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Original Article

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Mechanical and morphological characterization of recycled HD-PE bio-composites based on alfa fibers and natural pozzolan

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Abstract: In this research work, waste plastic bottle caps made of high-density polyethylene (HD-PE) were reincorporated as a matrix and reinforced by alfa short fibers and natural pozzolan particles. Using different weight percentages of both fillers of 5 wt% up to 30 wt%, three types of bio-composite materials have been produced; alfa short fibers/HDPE, pozzolan particles/HDPE, and alfa fibers pozzolan/HDPE. Specimens for each type of the biocomposites were prepared through the compression molding method. The objective of this study is to investigate the effect of different content of alfa short fibers and pozzolan particles on the mechanical and morphological properties of the recycled HDPE matrix. Tensile test results revealed an enhancement in the mechanical properties for the three types of the bio-composites, an increase in tensile strength reached the maximum of 3573 MPa plus an interesting improvement in Young's modulus with a maximum value of 3696 MPa. The toughness of the neat recycled HD-PE decreased by 212% by adding the natural filler whereas the modulus of resilience exhibited an increase of 138% compared to the neat recycled HD-PE. Therefore, the good rheological behavior of these bio-composites makes it possible to produce competitive materials and allows the reduction of plastic waste in the environment.

Keywords: alfa short fibers; bio-composite; mechanical properties; natural pozzolan; recycled high-density polyethylene; rheological behavior.

1 Introduction

In recent years, composite materials have received great interest and importance due to the way they were tailored to act synergically and have a long life span. This makes composite materials interesting for different varieties of applications in many industrial sectors such as automobiles, aerospace, electronics, packaging, construction, and sports goods, among others [1, 2].

Natural fiber-reinforced polymer composites (NFRPCs) are partially biodegradable composites; when natural fibers are used as reinforcement in petroleum-based polymers matrix, but the resulting composites can be green composites when using biodegradable polymers. NFRPCs are eco-friendly materials because they can be easily disposed of or composted. The current environmental regulation concerning plastic materials wastes has incited different industries to rely on these biomaterials. Moreover, NFRPCs are an excellent opportunity to reduce the dependency on petroleum-derived products [3, 4].

Natural fiber could be used as substitutes for most frequently used synthetic fibers such as glass and carbon fibers. There has been growing interest in using natural fibers as reinforcement to produce composite materials with multiple advantages like; low density and cost, high specific properties, availability, plus biodegradability, etc. Several studies have investigated the use of textile plants (ramie, jute, kenaf, cotton, sisal, flax, coir, wood, hemp,

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etc.) as fillers to decrease the environmental pollution. These natural fibers are considered composite materials, composed mainly of cellulosic micro-fibrils that reinforce the lignin and hemicellulose matrix. The cellulosic micro-fibrils give these plants their stiffness and strength. Generally, with increasing cellulose content, the tensile strength along with Young's modulus of fibers is increased since it requires high energy to uncoil the oriented fibrils [5, 6].

Among other vegetable fibers, alfa fibers are originally found in the arid and semi-arid regions of North Africa and Spain (alfa is the Arabic name of esparto). The alfa plant belongs to the Gramancie family which is a xerophilous herb that exists in the form of bundles constituting stems that grow to a height of about one meter; this plant does not need insecticides nor pesticides or much water to grow as it plays an important role in withstanding desertification. Hence, these species are neglected due to the misunderstanding of their economic and technical importance. This grass is mostly used in the production of handcrafted goods and paper of superior quality [6, 7].

Karama Elfehri Borchani et al. [8] mentioned after electronic scanning microscopic views of longitudinal and cross-sections of *Stipatenacissima* fiber that alfa stem surfaces were found to be smooth, the long fiber is composed of bundles (nanotubes) bounded together with lignin and the diameter is about 200 μm in section. The SEM also shows that a singular fiber constituting the bundle consists of microfibrils.

Recently, an increasing interest has been focused on the usage of alfa fibers as reinforcements in bio-composite materials. It is commonly used with polyolefin polymers especially thermoplastics due to their low price and their recyclability without any risks of serious damage, unlike thermoset polymers. Moreover, the thermoplastics processing temperature is less than 220 °C which allows to avoid thermal degradation of natural fibers [9]. A study conducted by Sahar Salem et al. [10] have shown that alfa fibers reinforced HD polyethylene composite possess higher performance than wheat straw, cornstalk, and corncob reinforced HDPE composites concerning the tensile strength, its strength increases with increasing treated alfa fibers content.

