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Research Article

Development of a Composite Material Based on a Polymer and Plant Fibers

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ABSTRACT

The composite materials industry has expanded significantly due to their enhanced performance. However, concerns about the environmental impact of synthetic fibers and petroleum-based polymers have shifted focus toward natural fibers. These fibers are renewable and often biodegradable, making them a sustainable alternative that reduces ecological harm. This shift has paved the way for the development of more eco-friendly composite materials across various industrial applications. This study explores the development of hybrid and non-hybrid composites using high-density polyethylene (HDPE), potato starch, and pomegranate peels. A two-roll mixer and compression molding were employed to fabricate these composites. The findings indicate that incorporating these natural fillers enhances the hardness of the composites. The mechanical results showed that the combination of pomegranate peels/potato starch fillers has a positive effect compared to composites prepared without hybridization. Additionally, the composites exhibit lower density and water absorption properties that vary with the immersion time and the type of filler used. Microscopic analysis reveals that the composite surfaces are heterogeneous and irregular, highlighting the influence of filler distribution. Overall, this research demonstrates the potential of utilizing natural materials in composite production, promoting sustainability while addressing performance needs. Such innovations are essential for reducing the reliance on non-renewable resources and aligning with global efforts to minimize environmental impact.

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1. Introduction

A new approach to polymer composite materials is the hybrid filler, consisting of at least two types of fillers, either both in particle form or fibers, or a combination of particles and fibers. Hybrid composites exhibit superior physical, mechanical, and thermal properties compared to composites with a single type of filler due to the optimization and synergy between the two types of fillers.

Additionally, they offer economic advantages, as some fillers can be very expensive [1–6]. These hybrid polymer composites have potential applications in furniture manufacturing, various automotive industry components, construction elements, and other commercial applications [1], due to their excellent recoverability and biodegradability properties.

Hybrid polymer composites with natural fiber fillers have gained significant attention as a more sustainable alternative to polymer composites with single fillers [6]. However, a major challenge in manufacturing these composites is the incompatibility between hydrophilic natural fibers (due to hydroxyl groups in cellulose) and the hydrophobic polymer matrix [7]. This incompatibility results in weak interfacial adhesion and reduced mechanical strength, which is a major disadvantage of these composites [8]. To address this incompatibility, chemical, physical, biological, and nanotechnology-based fiber treatment methods have been explored to enhance filler adhesion to the polymer matrix.

Various researchers have studied filler adhesion by treating polypropylene-based composites with

fiber fillers using alkali treatments [9], as well as through other advanced methods and studies [10,11].

The compatibilization process introduces a third component that acts as a coupling agent, eliminating incompatibility by improving adhesion between the filler and the matrix. The Palsule process [12] addresses this incompatibility by using a functionalized polymer that reacts with the hydroxyl groups present in natural fibers. The processes researched by Palsule are based on polyethylene functionalized with glycidyl methacrylate and polyethylene functionalized with maleic anhydride by grafting [13–17]. These polymer materials have cutting-edge applications across various industries, particularly in the automotive sector [18–20]. In this context, this study aims to examine the influence of the incorporation of potato starch and pomegranate peel on the mechanical, rheological, physical, and morphological properties of HDPE/potato starch, HDPE/pomegranate peel, as well as HDPE/potato starch/pomegranate peel hybrid composites. The final objective of this research is to promote the development of more environmentally friendly and sustainable composite materials, meeting the growing needs of the industry while reducing the environmental impact.

2. Materials and Methods

2.1. Materials

In this present study, high-density polyethylene (HDPE) thermoplastic resin of grade 5502 was used as the matrix.

Potato starch has been used as a natural filler.

The potatoes were peeled, grated, pressed, and then filtered to recover the potato starch. The latter was air-dried for a week, followed by drying in an oven at 60°C for 24 hours. The dried potato starch was then ground into a fine powder using a porcelain mortar and pestle, then sieved to obtain a flour with a particle size of less than 63µm.

Pomegranate peel was the second filler we used in our study.

The pomegranate peels were washed and air-dried for 4 days. They were ground and sieved to obtain a flour with a particle size less than 63 µm.

2.2. Composites Preparation

Neat HDPE and its composites, no hybrid and hybrid, were mixed using a two-roll mill (IQAP LAP, RLS-110) with a rotational speed of 32 rpm at a temperature of 170 °C and a mixing time of 12 min to ensure effective blending, with the composition described in **Table 1.**

Table 1: Mass compositions of the different formulations.

