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Original article

Mechanical Properties of Styrene-Butadiene Rubber Composites Made with Mixed Local Libyan Grasses "Halfa and R'tem"

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Abstract
The paper aims to investigate the effect of using a mixture of two local Libyan grasses (Halfa and R'tem) as reinforcing fillers for styrene-butadiene rubber (SBR). SBR composites with different filler content (10, 20, 30, and 40 wt%) were prepared by extrusion process. The effect of these grasses on the mechanical properties such as tensile strength, elongation at break, impact strength, and micro-hardness was investigated. Results indicated that the highest stress at break value was exhibited by the composite made with 20% filler content. Contrarily, all composites containing 10, 30, and 40 wt% of the filler had lower stress at break than that of pure SBR. Results also indicated that the addition of these grasses significantly decreased the elongation at the break of the SBR polymer. As the amount of the filler increased, the elongation at the break of SBR decreased significantly. However, the impact strength of SBR composites showed a considerable increase with the addition of the same grasses, where composites containing 20 wt% filler had the maximum impact strength value. Similarly, the micro-hardness of the composite was increased with the addition of the grasses, where composites containing 40 wt% grasses had the maximum micro-hardness value. Optical microscope images demonstrated various defects in composites made with 10, 30, and 40 wt% grass content, including the formation of filler aggregation and the presence of bubbles. None of these were observed in a composite made with 20 wt% grass content, which was in good agreement with the mechanical properties results.
Keywords. Polymer Composites, Mechanical Properties, Local Grasses, Halfa, R'tem.

Introduction

Composites are created by combining two or more constituent materials with dissimilar chemical or physical characteristics. When these materials are combined, new properties will be created that are better than the original materials [1]. Composites usually comprise one continuous phase known as a matrix together with one (or more) discontinuous phase that is called reinforcement filler. The matrix carries out several essential roles, such as keeping the filler oriented and spaced properly and shielding it from environmental factors and abrasion. Compared to the matrix, the reinforcing filler provides new properties such as rigidity and strength, where the resultant composite materials become tougher, stronger, and stiffer. The matrix is usually made from polymers, metals, or ceramics and the reinforcement filler is often a fiber or particle. Therefore, composites come in three different varieties: polymer matrix composites (PMCs), ceramic matrix composites, and metallic matrix composites.

Probably the most commonly used class of composites is PMCs, because of their improved mechanical, thermal, and tribological properties as well as their low weight and cost [2]. The scientific and technological communities have long been interested in PMCs, which are now acknowledged as the most appropriate material for a variety of engineering applications due to their high stiffness and strength [3]. Moreover, PMCs are renowned for their affordable prices and simple manufacturing process [4]. They are made with specific fillers and/or fibers that are combined with a polymer matrix to provide the appreciated properties, which are essential for many industries, including aerospace, automotive, electrical, marine, sports, and others [3-4]. For all of the facts and advantages mentioned above, PMCs have become known as the most popular and rapidly growing composite materials because of their inherent qualities and vast range of uses [5].

PMCs can be divided into two main categories: thermoset (epoxies, polyesters, phenolics, etc.) matrix composites and thermoplastic (e.g. low-density polyethylene, high-density polyethylene, polypropylene, polyvinyl chloride, nylon, and acrylics, etc.) matrix composites [6-7]. On the other hand, a wide variety of reinforcement material types and forms are used in PMCs. Fibers (natural or synthetic) and fillers (organic or inorganic) could be used to reinforce polymers. However, because of their advantages over traditional synthetic fillers, natural fibers (NFs) have been gaining interest from researchers and scientists as an alternative reinforcement in polymer composites [8]. Recently, composites containing NFs, which are often referred to as NF-reinforced polymer composites (NFRPCs), have grown in value, dramatically. NFs (such as hemp, sisal, jute, kenaf, flax, coir, banana, and many others) have been used as reinforcing materials (fillers) in these PMCs [9-10]. They have a

