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Estimation of fiber/polymer bond strength from maximum load values recorded in the micro-bond tests

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ABSTRACT

In the last few years, several techniques for separate determination of adhesion and friction in micromechanical tests have been developed but their experimental realization is rather complicated, because they require an accurate value of the external load at the moment of the crack initiation. So, in this perspective, an effort is done to estimate the interfacial parameters between two kind of thermoset resins and the natural Alfa fiber determined from Microbond tests. As known, the diameter of natural fiber is a crucial factor that participates in the estimation of the interfacial adhesion characteristics. In others words, the measurement of the maximum force required to pull out a fiber from the polymeric resins is investigated by using the micro-droplet test.

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1. Introduction

Plant fibers are very commonly used in the manufacture of several high-strength compounds, where metals have been replaced by these advanced compounds or the so-called green or semi-green compounds in many applications due to their excellent mechanical properties, which give them strength and high hardness, as well as their low density, which made these compounds light very desirable, especially in the field of aviation and transportation, thanks to the reduction of both weight and fuel consumption associated with it, which made the bets more on these materials [1], This prompted researchers to develop permanent green compounds that are environmentally friendly and at the same time biodegradable [2], as we will discuss in this article refers to one of the methods of characterization that enables us to ascertain the efficacy of these applications and prove their reliability and use in the future with further progress in relation to resins and this vegetables fibers. Thus, we should ensure that we obtain stronger green compounds that are more stringent and better than their predecessors [2]. In recent years, we have noticed the use of several techniques for separate determination of adhesion and friction in micromechanical tests with their development each time, but their experimental achievement is still somewhat complicated because they require accurate values for the external structures at the moment of separation beginning called Crack initiation. Rokbi et al. have studied Unsatured Polyester (UP) [3], and Chang-Uk Kim et al. have studied vinyl ester (VE) [4]. This study aims to estimate the adhesion strength between the reinforced fibers utilizing two types of this latter untreated and treated by5 %NaOH aqueous solution with the polymer matrix also in both types, polyester and vinyl ester by the microbond tests, that depend on measuring the maximum strength resulting from the take out of the fiber from the matrix droplet. This maximum strength value is used in calculating several parameters such as the interfacial shear strength like the apparent bond strength τ_{app} and the local surface shear strength τ_d [5].

2. Experimental

2.1. Materials

The fibers used in this study were extracted from Alfa plant (M'sila region, Algeria), in February 2020. First all, the stems of Alfa (Stipa tenacissima) were completely immersed into the container filled with tap water and covered for four weeks to extract the

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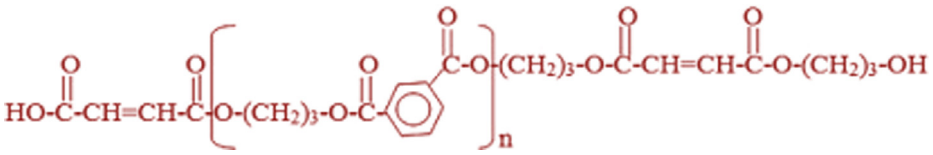
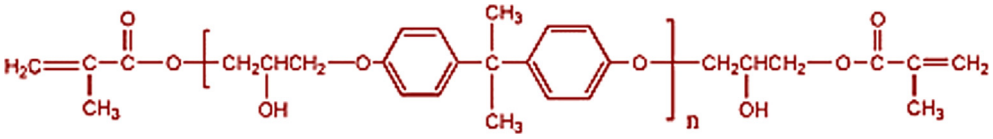
fibers using biological retting technique. Next, the fibers were removed from the stems with the manual method utilizing a metal brush. The extracted fibers were washed with distilled water to eliminate unwanted contaminants left behind the fiber surface, then dried in an oven at 80 °C for 6 h [6]. Like is shown in Fig. 1.

Two types of resins were used in this work, namely Unsatured Polyester (UP) and vinyl ester (VE)and their chemical compositions is shown below.

UP resin [7]



VE resin [8]



The UP and the VE resins were provided by Cray and Valley Company [749]. The mechanical properties of both resins are shown in Table 1.