Formulations/ Compositions	HDPE (%)	Potato starch (%)	pomegranate peels (%)
HDPE	100	0	0
30Ps/0Pp	70	30	0
0Ps/30 Pp	70	0	30
20Ps/10Pp	70	20	10
10Ps/20Pp	70	10	20
15Ps/15Pp	70	15	15

Before melt mixing, the potato starch and pomegranate peels particles were all dried in an oven at 60 °C for 24 h to remove all absorbed moisture. They were then manually mixed with the polymer at a 30/70 wt% ratio of fiber to polymer before being transferred to the two-roll mill. The resulting product that came out of the

two-roll mill was pelletized by a mill into small pieces to prepare it for further processing. After preparing hybrid and non-hybrid composites, the thermocompression technique is then used to fabricate sheets with suitable thicknesses according to the specimen requirements of each analytical technique. This is achieved through the utilization of a hydraulic thermocompression press (IQAP LAP, PLA-30) by placing the small pieces of different formulations within the mold between two insulating polystyrene sheets, which are further sandwiched between two metal plates. The process was manufactured by applying a pressure of 100 kg/cm^2 and a temperature of 177 °C; the preheating duration was 10 min, followed by 1 min of degassing and 7 min of compression. The sheets of different formulations obtained have been cooled to room temperature and subsequently used to prepare test specimens.

3. Characterization

3.1. Mechanical Tests

- *Tensile Test*

The dumbbell-shaped specimens with dimensions of (95 x 13 x 3) mm^3 were placed in a TesT GmbH tensile testing machine, following the ASTM D638 standard, to evaluate their mechanical properties until failure. The deformation speed was set to 20 mm/min, recording the required force at each moment.

- *IZOD Impact Test*

A rectangular test specimen with dimensions (63 x 13 x 3) mm^3 was notched in a V-shape with a depth of 2.5 mm at the center using a CEAST notching machine. It was then subjected to the impact of a 1-joule hammer fixed to the end of a pendulum, which was released to deliver the impact. The test was conducted using CEAST equipment of the Resil Impactor type, compliant with the ASTM D-256 standard, resulting in the specimen breaking.

- *Shore D Hardness Test*

For this evaluation, the Shore D method was employed in accordance with ASTM D-2240 standard for hard polymers. Specimens with dimensions of (50 x 50 x 3) mm^3 were used. The samples were placed under the needle of a CEAST durometer, model 6767, with a load of 4.5 to 5 kg applied. The hardness value was recorded once the needle stabilized its position. Three measurements were taken on each sample at points approximately 3 mm apart and located about 12 mm from the edges of the specimen. The results are reported as the average of the three values obtained.

3.2. Study of Rheological Properties

- *Fluidity Index Test*

To estimate the flow properties of each sample, 3.5 g of each mixture was extruded through a die with a diameter of 2.09 mm and a length of 8 mm at 190°C under a 2.50 kg load, following ASTM D1238 standard, using a Gottfert rheometer. Before the test, the rheometer cylinder was preheated and cleaned, and the die was placed after verifying its diameter, then preheated to 190±2°C for at least 15 minutes. The sample was loaded into the cylinder, preheated without weight for 3 minutes, then with weight for 3 minutes while removing air bubbles. The extruded portion was discarded, and the resin flowed for 10 minutes before being cut and weighed to determine the MFI, which represents the amount extruded in grams per ten minutes.

3.3 Study of Physical Properties

- *Water Absorption Test*

The samples were dried for 24 hours at 100°C in an oven. After drying, they were weighed on a high-precision analytical balance (0.00001g). The samples were then immersed in distilled water at room temperature. Every 24 hours, the samples were removed from the water, quickly dried to remove surface excess water, weighed promptly to minimize errors from evaporation, and then returned to the water. Water absorption was calculated with the following equation:

$$W_a = \frac{W - W_0}{W_0} x100 \quad (1)$$

where W_0 and W are the sample weights before and after immersion in distilled water for one month, respectively?

- *Density Test*

The apparent density is measured by the pycnometric method, according to the standard NFT51-063. Distilled water is the displacement solvent used, ensuring good wettability of the sample [21].

3.4 Morphological Analysis Through Optical Spectroscopy

In order to study the morphology of the materials and verify the dispersion of fibers in the composite materials, optical microscopy is used to take surface photos of the thin films that have already been prepared. The equipment used is an OPTIKA Microscopes ITALY optical microscope.