The mechanical properties of NFRPCs are mainly determined by the dispersion, distribution of the fillers in the matrix, and the processing techniques employed. Interest in using fiber-reinforced thermoplastic composite materials as structural and semi-structural components for the automotive industry keeps growing. These innovative materials can be elaborated mainly for car interior parts like a dashboard, door panels, seatbacks, and other components [11, 12].

The end-of-life management of natural fiber-reinforced thermoplastic composites (NFRTCs) is another advantage over common thermosetting composites. The recycling of these Innovative materials is driven by social awareness concerning the harmful impact of polymeric materials waste on the environment. Indeed, the recycling of these Innovative materials is driven by social awareness concerning the harmful impact of polymeric materials waste on the environment. Indeed, the ability of NFRTCs materials to be recycled either during the manufacturing process (internal recycling) or at the end of their life cycle (external recycling) is an advantageous option for the growth and development of the bio-composite materials industry [13, 14].

Researchers investigated the effect of recycling on alfa fiber reinforced thermoplastics composites. Dalila Hammiche et al. [15] have studied the reprocessing cycle effect on alfa fiber-reinforced polyvinylchloride (PVC) composites with maleic anhydride-grafted polyvinyl chloride used as a compatibilizer, which resulted in an enhancement of tensile strength and Young's modulus after four cycles with an increase in glass transition temperature of the PVC/alfa composites compared to the original composites. Moreover, to evaluate the effect of recycling on alfa fibers reinforced polypropylene composites, and the impact of alkali treatment on alfa fibers. Fatima Ezzahra El Abbassi et al. [16] have subjected these NFRTCs to static and dynamic mechanical analyses, The results obtained showed a decrease in tensile and flexural strengths of the recycled composites, this is due to the inherent lower thermal stability of lignocellulosic fibers as well as a drop in the storage modulus as shown for the case of hemp fibers-based composites.

Similarly, as mineral filler, the pozzolan particles can be obtained from a pyroclastic rock (natural pozzolan) or artificially manufactured. Many industries have used these inorganic fillers such as clay, black carbon, talc, and calcium carbonate for polymer-based composites due to their availability and low cost. They were found useful in improving certain mechanical properties and dimensional stability [17].

In this work, we recycled waste plastic bottle caps made of high-density polyethylene (HD-PE) and used it as a matrix for different types of composite materials which are the following; natural pozzolan powder/recycled HD-PE composite, short alfa fiber/recycled HD-PE composite, and hybrid composite combining short alfa fiber plus natural pozzolan powder. To our best knowledge, this is the first time in materials sciences that natural pozzolan is used as filler with alfa fibers in a recycled HD-PE matrix. The objective of this investigation is to develop biocomposite materials by reprocessing plastic waste with natural abundant reinforcements of different kinds and sizes (natural pozzolan powder and short alfa fibers) while making a comparison of the three types of bio-composites stated previously. We also study the effects of adding different contents of natural pozzolan powder and short alfa fibers on the recycled HD-PE matrix. Furthermore, we characterized samples of the produced biocomposites, by initially using the Fourier transform infrared red (FTIR) spectroscopy to evaluate the structural properties of the biocomposites components. Then, we obtained the mechanical properties of the samples by a tensile test. Finally, scanning electron microscopy (SEM) was applied to acquire some information on the morphology of the composite.

2 Materials and methods

2.1 Materials

The alfa fibers used in this study were extracted from the Stipatenacissima (Esparto grass plant) that have been harvested from the state of Ain Temouchent located in the North West region of Algeria. In the physical preparation, alfa (esparto grass) were cut into small pieces of approximately 1–2 cm in length and then dehydrated in an oven (MEMMERT) at 105° for 48 h. The little pieces were crushed with a blade crusher (MA garant) and recovered to get sifted in a vibratory sieve of 125 μm (RETSCH TYPE AS 200 amplitude 3 mm/g). Figure 1 shows the physical preparation of alfa fibers.

Figure 1: Physical preparation of the alfa fibres: (A) the alfa stems gathered; (B) the cut alfa stems; and (C) the alfa fibres collected from a 250 μm diameter sieve.