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relatively low cost of extraction and processing [11]. The resultant PMCs made with NFs can be recyclable, renewable, and sustainable. Moreover, they are eco-friendly, lightweight, strong, cheap, and biodegradable [8]. Most importantly, the synthesis of NFRPCs is dependent on parameters including fiber content, fiber orientation, fiber dimension, fiber placement, polymer matrix, and the adhesion between filler and polymer matrix, which may result in a wide composite with various properties.
In recent years, the interest in new fiber sources, including grass, aquatic plants, herbaceous plants, crops, and their byproducts, has grown significantly [12]. Similar to other NFs, grass cell walls contain cellulose, hemicelluloses, protein, lignin, cutin, waxes, and minerals [13]. In previous studies carried out by our group [14-16], Libyan grasses such as Stipa tenacissima (locally known as Halfa), Stipagrostis pungens (locally known as Esbat) and Retama raetam (locally known as R'tem) found to be a good alternative to produce low cost and low density (i.e., lightweight) polymer composites with acceptable mechanical properties. These types of grasses are typically found in hot and dry areas in Libya. In these studies, Halfa appeared to be relatively better than Esbat and R'tem in reinforcing styrene-butadiene rubber (SBR). Furthermore, as shown by our group when Halfa and Esbat are mixed SBR composites with different properties can be obtained [16]. Results indicated that mixing Halfa and Esbat appears to be relatively better than individual Halfa or Esbat for producing composites with good strength properties. On the other hand, individual Halfa or Esbat showed to be better than mixing them in producing composites with relatively better impact strength and hardness. According to Alessandro et al. [17] these composites could be termed as hybrid PMCs because they contain more than one type of fiber as reinforcement in a single polymer matrix. In the current study, the effect of mixing Halfa and R'tem as a filler on the mechanical properties of SBR composites was investigated to draw an accurate conclusion about the use of these grasses. This is crucial, after employing these local grasses as reinforcing filler separately.

Material and methods

Materials

Halfa (Libyan local name for Stipa tenacissima grass) and R'tem (local name for Retama raetam grass) were utilized as fillers. They were collected from a dry area near the Libyan city of Nalut. The grasses were cleaned with distilled water and dried before being used. They were then chopped and sieved to achieve fivers with particle size of 212 μm. SBR from Parc Scientific (UK) was employed as a matrix.

Preparation of SBR composites

Halfa and R'tem were mixed and dried in an oven at 60 °C for 4 h. They were mixed with SBR using twin screw extruder (Brabender, Germany) (L/D ratio of 48) with a speed of 70 r.p.m. at 180 °C. Various composites were prepared with different filler content as shown in Table 1. Composites containing individual Halfa or R'tem (from a previous study carried out by our group [15]) are also displayed in Table 1 for comparison.

Table 1. Composite compositions and their codes.

Sample	SBR, wt%	Halfa, wt%	R'tem, wt%
SBR	100	0	0
Composites made with Halfa or R'tem individually [15]			
SBRH1	90	10	0
SBRH2	80	20	0
SBRH3	70	30	0
SBRH4	60	40	0
SBRR1	90	0	10
SBRR2	80	0	20
SBRR3	70	0	30
SBRR4	60	0	40
Composites made with Halfa and R'tem combined			
SBRM1	90	5	5
SBRM2	80	10	10
SBRM3	70	15	15
SBRM4	60	20	20

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Characterization

Samples Preparation

Tensile and impact test specimens were prepared using injection molding machine (Xplore 12 ml, Netherlands) with injection temperature of 180°C, packing pressure of 10 Bar and packing time 1 sec.

Tensile strength test

Tensile test measurements were carried out at the Industrial Research Center (Tripoli, Libya) using QC-506M1machine (Cometheck) at room temperature. Four specimens (73 mm - 4 mm - 2 mm) were tested for each composite specimen under speed test of 100 mm/min.

Impact strength test

Charpy impact test was carried out using (CEAST Resil Impactor tester) with impact energy of 15 J at room temperature. The specimens for impact test were prepared and notched according to ASTM (D256-10). A minimum of five specimens were tested and an average value was taken.

Micro-hardness test

Micro-hardness test was performed using micro-Vickers hardness tester (MVT-1000Z) at room temperature. The samples were measured at 100 gf load and 10 s dwell time. The micro-hardness value for each specimen was taken as an average of at least 10 indentations, which were randomly made in each sample.

Morphological properties

Microscopic observations of SBR composites were obtained by an optical polarizing microscope (XP-501, Turkey), equipped with a color digital camera (Moticam 2) and software (Motic Images Plus 2) at different magnifications.

