitudes and cumulated numbers of the AE events as a function of the loading time for all aged and unaged HFRP composites.

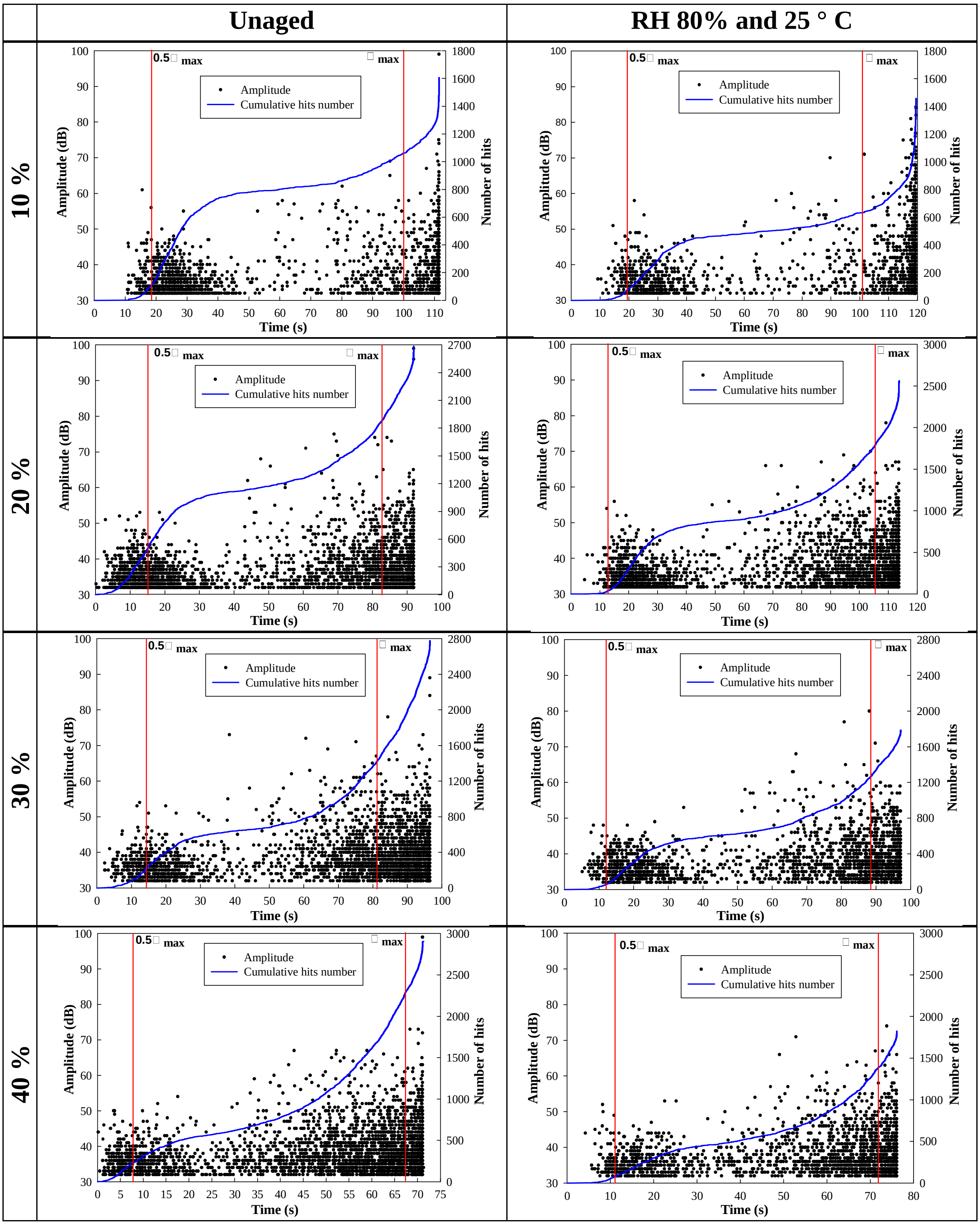


Figure 4. AE amplitude and cumulated number of AE events as a function of loading time for all the composites (Unaged and aged, with hemp content of 10, 20, 30 and 40 %)