Natural pozzolan powder has been used in this investigation; this natural mineral filler is locally available. Algeria has a significant amount of pozzolanic materials of volcanic origin existing along 160 km between the Algerian–Moroccan border and the Sahel of Oran. The natural pozzolan powder is extracted from a siliceous volcanic rock of variant colors ranging from red to black. The chemical composition is shown in Table 1 [18]. A Retsch Jaw Crusher BB (100 w/Mn Steel Jaws & SS Plates, 220 V 60 Hz) has been used to crush the pozzolan rocks as illustrated in Figure 2. The obtained powder was sifted with a 250 μm diameter sieve (RETSCH TYPE AS 200 amplitude 3 mm/g).

2.2 Preparation and elaboration of the composites

The work was done manually where waste plastic bottle caps were reincorporated as a thermoplastic matrix for the bio-composites; these bottle caps that are made of high-density polyethylene were grinded by an industrial knife crusher. The resulting shredded plastic bits were heated under 200 °C on a laboratory hot plate. When the heated plastic takes the form of a paste, the heating temperature is reduced to 180 °C. Then, the alfa short fibers along with the pozzolan powder were added to the matrix and blended manually. The composites were finally molded at 50 °C through the compression molding method. The experiment steps were repeated following the different fractions of each component for each composite as indicated in the following Tables 2 and 3.

The obtained molded specimens were machined using a numerical controlled (NC) miller (KOSY A3S controlled with MegaNC-2D-2005) as shown in Figure 3.

3 Characterization

3.1 Attenuated total reflection (ATR) – Fourier transform infra-red analyses

The structural properties of the used fillers (pozzolan and alfa) were evaluated using a Thermo Scientific Nicolet Summit FT IR spectrometer with the Everest ATR accessory configured for specular reflectance. The spectra were obtained with an accumulation of 16 scans with a resolution of 4 cm^{-1}.

3.2 Tensile test

The tensile test was conducted on eighteen specimens according to the ISO 6259-1 standard. The test was performed on a universal testing machine ZWICK Z100 at a

Table 1: Chemical composition of natural pozzolan.

Composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	Cl^-	LOI
wt%	61.57	18	4.93	6.69	2.63	0.1	1.95	1.65	0.04	2.15

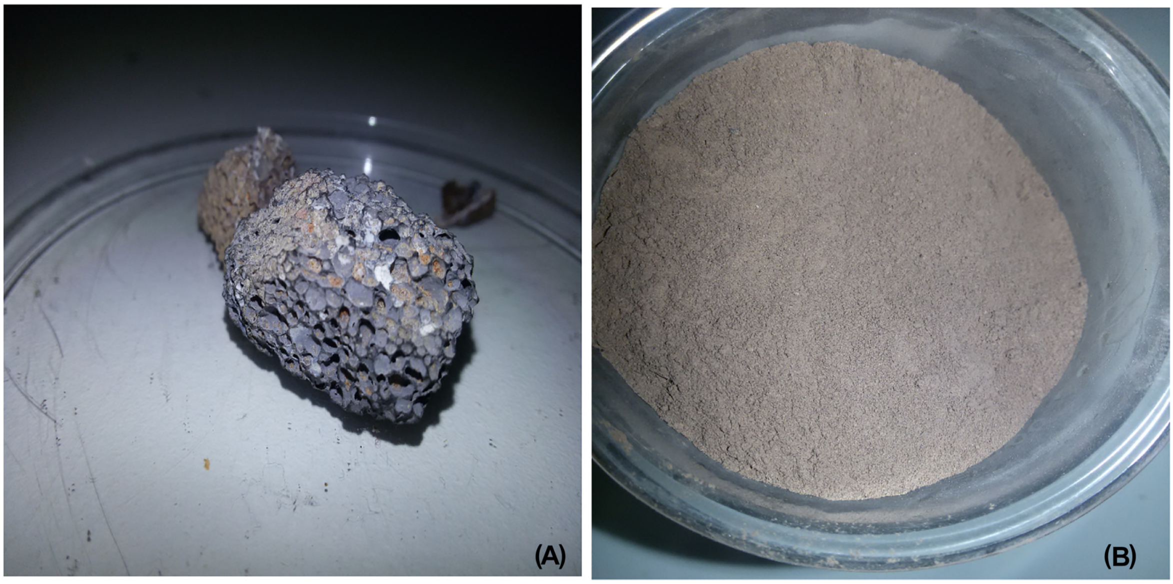

Figure 2: Physical preparation of pozzolan: (A) pozzolan rock and (B) pozzolan powder.

Table 2: List of the samples produced with their composition for PZ/HDPE composite and Alfa/HDPE composite.