2.2. Fiber treatment

First all, Alfa fibers were soaked in a solution of 5 % NaOH at 28 °C. The fibers were immersed in the alkaline solution for 4 h, then the treated alpha fibers were washed several times with distilled water. After that, sulphuric acid was used at a rate of 2 % for 10 min in order to neutralize the traces of sodium hydroxide remaining on the surface of the fibers. Then the fibers were washed again with distilled water until a pH = 7. Finally the fibers were dried at 60 °C for 6 h as stated by Rokbi et al. [2].

2.3. Microbond specimens' preparation

Untreated and treated Alfa fibers were glued into frames made of wood. We took a thin wooden needle to put small drops of resin of both types of resin UP and VE on the fibers then let them dry for 24 h [10]. After that, we selected the fibers with the ideal drops, and then we glued paper clips at the tip of each fiber to finally get the microbond specimens ready for testing. All this is shown in Fig. 2.

2.4. Morphological investigation

Parameter fibers matrices include the diameters of the Alfa fibers and the lengths of the resin drops were measured using an OPTIKAPRO 3 Digital Camera light microscope equipped with a digital camera photomicrography system [11].

2.5. Specimens 'measurement

As indicated above, UP and VE resins were used to predict the interfacial characteristics between both resins and the untreated and treated Alfa fibers. For each of the samples, we measured the diameter of the fiber ten times in different areas of the areas applied by the polymer, as well as the length of the droplet ten times also to get the average diameter of the fiber and the average length of the droplet each time for each of the samples, all this is shown in Table 2.

This is for the purpose of an accurate results [12], and the view with measurement like showed in the Fig. 3.

2.6. Microband tests

Microbond tests were conducted using Instron universal testing machine (UTM) at a speed of 2 mm/min according to ASTM D 30393tensile tester. The device contains two steel blades that can be positioned with micrometers like showed the Fig. 4.

The role of the steel blades is to support the droplets and hold them during the debonding of fiber [13]. If the shear stress is constant along the interface, the average values of IFSS were calculated by using the equation below.

$$\tau_{app} = F_{max}/(\pi.D.L)$$

where: (D) represent the diameter of fiber, (L) the length of the droplet and (F_{max})is the maximum force was measured during the pull out of fiber. The microbond tests were carried out in order to estimate the values of the bond strength between the untreated and treated Alfa fibers and both used resins. A total of 120 samples were tested. And the positive results were taken.

3. Results and discussion

Fig. 5 shows the force–displacement curves obtained from microbond tests for the tested specimens, it represents the force needed to pull out the fiber from the droplet in terms of displacement. For untreated Alfa/UP the value of force is 0.116 N, untreated Alfa/VE is 0.098 N, treated Alfa/UP is 0.235 N and treated Alfa/VE is 0.220 N ,where we notice that the debonding occurs at the threshold of the curve at the greatest value of the force to pull the fiber from the matrix who is called crack initiation, then the value of the force decreases to almost non-existent and remains like this until the total exit of the fiber from the matrix due to the friction. From these results, we note that the force required to pull out the treated fibers from both matrix is greater than compared to the untreated one**.** The greater pulling out force of the alkaline-treated alpha fibers compared to the untreated one is due to the removal of impurities from the surface of the alpha fibers and the improvement of mechanical properties.

There is also a point worth mentioning, which is that we have observed many times a relatively large difference in the force recorded for approximately equal dimensional samples, and vice versa. We note the same force recorded for samples of varying