4. Results and Discussion

4.1. Mechanical Characterization

- *Tensile Test*

The analytical results of hybrid composites made from pomegranate peel fillers and potato starch, obtained from tensile tests, are presented in **Table2.** All composite samples were tested in accordance with standards, and for each composite, a total of five samples were tested to record the average values.

Table 2: The Tensile Test Results

Formulations	Tensile strength (MPa)	Elongation at break (%)	Young's Modulus (MPa)
HDPE	23.863	10.062	237.159
30Ps/0Pp	19.108	7.483	255.352
0Ps/30Pp	16.052	4.386	365.983
20Ps/10Pp	14.796	3.909	378.511
10Ps/20Pp	12.299	2.692	456.872
15Ps/15Pp	13.128	3.135	418.755

A decrease in the tensile strength of fiber composites (Ps/Pp) with different proportions is clearly observed in Fig. 1.

The presence of particles of the fillers used with a hydrophilic nature in the hydrophobic HDPE matrix reduces the tensile strength of the composites developed due to the incompatibility and poor interfacial adhesion between the different constituents.

The impact of hybridization is significant, as hybrid composites generally exhibit lower tensile strength values. This phenomenon is particularly noticeable when the proportion of pomegranate bark particles increases (10Ps/20Pp), which is attributed to the composition of these fibers, thereby reducing the breaking strength.

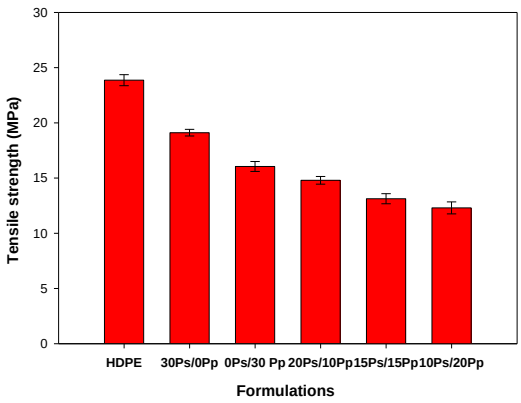

Fig.1. Evolution of the tensile strength of different formulations

The results are predictable and align with numerous studies, including those by Sonia et al. [22], Demir et al. [23], Khalid et al. [24], and Kaci et al. [25]. These researchers have attributed this decrease to the reduction in the bonding strength between the fillers and the matrix, which hinders stress propagation. Additionally, the tendency of the fillers to cluster and form agglomerates leads to heterogeneities and non-uniform stress transfer within the matrix, ultimately resulting in the embrittlement of the composite material.

The variation in the modulus of elasticity of the different composites produced is shown in Fig. 2.

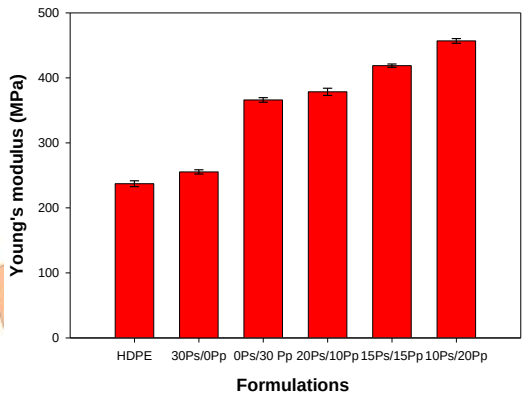

Fig.2. The evolution of Young's modulus of the developed composites.

When potato starch, pomegranate peel, or a combination of both is incorporated into the high-density polyethylene (HDPE) matrix, it results in an increase in the material's stiffness and a reduction in its elasticity. In other words, the Young's modulus increases (Fig. 2), and this increase is particularly significant for the hybrid composite (10Ps/20Pp). This observation can be explained by the fact that the filler particles (Ps/Pp), being rigid, tend to form a reinforcing structure within the composites, which imparts high strength to the material. This phenomenon was also observed by A.Atmakuri et al [26] and R. E. Rowlands [27].

The variation in elongation at break of the different composites developed is shown in Fig. 3. We observe a decrease in the elongation at break of the composites compared to virgin HDPE.

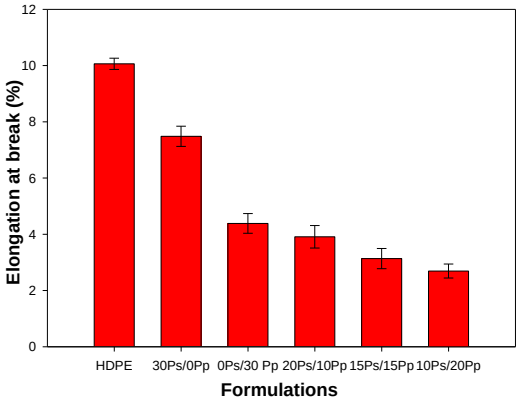

Fig.3. Evolution of the elongation to rupture of developed composites.