	PZ/HDPE composite.		Alfa/HDPE composite.	
Sample code	HDPE content (wt%)	PZ content (wt%)	HDPE content (wt%)	Alfa fibres content (wt%)
95:5	95	5	95	5
90:10	90	10	90	10
85:15	85	15	85	15
80:20	80	20	80	20
75:25	75	25	75	25
70:30	70	30	70	30

HDPE, high density polyethylene; PZ, puzzolana.

Table 3: List of the samples produced with their composition for PZ + Alfa/HDPE hybrid composite.

Sample code	HDPE content (wt%)	PZ content (wt%)	Alfa fibers content (wt%)
0:0	100	0	0
5:25	70	5	25
10:20	70	10	20
15:15	70	15	15
20:10	70	20	10
25:5	70	25	5

HDPE, high density polyethylene; PZ, puzzolana.

transversal crosshead speed of 3 mm/min in an ambient temperature using a 10 KN load sensor as illustrated in Figure 3F, wherein stress-strain curves were generated. The test ended when the specimens fractured.

The toughness (U_T) is calculated from Eq. (1) as follows:

$$U_{\mathrm{T}} = \int_{0}^{\epsilon_{\mathrm{f}}} \sigma_{\mathrm{e}} \mathrm{d}\epsilon_{\mathrm{e}} \tag{1}$$

where σ_e is the engineering stress, ϵ_e the engineering strain, and ϵ_f the fracture strain of the specimen, respectively.

Moreover, the modulus of resilience (M_R) is calculated from Eq. (2):

$$M_{\mathrm{R}} = \int_{0}^{\epsilon_{\mathrm{y}}} \sigma_{\mathrm{e}} \mathrm{d}\epsilon_{\mathrm{e}} \tag{2}$$

In which σ_e is the engineering stress, ϵ_e the engineering strain, and ϵ_y the yield strain at which linear elastic behavior ceases.

3.3 Scanning electron microscopy (SEM)

The morphology of the bio-composite was investigated using an electronic microscope Hitachi TM1000 electron.

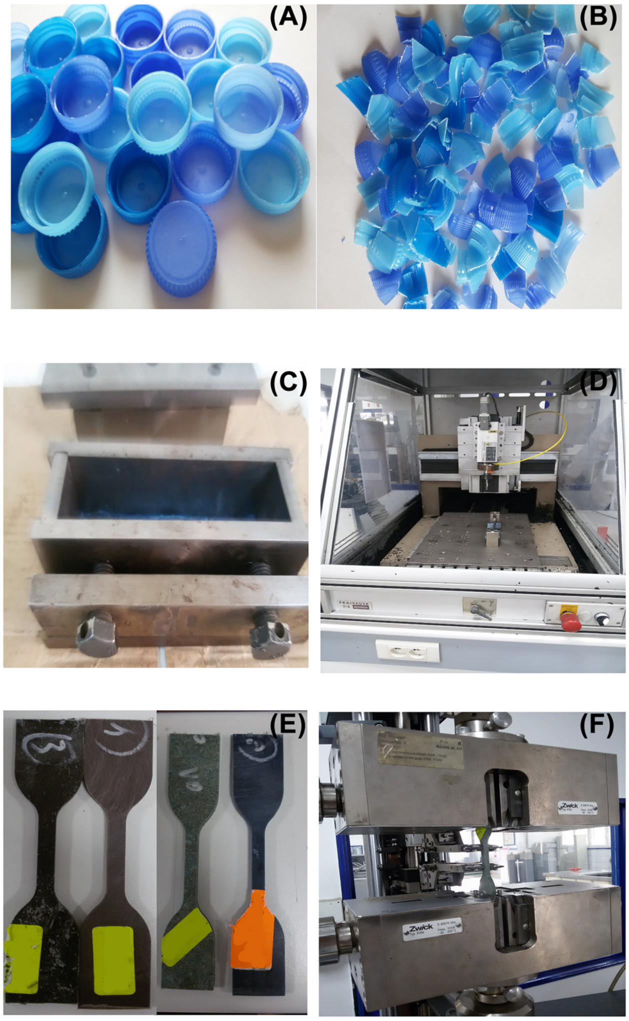

Figure 3: The processing steps of the bio-composites: (A) bottle caps waste; (B) grinded bottle caps; (C) the molding step; (D) the machining of the platelets; (E) samples of the produced bio-composites; and (F) the tensile testing of samples.

The tested specimens have been observed to analyze the fractured surfaces and the state of the components interface.