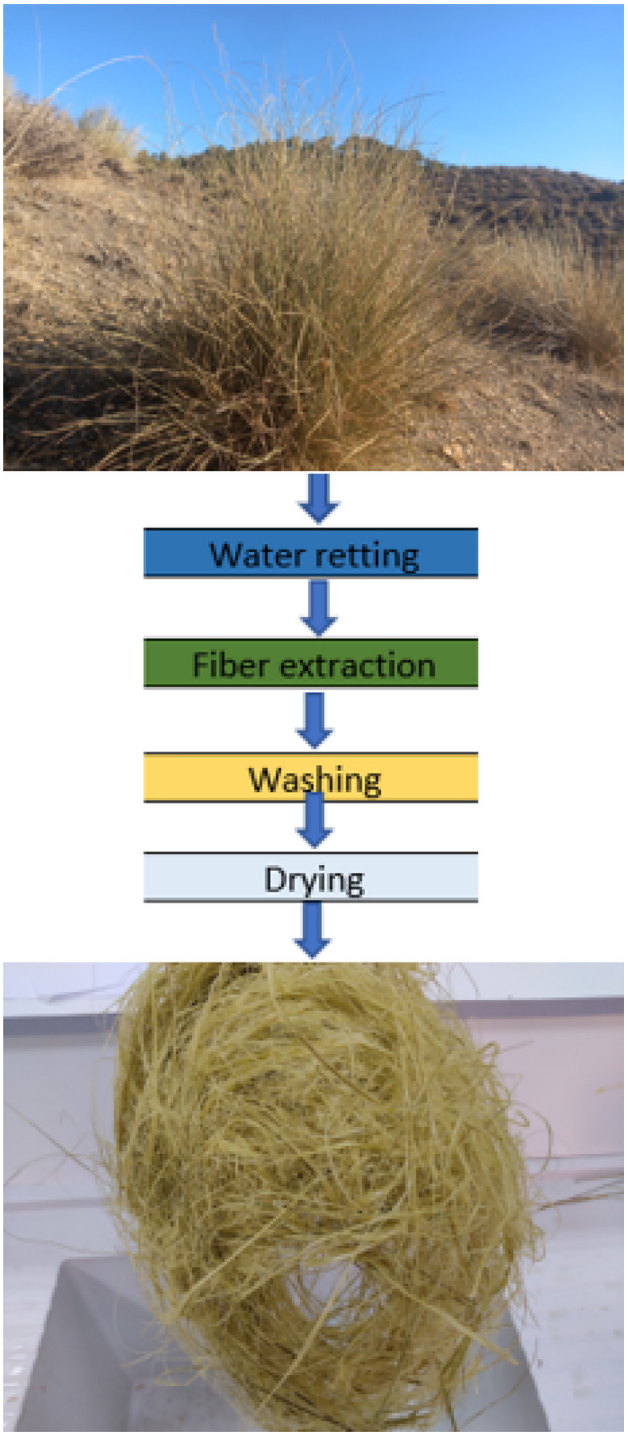


Fig. 1. Plant and Extracted fiber.

dimensions with a significant percentage, this is due to the difference in the components and structure of plant fibers, which in turn leads to the difference in mechanical properties. A large scattering in results can be seen. This dispersion can be explained by many different factors that influence the quality of fibers [1214], such as:

- Variable growth conditions (weather, soil quality, maturity, climatic conditions……),
- Extraction methods,
- Test parameters/conditions,

The increased force of alkali treated Alfa fibers due to the elimination of impurities from the Alfa fiber surface. Previous research shows the values of tensile strength from plants fiber was approximate with

After obtaining these results, we were able to calculate the apparent IFSS values for the different types of samples that we touched upon in this study, as shown in the Fig. 6, which is a bar graph showing the apparent average IFSS corresponding to each of the aforementioned types of samples saw from this result, the higher IFSS was treated Alfa/UP with 5.36 ± 0.18 MPa, then treated Alfa/VE with 5.18 ± 0.87 MPa, followed by untreated Alfa/UP, and untreated Alfa/VE with 2.47 ± 0.64 MPa and 2.25 ± 0.41 MPa, respectively. The increased value of the IFSS of treated Alfa/UP was 138.22 %, treated Alfa/VE was 130.77% and untreated Alfa/UP by 9.77 %compared to the untreated Alfa/VE. The data obtained is comparable to the IFSS of Inula viscosa, flax, hemp, and sisal by Moussaoui et al. [10]. The bonding between the both types of resin and the Alfa fiber was improved by NaOH treatment.

According to the results, the adhesion between the alkali treated Alfa fiber/resins was better than the adhesion between the untreated one/resin. In others words, the alkali treated Alfa fiber require greater strength to pull out compared to untreated one. These results are in concordance with the work of Moussaoui et al .[10], Auar et al. [13]and Sanjay et al. [14]. This is due to the chemical treatment of Alfa fibers, which made the fibers have stronger and better mechanical properties, as well as the effect of this treatment on the surface of the fibers, which made it rougher compared to raw fibers. Rokbi et al. [2], concluded that chemical treatment played a major role in complete removal of waxes, oil, residual lignin, and impurities, lignin, and hemicelluloses, and this is confirmed by this the values of the interconnections obtained in this study.