This decrease is particularly pronounced for the hybrid composite (10Ps/20Pp). This finding

is in perfect agreement with many authors, such as Pasquini et al [28].

The loss of this property is mainly due to the addition of pomegranate peel and potato starch, which are inherently rigid materials. Their integration into the HDPE matrix reduces the mobility of polymer chains, causing the samples to break more quickly under low stress.

- *IZOD Impact Resistance*

The purpose of the impact resistance test is to measure the energy required to break a notched specimen in a single impact [29]. Fig. 4 illustrates the variation in the transverse resilience of the different composites developed.

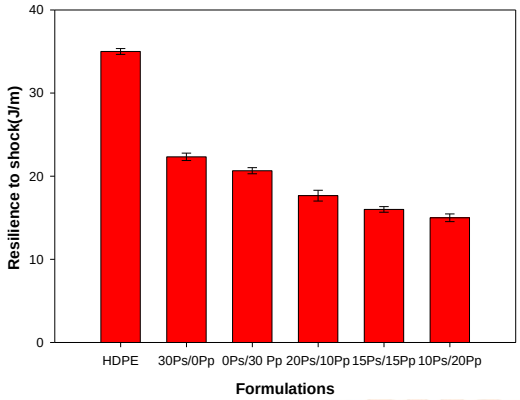

Fig.4. The evolution of shock resilience in advanced composites.

The developed composites show impact resistance values lower than those of virgin HDPE, confirming that our fillers are characterized by high rigidity, significantly increasing the stiffness of the composite materials, which also leads to a decrease in impact resistance. This decrease in impact resilience is also explained by the low interactions (physical interaction) between the matrix and the filler, resulting in poor interfacial adhesion. Among all the composites, hybrid composites demonstrated superior properties compared to single-filler composites, with values being close to each other. This has already been confirmed by the works of Andrzej K. Bledzki et al., and Ajay Karmarkar et al. [30, 31].

- *Shore D Hardness*

The hardness of a particular sample refers to its resistance to penetration when a load is applied, and it depends on the distribution of reinforcement in the matrix [32]. Figure 5 shows the variation in the Shore D hardness of the different composites produced.

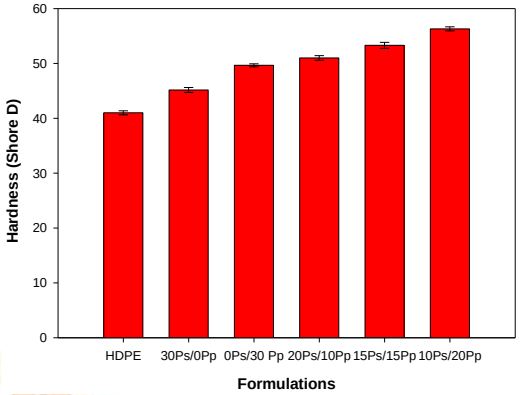

Fig.5. Evolution of the Shore D hardness of the developed composites.

The hardness increases with the incorporation of two types of fillers (Ps/Pp) into the HDPE matrix. This increase is particularly noticeable for the hybrid composite (10Ps/20Pp). These results are expected, as the fillers consist of cellulose microfibrils, classified as hard fibers, which make it more difficult for the durometer needle to penetrate the composite material. This result has been confirmed by J. JACOB Maya [33]. According to Georgopoulos, S.Th [34], hardness values are a measure of wearand abrasion resistance, since hard materials are more resistant to friction.

4.2. Rheological Characterization

- *Measurement of the Fluidity Index (Melt Flow Index, MFI)*

The Fluidity Index (FI or MFI) is the most commonly used test in the polymer industry and sometimes the only rheological information used. It is indicative of the flow characteristics and thus the processability of the polymer in its molten state [35]. The variation in the fluidity index of different developed composites is shown in Fig. 6.