Results and Discussion

Mechanical properties of composites made with Halfa or R'tem

As shown in Table 2, the stress at break of SBR increased only with the addition of 20 and 30 wt% of Halfa. The highest value of stress at break was obtained by composite made with 20 wt% Halfa. All composites made with R'tem had lower stress at break than pure SBR and composites made with Halfa. The elongation at break was significantly decreased with the addition of Halfa or R'tem. Generally, the elongation at break of the composites made with R'tem was slightly higher than that of composites made with Halfa. On the other hand, the addition of Halfa resulted in composites with relatively higher impact strength.

Table 2. Mechanical properties of pure SBR and its composites made with Halfa or R'tem.

Sample	Stress at break, N/mm2	Elongation at break %	Impact strength, KJ/m2	Micro - hardness
SBR	29.17 (0.8)	25.63 (2.9)	3.50 (0.07)	7.20 (0.3)
SBRH1	27.49 (1.3)	12.17 (1.1)	3.53 (0.06)	7.41 (0.2)
SBRH2	35.12 (1.4)	7.04 (1.2)	4.28 (0.07)	8.40 (0.4)
SBRH3	30.66 (1.1)	5.75 (0.9)	4.89 (0.07)	8.83 (0.4)
SBRH4	29.01 (1.3)	3.53 (0.9)	4.84 (0.06)	9.92 (0.7)
SBRR1	27.52 (2.3)	17.50 (1.2)	3.93 (0.06)	7.55 (0.2)
SBRR2	25.19 (1.6)	11.53 (0.8)	3.66 (0.06)	8.52 (0.2)
SBRR3	22.55 (1.1)	8.75 (0.7)	3.75 (0.08)	9.41 (0.3)
SBRR4	20.67 (0.91)	4.98 (0.6)	4.01 (0.07)	10.23 (0.5)

The standard deviation is given in parentheses.

The impact strength of these composites was increased with increasing the Halfa content up to 30 wt% after which it was decreased noticeably. However, the impact strength of composites made with R'tem did not follow a clear path with increasing R'tem content. Furthermore, the incorporation of Halfa or R'tem resulted in a considerable increase in the micro-hardness of the composites. However, R'tem was shown to produce composites with higher micro-hardness compared to those made with Halfa. This means that composites made with Halfa possess better toughness and ductility to a certain degree in comparison to composites made with R'tem. Therefore, Halfa appears to be relatively better than R'tem in reinforcing SBR. The results of this part have been discussed in detail in a previous study carried out by our group [15].

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Mechanical properties of composites with Halfa and R'tem combined

Mechanical properties, namely stress at break, elongation at break, impact strength and micro-hardness of composites made with Halfa and R'tem are shown in Table 3. Although values are different, however the mechanical properties are mostly similar to those observed for composites made with individual Halfa or R'tem. Figure 1 shows the stress at the break of pure SBR and its composites prepared with a mixture of Halfa and R'tem. Figure 2 shows the elongation at break of pure SBR and its composites prepared with Halfa and R'tem.

Table 3. Mechanical properties of SBR and its composites with Halfa and R'tem combined.

Sample	Stress at break, N/mm2	Elongation at Break, %	Impact strength, KJ/m2	Micro - hardness
SBR	29.17 (0.8)	25.63 (0.9)	3.50 (0.1)	7.20 (0.3)
SBRM1	24.30 (1.1)	18.85 (0.9)	4.68 (0.4)	8.63 (0.3)
SBRM2	32.17 (0.9)	8.09 (0.8)	4.56 (0.5)	8.98 (0.3)
SBRM3	26.99 (0.8)	6.06 (0.6)	4.15 (0.1)	9.13 (0.4)
SBRM4	22.12 (1.2)	3.95 (0.3)	4.37 (0.1)	9.14 (0.3)

The standard deviation is given in parentheses.