Table 2
Dimension of micro bond tests specimens.

Types of specimens	Number of specimens	Average fiber diameter (μm)	Average length of resin droplet (μm)
Untreated fiber/UP	30	474	3348
Untreated fiber/VE	30	437	3087
Treated fiber/UP	30	476	3244
Treatedfiber/VE	30	422	3117

4. Conclusion

In this article, the bond strength between the Alfa fibers and the epoxy resin, we investigated it using the microbond test technique. We were able to estimate the bond strength between the fibers and

Table 1
Mechanical properties of UP and VE resins.

Resin type	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at break ξ (%)	Flexural strength (MPa)	References
UP resin	3.4–3.8	60–80	2.5–3.5	80–100	[7]
VE resin	3.66	78	7.4	76–88	[4,9]

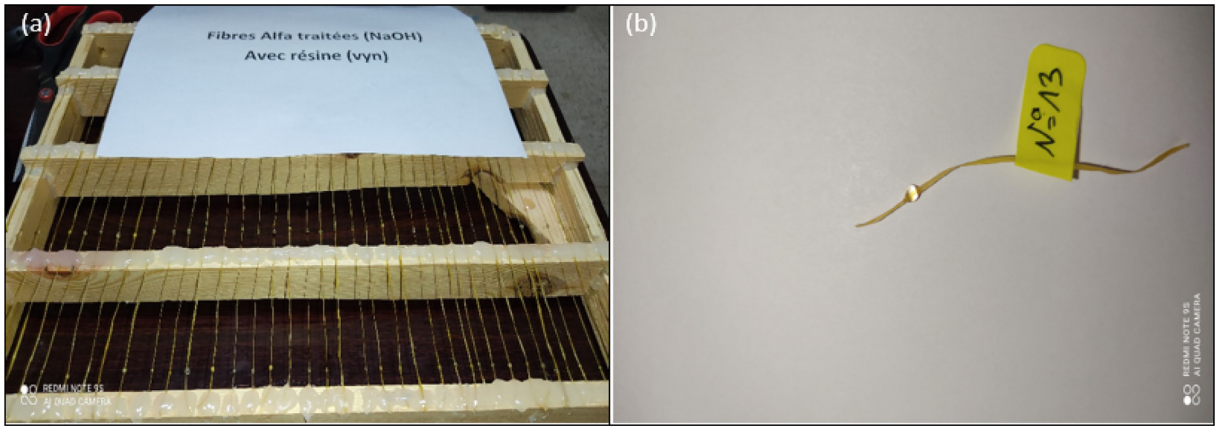


Fig. 2. (a) Application of drop on the fibers; (b) Microbond specimens.