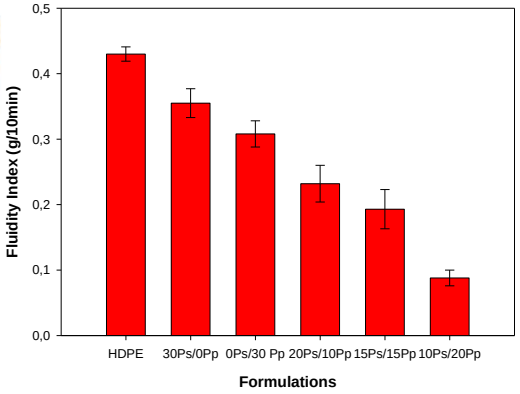

Fig.6. The evolution of the flow index of the developed composites.

It is observed that the flow index of hybrid composites (HDPE/Ps/Pp) and non-hybrid composites (HDPE/Ps) and (HDPE/Pp) is lower than that of virgin HDPE, with hybrid composites showing lower values than non-hybrid ones. The incorporation of rigid fillers into the HDPE matrix significantly reduces the mobility of the polymer chains and causes them to lose their flexibility, which accelerates the fracture of low-stress specimens. This results in a reduction in elongation at break, due to poor filler/matrix interfacial adhesion. Also, this is due to the agglomeration of the two fillers (Ps/Pp), which creates obstacles to the free movement of the polymer chains and hinders the flow of the material, resulting in a very high viscosity that reduces the flow index. The results of this test are consistent with those obtained by Djoudi Tarek et al. [36].

4.3. Physical Characterization

- *Water Absorption Test*

The affinity of plant-based fillers with water is one of the major drawbacks of using them as reinforcement in the field of composites [29]. These fibers are hydrophilic and therefore undergo significant deformations when exposed to a humid environment. The absorbed water leads to swelling of the fillers, and the matrix structure may also be affected by water absorption, such as chain reorientation and shrinkage [36]. The evolution of water absorption rates in the developed composites is shown in **Fig. 7.**

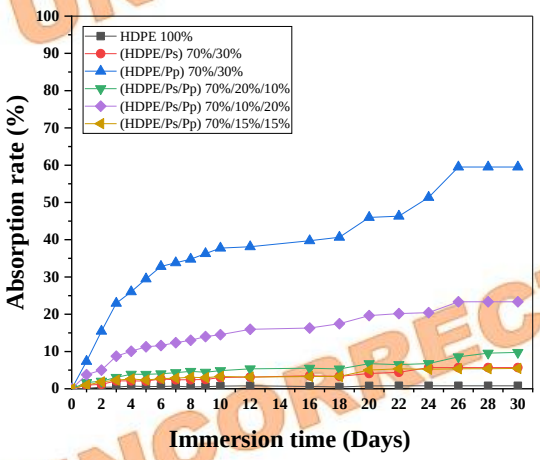

Fig.7. The evolution of the water absorption rate of the composites developed.

This test confirmed that the matrix (HDPE) is hydrophobic. Indeed, its water absorption rate remains below 0.78%, regardless of the immersion time. This increase is significantly visible in the composite (HDPE/Pp), where its absorption rate reached 59.5%, and the hybrid composite (10Ps/20Pp) reached 23.4%. This increase is due to the higher concentration of hydroxyl groups, which have a strong affinity for water. This phenomenon can also be attributed to the poor adhesion between the filler and the matrix, leading to an increase in microvoids. The results obtained are consistent with studies by K. Mazur et al [37].

- *Density*

The low density of lignocellulosic material is one of the major advantages of using it as a filler in thermoplastic-based composites [6]. The results of the density test for the PEHD/Ps, PEHD/Pp, and PEHD/Ps/Pp composites are presented in Fig. 8.

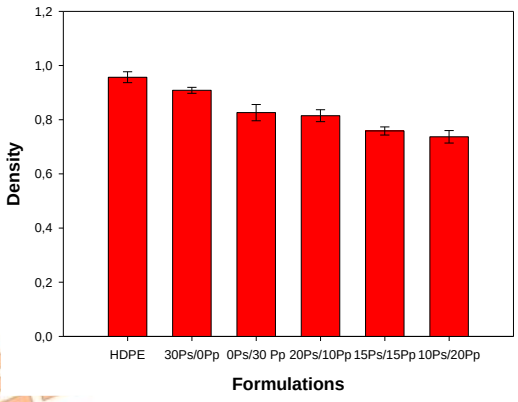

Fig.8. The evolution of the density of the developed composites.

We observe a decrease in density with the incorporation of the different fillers (Ps/Pp) into the HDPE matrix. This can be explained by the low density of these fillers, which are 0.8273 and 0.6577, respectively. Thus, the integration of these two fillers into high-density polyethylene allows for the creation of low-density composites. The 10Ps/20Pp composites exhibited the lowest density as they contain 20% Pp. Similar results have been reported by EDHIREJ Ahmed et al. [38].