4 Results and discussion

4.1 ATR-FTIR analyses

The attenuated total reflectance (TAR) has been used to investigate the structure of the alfa fibers and the pozzolan powder in order to determine their composition.

FTIR spectroscopy allows measuring the variations of the alfa compositions. The alfa fibers contain respectively 46% in weight of celluloses, 19.9% of lignin, and 30.2% of

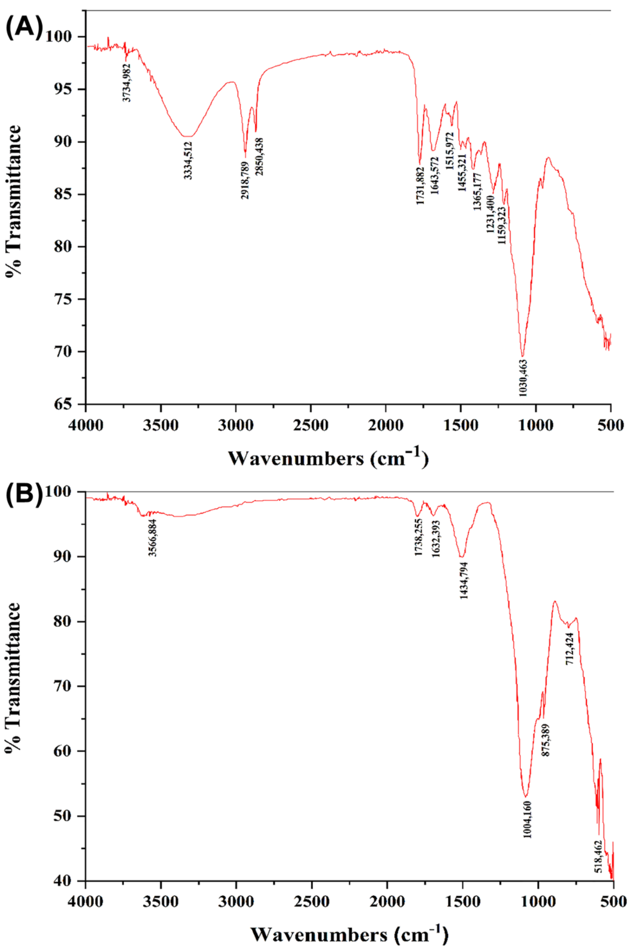

Figure 4: FTIR measurements of (A) raw alfa fibers and (B) natural pozzolan.

hemicelluloses. The infrared spectra of alfa fibers are illustrated in Figure 4A. The intensive bands observed in the FTIR spectra in the alfa fibers are hydroxyl groups of H-O celluloses with wider bands of approximately 3334 cm^{-1} [19]. The vibration of C–H of CH and CH_2 appears at 2918 cm^{-1} and 2850 cm^{-1} which is due to the celluloses-hemicelluloses [20].

The instance band at 1731 cm^{-1} corresponds to the stretch of the C=H of the carboxylic acids and ester groups [8]. A little peak is observed at 1620 cm^{-1} associated with the presence of water [21]. The other picks at 1515 cm^{-1} and 1455 cm^{-1} are attributed to the C=C vibration (aromatic skeleton) and to the deformation of C–H, respectively, which are present in the lignin [8]. The peak of about 1164 cm^{-1} is due to C–O–C asymmetric vibration of β – glycosidic linkage [22]. The wider band with a shoulder at 1030 cm^{-1} corresponds to stretched ring C–O in celluloses and hemicelluloses [8].

Figure 4B shows the FTIR of the natural pozzolan used. A wide intensive band is observed at 3566 cm^{-1} and 1632 cm^{-1} due to the O–H bending of the absorbed water molecule H2O at the surface [23, 24]. The wide band at approximately 1434 cm^{-1} is associated with the stretching vibrations of O–C–O which is the result of atmospheric carbonation [23, 24]. Another wider band at 1004 cm^{-1} is related to the asymmetric stretching of the bridging oxygen atom in the T–O bond of the (Si–O–Si, Si–O–Al) of the aluminosilicate structure [23]. An absorption band located at 712 cm^{-1} corresponds to the asymmetric stretching of the AL–O bond in $(A^{IV})–O–A^{IV}$ [25].

4.2 Mechanical properties of composites

The mechanical properties of the three types of NFRTCs were determined using a tensile test and compared versus different content of reinforcement. The mechanical characteristics gathered from the tensile stress–strain curves are tensile strength, tensile strain, Young's modulus, toughness, and modulus of resilience.