The stress-strain curve was divided into the following three ranges on the basis of the failure stress value: (I) 0 – 0.5 $\sigma_{\max}$, (II) 0.5 $\sigma_{\max}$ – $\sigma_{\max}$ and (III) $\sigma_{\max}$ – σ_F as proposed in [10]. The choice of these three ranges is explained by the AE activity and the shape of the cumulative events curves. In fact, about 93% of the AE events number was detected during the second and third stages (between 0.5 $\sigma_{\max}$ and σ_F). Figure 4 shows that the cumulative hits number of AE events increases with increasing the hemp fibre content for both unaged

and aged HFRP composites. In fact, the cumulative hits number of AE signals is found to be approximately equal to 1600 for the unaged 10% HFRP composite, while it is equal to 3000 for the unaged 40% HFRP one. This difference can be due to the increasing of the hemp fibres damage within the composite (fibre/matrix debonding and pull-out of the hemp-fibres).
For aged composites, we observe a decrease on the acoustic activity when compared to the unaged ones. For example, 3000 signals were detected for the unaged 40% HFRP while only 1800 within the aged one. This reduction in acoustic activity is caused by the loss in stiffness of HFRP composites (figure 3a). For the second region (between 0.5 $\sigma_{\max} - \sigma_{\max}$), the curve of cumulative hits number present a linear region with a moderately increase of the slope. The length of this part is found to decrease when the hemp fibre content increases. This is seen for both unaged and aged HFRP composites. Finally, the mean amplitude of AE signals were found to be around 37 dB and 40 dB for the second and third regions respectively, for the aged and unaged HFRP composites, which shows that the damage mechanisms occur essentially within these two regions.

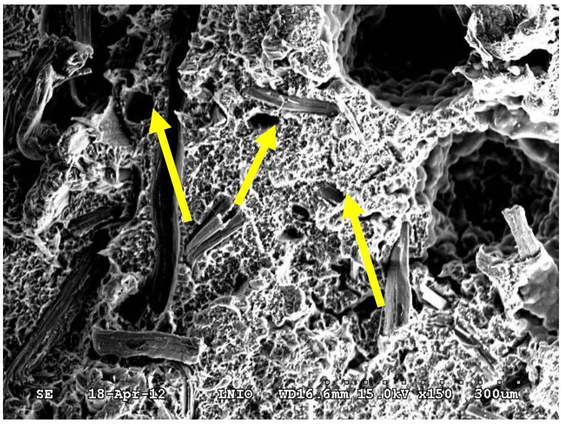
3.3.2 Electron microscope observations

In order to distinguish the different damage mechanisms of the HFRP composites due to the hygrothermal ageing, and occurring at the microscopic scale, the specimens surfaces were examined using a Jeol Scanning Electron Microscopy (SEM). After the tensile test, small sections of the unaged and aged HFRP composites were cut transversely to the beam axes 5 mm away from the failed centre region. Figure 5 shows representative SEM images taken at suitable magnification for each failed specimen. The analysis shows that the failure of the tested unaged and aged composite materials is essentially induced by the following damage mechanisms: the fibre-matrix debonding (figure 4c), the hemp fibre pull-out (figures 4a and 4e) and the hemp fibre fracture (figure 4f). The hemp-fibre failure occurs according to several processes: the pull-out of micro-fibrils (figure 4d) and the explosion of micro-fibrils (figure 4b). These figures also show that many fibre pull-outs occur, as illustrated by the large number of unbroken fibres sticking out of the fracture surfaces, as well as the large number of holes present within the matrix from which the fibres were pulled out. These observations also indicate that the ageing at 80% RH and 25 °C does not induce other damage in the studied HFRP composites. The addition of a coupling agent during the injection molding of these HFRP composites could contribute to improve the interfacial adhesion between hemp fibre and polypropylene matrix.

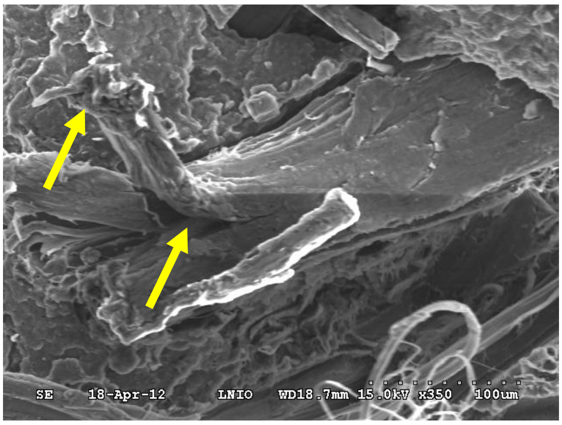
4 Conclusions

This paper presents the results of experimental investigations of the effects of a hygrothermal ageing on HFRP composites. Four hemp fibre contents were considered (10, 20, 30 and 40%) and the hygrothermal ageing was defined by a relative humidity of 80% and 25°C. The static strength and residual stiffness of aged specimens were evaluated with tensile tests after an exposure of 1200h to hygrothermal ageing.
The obtained results show that the mechanical properties (Young’s modulus, maximal stress and maximal strain) of the studied HFRP composites were moderately affected by the chosen hygrothermal ageing (80% RH and 25 °C) except for the Young’s modulus of the aged 40% HFRP. The analysis of the AE signals enabled to identify, on the one hand, the chronology of the various damage mechanisms in HFRP composites and, on the other hand, to characterize the evolution of this chronology according to the hygrothermal ageing. SEM observations allowed a better understanding of this evolution, revealed by the AE signals detected during