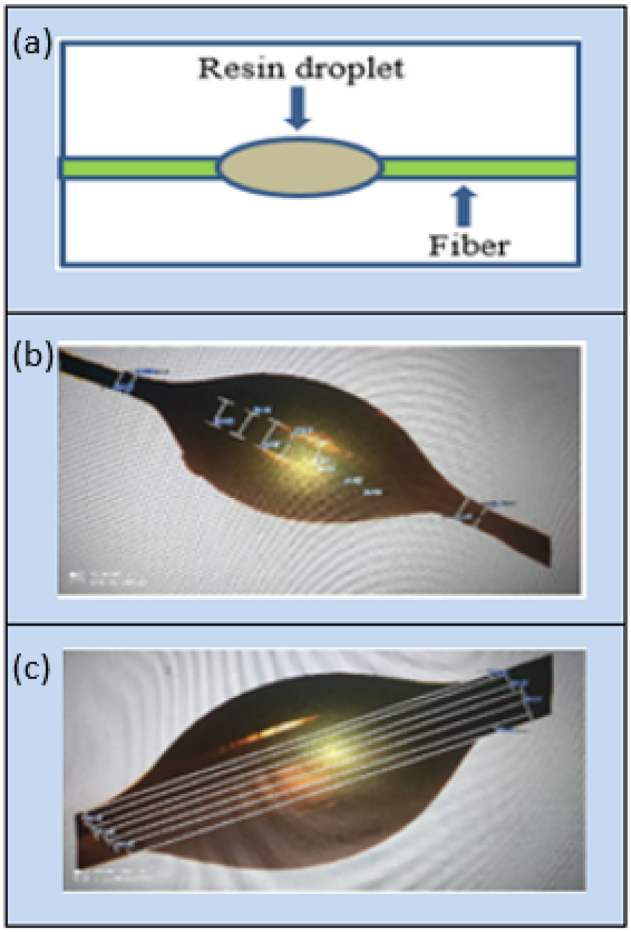


Fig. 3. a) Schematic view of fiber and resin droplet, b) Measurement of fiber diameter and c) Measurement of droplet length.

the epoxy resin using the results obtained after calculating the Interfacial Shear Strength, depending on the average of the maximum strength values for each type of compound that was subjected to the microbond test. The largest value of the bonding strength is for NaOH treated Alfa fiber/polyester resin, after that the value followed by the NaOH treated Alfa fiber/vinyl ester resin, and then the value of the raw Alfa fiber/polyester, and finally the value of the raw Alfa fiber/vinyl ester.

It is also worth noting that chemical treatment played a major role in increasing the mechanical properties and performance of the fiber/resin composite, and therefore these properties confirmed the existence of a great potential for using these fibers in high performance polymer compounds in the future.

CRediT authorship contribution statement

Sid Ali ZERNADJI: Resources, Methodology, Investigation, Writing - Data curation, review & editing. **Mansour ROKBI:** Resources, Methodology, Writing-original draft, Supervision, Investigation & supervision ,Writing - review & editing. **Mohamed BENHAMIDA:** Methodology, Formal analysis, supervision and Investigation, Writing - review & editing. **Dalila HAMMICHE:** Formal analysis and Investigation, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

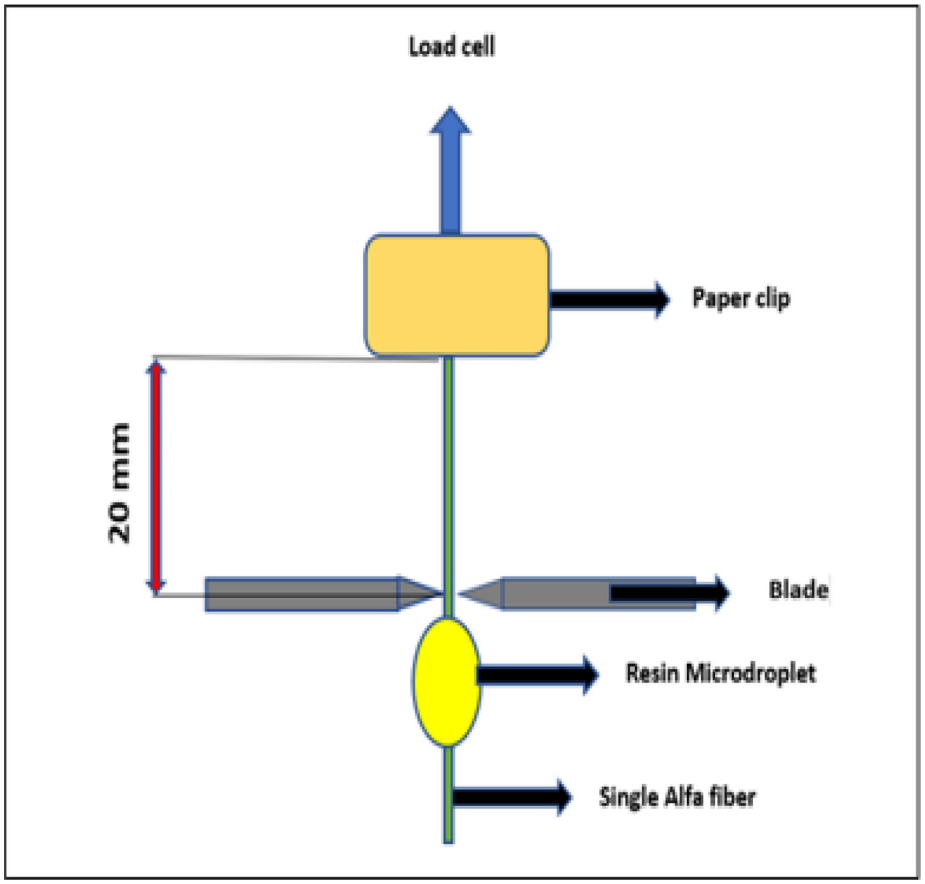


Fig. 4. Schematic view of Microbond test.