Figure 5 illustrates the stress–strain curves of the three types of NFRTCs alfa/recycled HD-PE, PZ/recycled HD-PE, and alfa + PZ/recycled HD-PE.

In Figure 5A and B the addition of both reinforcement (PZ or alfa) to the neat recycled HD-PE shows significant improvement in tensile strength of 3573 MPa and 30,099 MPa for alfa/recycled HD-PE and PZ/recycled HD-PE composites respectively, a gain of about 54% and 16% compared to the neat recycled HD-PE.

It can be observed from Figure 5C that the hybrid composite alfa + PZ/recycled HD-PE tensile strength indicates a considerable increase of 30.65 MPa for the sample (10:20); this value represents a 19% increase in contrast to the neat recycled HD-PE.

It is interesting to see in Figure 6A the high values of Young's modulus for the three types of composites compared to the neat recycled HD-PE. This enhancement in Young's modulus is the consequence of the strong and stiff natural reinforcement that was added to the matrix. The maximum results that were attended by the three types of composites are as follow; the PZ/recycled HD-PE composite has reached a value of 2193 MPa which is a difference of 39% compared to the neat recycled HD-PE. Alfa/recycled HD-PE composite represents a maximum value of 3696 MPa a change of about 134% in contrast to the neat recycled HD-PE of Young's modulus for the alfa/recycled HPDE composite.

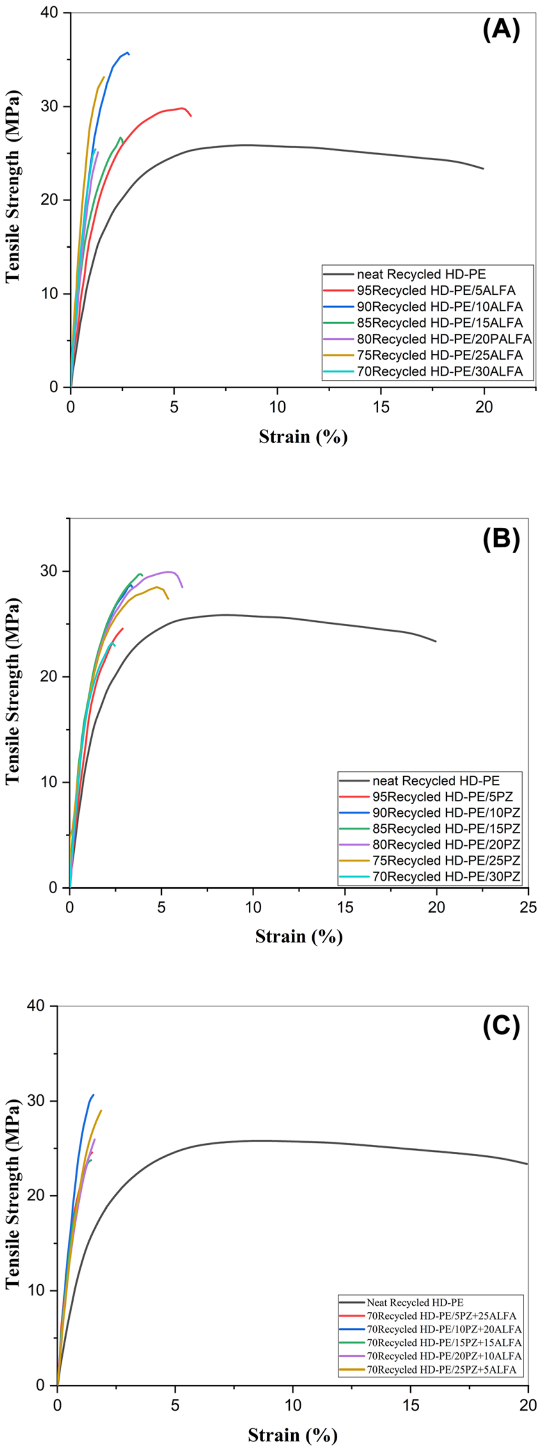

Figure 5: Stress-strain curves of (A) alfa/recycled HDPE composites, (B) PZ/recycled HDPE composites,and (C) hybrid composites Alfa + PZ/recycled HDPE.

Otherwise, the hybrid composites reached a value of 3449.3 MPa of Young's modulus and it is 118% higher than that of the neat recycled HD-PE.