the different stress ranges. These observations show the presence of several damage mechanisms such as the fibre / matrix debonding and the hemp fibres pull-outs. Extensions of this work would be to study the compatibility between the hemp-fibres and the polypropylene matrix with the aim of reducing the loss of mechanical properties. It would also be interesting to develop a numerical multi-scale model using a special finite element procedure to take into account several parameters; such as the geometric and mechanical properties of the hemp fibres and those of the matrix as well as the hygrothermal ageing conditions.



(a)



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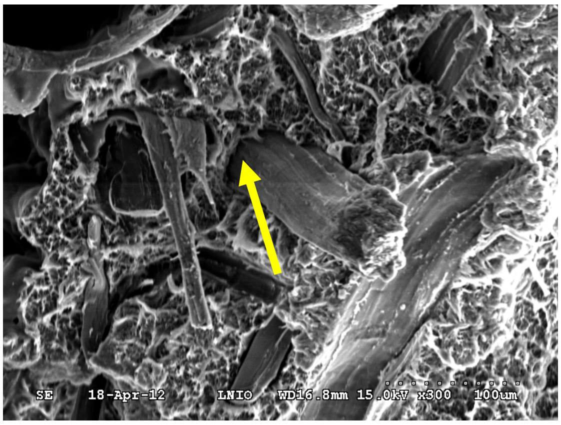

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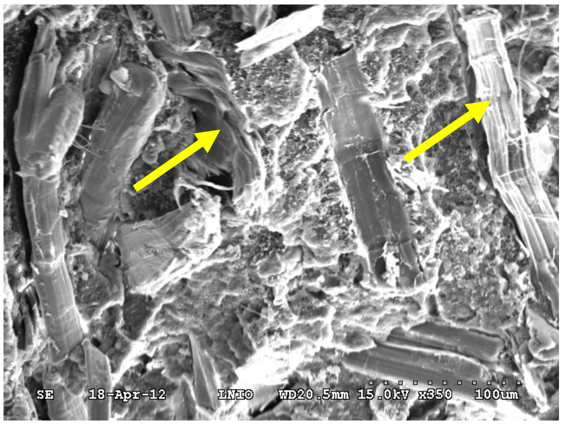

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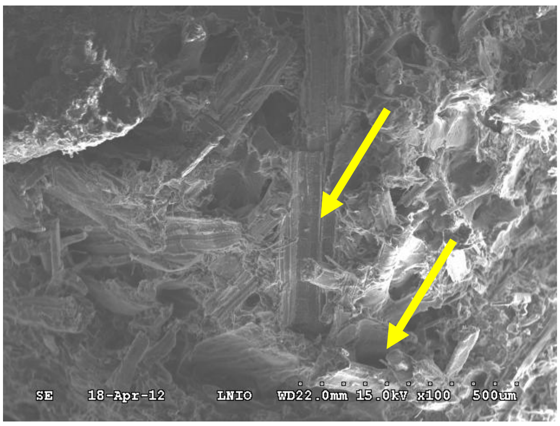

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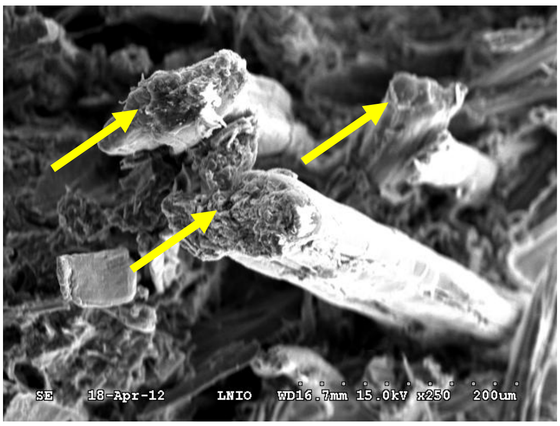

(f)

Figure 5. SEM pictures taken from the fracture surfaces of hemp-fibre reinforced polymer composite: a) 10% unaged, b) 10% aged, c) 20% unaged, d) 20% aged, e) 40% unaged, b) 40% aged.

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