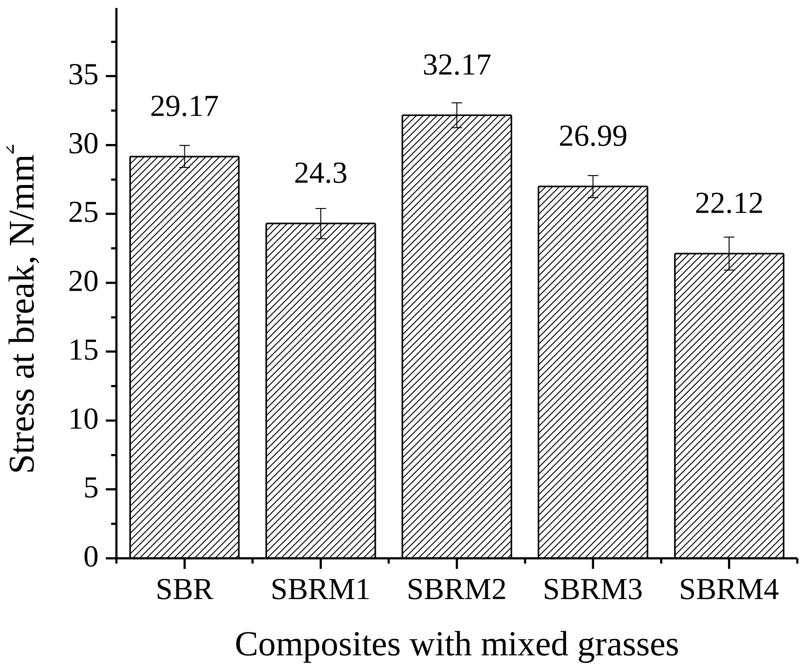

Figure 1. Stress at break of pure SBR and its composites prepared with Halfa and R'tem.

As shown in Figure 1, the composite made with 20 wt% of Halfa and R'tem exhibited the highest stress at break value. This was similar to the composite made with Halfa (see Table 2). The stress at break of other composites was lower than that of pure SBR. Correspondingly, the stress at break values were higher in composites containing 20 and 30 wt% mixed grasses than in other composites, which is similar to what was found in the case of using Halfa alone. This was not in agreement with the results of a previous investigation carried by our group where we studied the effect of adding Halfa and Esbat on the mechanical properties of SBR [16]. Those findings showed that mixing Halfa and Esbat had a greater effect on stress at break values of the SBR composites than those made from individual ones. Composites made with a mixture of Halfa and Esbat had higher stress at break values compared to pure SBR and composites made with Halfa or Esbat individually.

As stated by Ku et al. [18], tensile properties of NFRPC are primarily determined by interfacial adhesion between the fibers and polymer matrix. They assert that the NFRPC's tensile strengths increase to the optimal or maximum value before decreasing as the fiber content increases. Ichim et al. [19] claimed that the composite's tensile strength starts to increase as the amount of reinforcing fiber increases until it approaches a certain level. At this level, additional increase in fiber content would cause a weakening in the interfacial adhesion between the fibers and the matrix, as the fibers come too close together. This could lead to a reduction in the tensile strength. In fact, numerous factors may influence the qualities of composites, including reinforcing filler

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type, filler ratio, filler shape and dimensions, orientation and placement of the filler inside the matrix, interfacial adhesion between the fibers and the matrix, processing type and conditions [20].

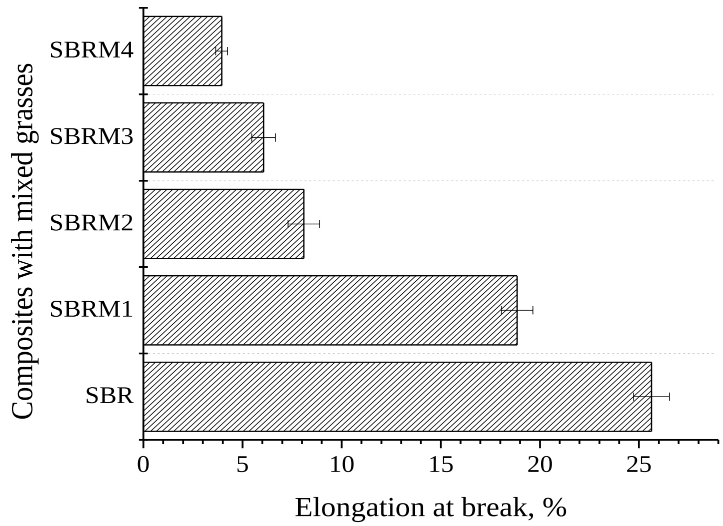

Figure 2. Elongation at break of pure SBR and its composites prepared with Halfa and R'tem.