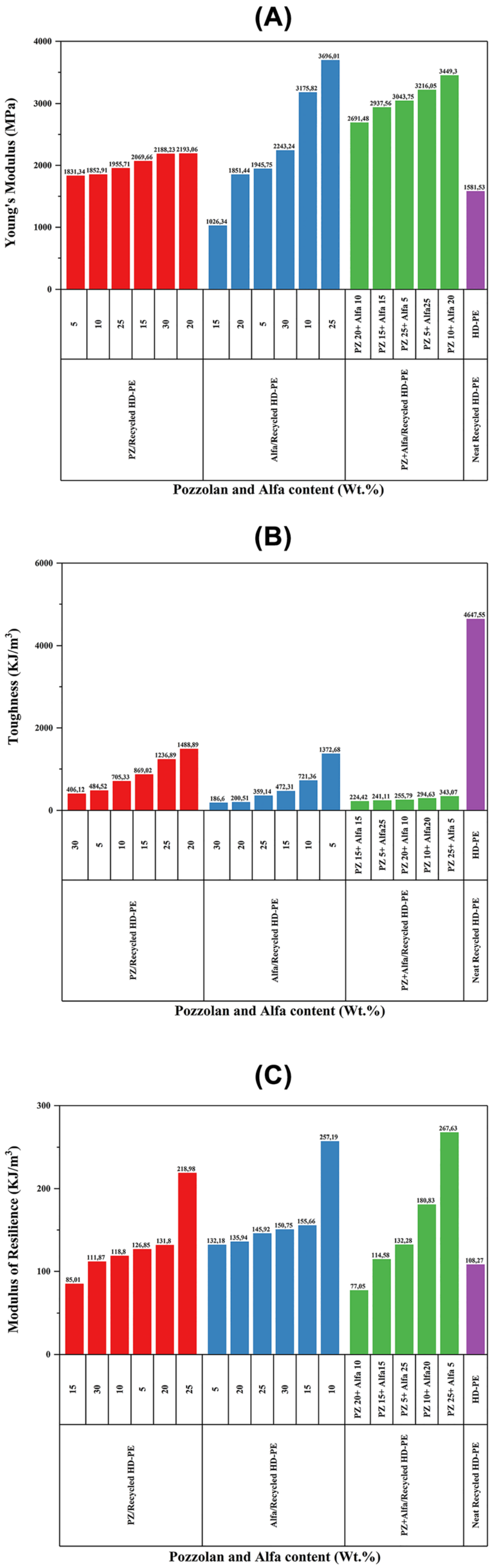

Figure 6: Effects of filler type and content on (A) composites' Young's modulus, (B) composites' toughnes, and (C) composites' modulus of resilience.

The improvement of Young's modulus of the three types of composites is associated with the stiffness of the reinforcements (PZ powder and alfa fibers). It is also due to the good adherence to the interface.

The toughness (U_T) of a material is its ability to absorb energy in the plastic range above the yield stress without fracturing, and it is measured by integrating the area under the stress–strain curve. Figure 6B represents the relation between U_T and (short alfa fiber and natural pozzolan powder) concentration. The three types of biocomposites showed a significant decrease in U_T when compared to neat HD-PE. This decrease in U_T may be attributed to the fact that short alfa fibers and natural pozzolan powder acted as stress concentrators. On the other hand, it can be seen that PZ/HD-PE composites are tougher in contrast with alfa/HD-PE and PZ + alfa/HD-PE composites. This indicates that PZ/HD-PE composites are more ductile and have a greater total elongation.

The area up to the yield point in the stress–strain curve is called the modulus of resilience (M_R) and it is defined as the material's ability to absorb energy when deformed elastically. Figure 6C exhibit the modulus of resilience as a function of pozzolan particles and alfa short fibers at different concentration. The three types of biocomposites reveal an increase in M_R by contrast to neat HD-PE. This increase in resilience is attributed to the increase in yield strength of these biocomposites. Higher M_R values are shown for alfa/HD-PE composites.

As stated by many researchers [26–29] with this natural and abundant plant alfa fiber and natural pozzolan, it is possible to produce new ecological materials useful for multiple structural applications. These observations are in good accordance with the morphologic results reported in the following section.

4.3 Morphological structure

A SEM analysis was carried out on the specimens of the three composites respectively alfa/HDPE, PZ/HDPE, and alfa + PZ/HDPE to investigate the dispersion and distribution of the fillers in the matrix and the mechanical behavior of the bio-composites.