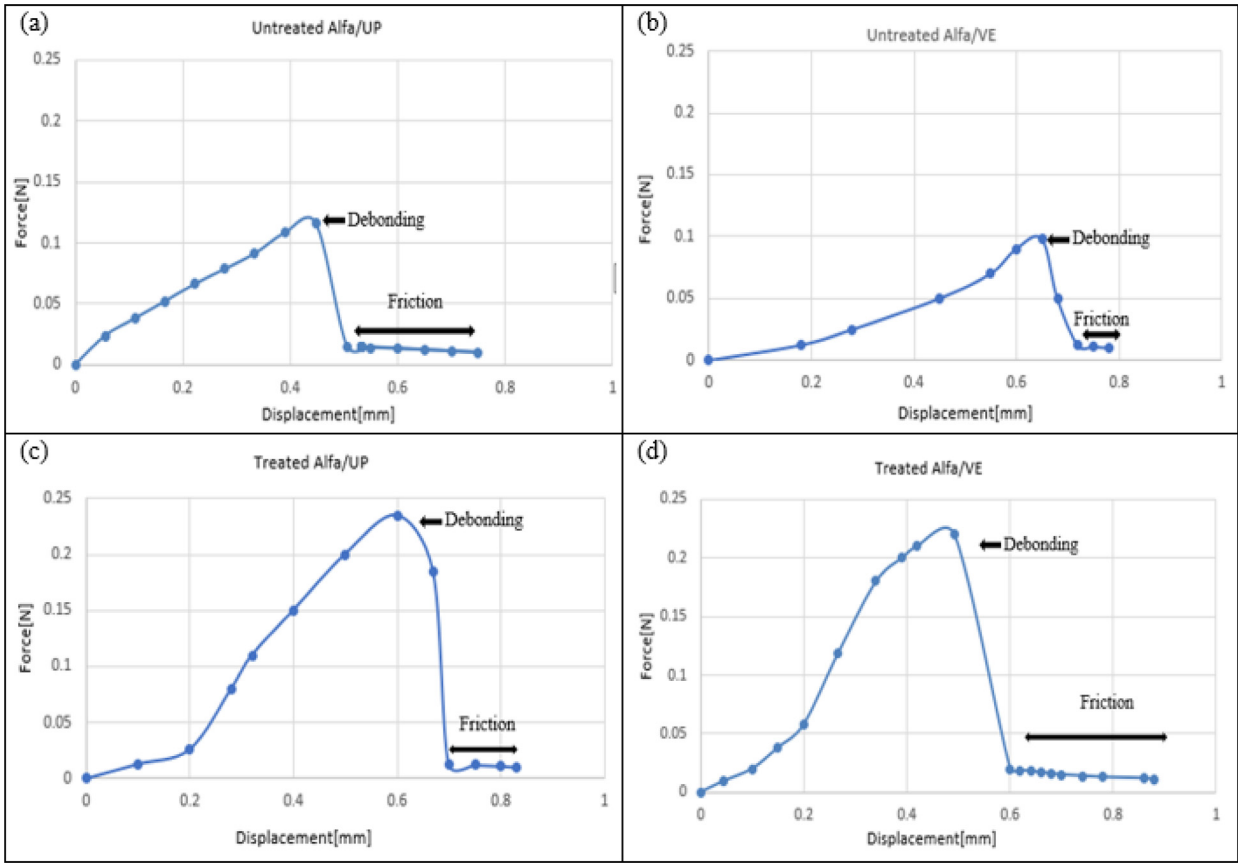


Fig. 5. Force–displacement curves obtained from microbond tests for a) Untreated Alfa/UP; b) Untreated Alfa/VE; c) Treated Alfa/UP and d) Treated Alfa/VE.