4.4. *Characterization of the Dispersion State of Fillers in High-Density Polyethylene (HDPE) Matrix*

Among the commonly used methods for evaluating the quality of the interface between the load and the matrix, which also rely on the effects of enhanced adhesion and proper dispersion [29], we chose to use optical microscopy in our study to characterize the morphology of the surfaces of the developed composites. Figure 9 presents the images acquired by an optical microscope of virgin HDPE as well as the different developed composites.

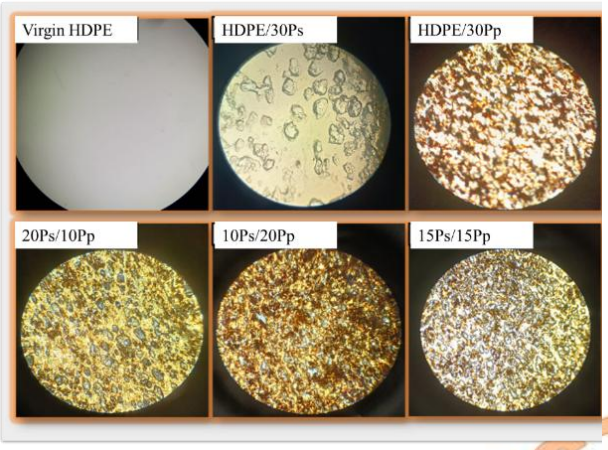

Fig.9. Optical microscope imaging of the surfaces of the developed composites and virgin HDPE.

The micrograph of the surface of virgin HDPE shows a homogeneous surface. In contrast, the micrographs of the composites (HDPE/30Ps), (HDPE/30Pp), as well as those of the hybrid composites (20Ps/10Pp), (10Ps/20Pp), and (15Ps/15Pp) reveal a heterogeneous and irregular surface, marked by the presence of distinct aggregates from the high-density polyethylene matrix. These aggregates are more frequent in the hybrid composites due to the incompatibility between the two phases. This incompatibility arises from a low interfacial adhesion between potato starch and pomegranate peel, both of which are hydrophilic, and hydrophobic HDPE. At the same time, good dispersion of the fillers (Ps/Pp) is observed, particularly in the hybrid composites, due to the size of the filler particles (63 µm).

5. Conclusions

This study aimed to develop hybrid and non-hybrid composites using transformation processes, calendering using a two-roll mixer, followed by compression molding. The composites were developed from a thermoplastic matrix of high-density polyethylene (HDPE) combined with potato starch and pomegranate peels. The mechanical, rheological, physical, and morphological characteristics of these composites were then studied. The analysis of the experimental results led to the following main conclusions:

➢ The mechanical analysis of the composites produced highlighted several significant conclusions: The results demonstrate a significant increase in the hardness of composites made from HDPE and potato starch, pomegranate peel, or a combination of both fillers, compared to virgin HDPE. However, this increase in hardness is accompanied by a reduction in resilience compared to virgin HDPE. Remarkably, the mechanical behavior of the composites reveals a decrease in stress and elongation at break, as well as a progressive increase in the modulus of elasticity.

➢ Rheological characterization, performed by measuring the melt flow index, showed a decrease in this index for HDPE composites filled with either potato starch, pomegranate peel, or both fillers combined, compared to the value obtained for virgin HDPE.

➢ Physical characterization, based on density and water absorption measurements, revealed the following: Composites made of HDPE/potato starch, HDPE/pomegranate peel, or the various hybrid composites HDPE/potato starch/pomegranate peel, exhibit a significantly lower density than virgin HDPE, suggesting that the fillers used contribute to making the composites lighter. Furthermore, the water absorption rate of the composites developed is influenced by both the immersion time and the nature of the fillers incorporated in the polymer matrix.

➢ Morphological analysis by optical microscopy of the HDPE matrix reveals a homogeneous and regular surface, while the hybrid (HDPE/potato starch/pomegranate peel) and non-hybrid composites (HDPE/potato starch, HDPE/pomegranate peel) present a clearly heterogeneous and irregular surface, with clearly visible aggregates.

➢ These trends are particularly pronounced in the case of the hybrid composite containing 10% starch and 20% pomegranate peel.

Further research is needed to functionalize the natural fillers to enhance their compatibility and uniform distribution within the HDPE polymer matrix and study the thermal properties.

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