Figure 7 shows the micrographics of the fractured surfaces after the tensile test. Few voids have been observed due to the tearing provoked by the tensile load [30].

Otherwise, after the fracture of the specimen, most of the alfa fiber residuals were adhered to the matrix revealing the strong interfacial adhesion. This explains the improvement of the resistance to the traction as it was reported in the mechanical analyses [31]. The SEM images also show the good dispersion of pozzolan particles and alfa fibers in the polymeric matrix.

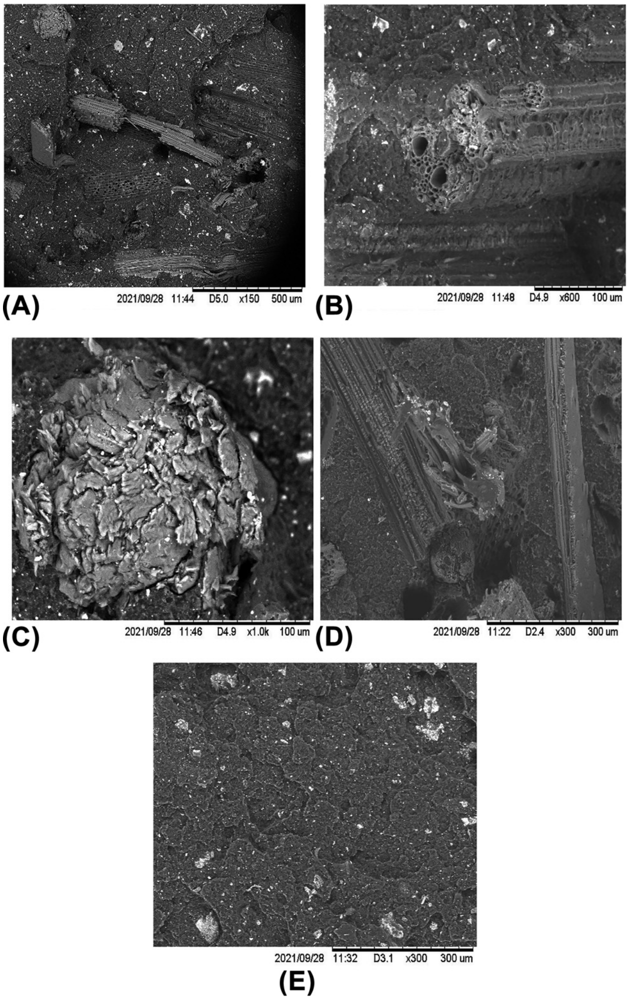

Figure 7: SEM micrographs of fractured surfaces of samples: (A–C) alfa + PZ/HDPE, (D) alfa/HDPE, and (E) PZ/HDPE.

In Figure 7B, it can be observed that the penetration of the matrix through the fibrils presents a good interaction between the fibers and the recycled HDPE matrix as assured and illustrated in Figure 7C showing the gluing between the polymeric matrix on the surface of the fibers [21].

5 Conclusions

The main aim of this experimental study is the reduction of plastic wastes and taking advantage of the existing abundant natural resources with excellent mechanical properties, to develop bio-composite materials with excellent and exceptional properties. Three types of new bio-composites have been successfully elaborated. Waste plastic bottle caps made of high-density polyethylene (HD-PE) have been reused as a matrix for these bio-composites. The alfa short fibers and natural pozzolan powder have been incorporated as reinforcements with different fractions. Morphological and mechanical analyses were carried out to investigate the effects of alfa short fibers and natural pozzolan powder on the mechanical properties of the bio-composites.

The results of this study confirmed that the use of natural filler improves the mechanical properties of the recycled HPDE. The tensile tests showed an improvement in Young's modulus, tensile strength, and the modulus of resilience of the three developed bio-composites, on the other hand, decries in toughness was noticed. The results vary with the variation of reinforcement's content.

An increase in tensile strength reached the maximum of 3573 MPa plus an interesting improvement in Young's modulus with a maximum value of 3696 MPa. The toughness of the neat recycled HD-PE decreased by 212% by adding the natural filler whereas the modulus of resilience exhibited an increase of 138% compared to the neat recycled HD-PE.

The obtained results indicate that the recycling of the HPDE reinforced with alfa fibers and pozzolan powder can represent a good potential in improving interfacial properties. The study shows an alternative for producing friendly biodegradable composites that can be a solution for plastic waste reduction and a substitute for petrochemical-derived products.

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