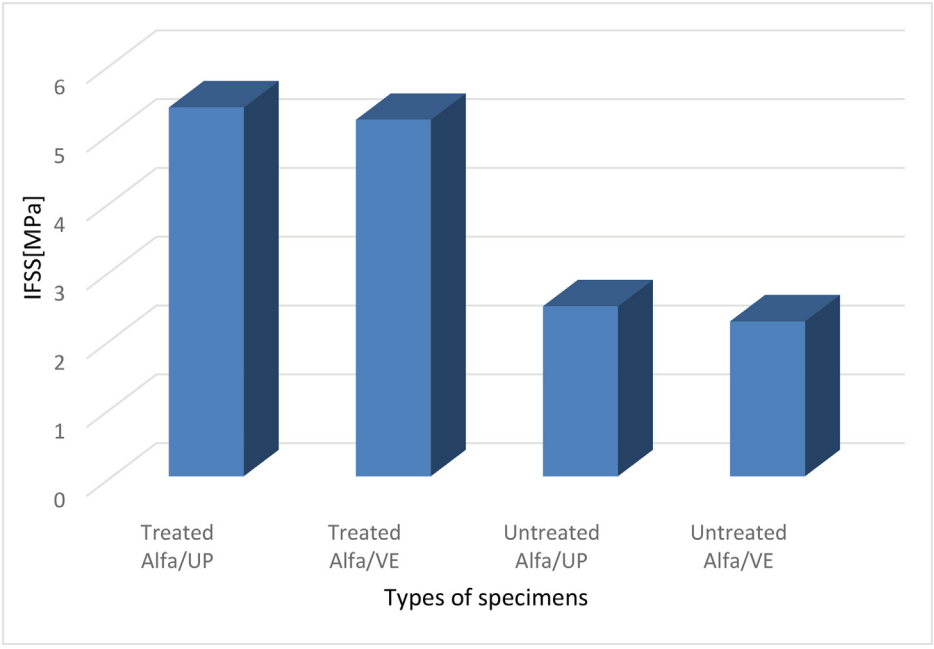


Fig 6. The Interfacial Shear Strength properties.

References

[1] P. Taylor, A.N. Netravali, X. Huang, K. Mizuta, Adv. Compo. Mater. 16 (2007) 269–282.
[2] M. Rokbi, H. Osmani, A. Imad, N. Benseddiq, Procedia Eng. 10 (2011) 2092–2097.
[3] P. Manimaran, M.R. Sanjay, P. Senthamaraikannan, B. Yogesha, C. Barile, S. Siengchin, J. Nat. Fibers. 17 (2020) 359–370.
[4] C.U.S. Kim, Fibers Polym. 21 (2020) 428–436.
[5] S.F. Zhandarov, E. Mäder, O.R. Yurkevich, J. Adhes. Sci. Technol. 16 (2002) 1171–1200.
[6] Z. Belouadah, A. Ati, M. Rokbi, Carbohydr. Polym. 134 (2015) 429–437.
[7] M.D.L. Chikouche, A. Merrouche, A. Azizi, A. Rokbi, M. Walter, J. Reinf. Plast. Compos. 34 (2015) 1329–1339.
[8] J. Jia, Z. Huang, Y. Qin, High Perform. Polym. 25 (2013) 652–657.
[9] P. Siva, I.K. Varma, D.M. Patel, T.J.M. Sinha, Bull. Mater. Sci. 17 (1994) 1095–1101.
[10] N. Moussaoui, M. Rokbi, H. Osmani, M. Jawaid, A. Atiqah, M. Asim, L. Benhamadouche, J. Polym. Environ. 29 (2021) 3779–3793.
[11] Bouguesir, Harkati, H. Rokbi, M. Priniotakis, G. Vassilliadis, S. Vasilakos, S. Boughanem, H. Fellah, L. J. Comput. Methods Sci. Eng. 18 (2018) 129–147.
[12] M. Rokbi, A. Ati, F.Z. Aiche, Res. J. Text. App. 22 (2018) 195–211.
[13] H. Anuar, A. Zuraida, B. Morlin, J.G.J. Kovács, Nat. Fibers. 8 (2011) 14–26.
[14] P. Manimaran, S.P. Saravanan, M.R. Sanjay, S. Siengchin, M. Jawaid, A. Khan, Integr. Med. Res. 8 (2019) 1952–1963.