As revealed in Figure 2, the SBR's elongation at break was drastically decreased when Halfa and R'tem were added. Furthermore, as the Halfa and R'tem content increased the elongation at break was considerably decreased. The same pattern was observed when each grass was added individually (see Table 2). Many researches have reported this common finding [21-25]. Higher content may produce composites with higher strength and lower elongation at break values [21]. This means that the composites may become more rigid and brittle as the filler loading increases, which would cause the elongation at break to decrease concurrently [23]. Ismail et al. [24] claimed that the increase in the stiffness of PMCs led to decrease its ductility, which at the end lowered its elongation at break. Moreover, the addition of more filler content tends to impose extra resistance to flow and therefore leads to lower resistance to break [25]. This indicates that an increased restriction to the macromolecules' molecular mobility is anticipated as filler loading increases [26]. Similar findings were observed by Meissner and Rzymski [27], which showed that the elongation at break decreased as a result of increased filler content in the rubber matrix. They attributed this to the increased stiffness of the composite as a result of the filler loading.

As depicted in Figure 3, the addition of Halfa and R'tem combined resulted in a considerable increase in the impact strength of the composite. Adding more mixed grasses had less effect on the impact strength of SBR composites, similar to what resulted when these grasses were employed individually. The highest impact strength was obtained by composites with 10 wt% (4.68 KJ/m2) and 20 wt% (4.65 KJ/m2) mixed grasses content.

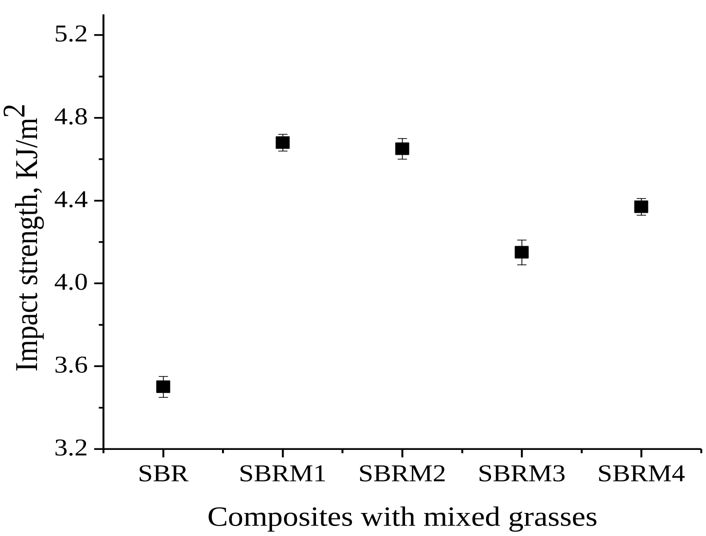

Figure 3. Impact strength of pure SBR and its composites prepared with Halfa and R'tem.

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When Halfa was used alone, the highest impact strength was obtained by composites with 30 wt% (4.89 KJ/m2) and 40 wt% (4.84 KJ/m2) Halfa content. Whereas, the highest impact strength was obtained by composites with 30 wt% (3.75 KJ/m2) and 40 wt% (4.01 KJ/m2) when R'tem was used alone. Using a combination of Halfa and Esbat grasses in the prior investigation also revealed similarities in the impact strength behavior [16]. These results might suggest that, even at high content, these grasses have considerable effect on impact strength properties. In general, PMC's impact strength is affected by filler type, filler qualities, filler content, matrix properties, and filler and matrix interface properties [28-30].

Figure 4 shows the micro-hardness of pure SBR and composites prepared with Halfa and R'tem. As can be seen in this figure, micro-hardness was increased with the addition of Halfa and R'tem combined (shown also in Table 3). This was in agreement to the micro-hardness results obtained when Halfa or R'tem was utilized individually (see Table 2). As shown in Figure 4, micro-hardness increased noticeably as the amount of Halfa and R'tem in the composites increased. SBR had the lowest hardness value of 7.20, whereas the composite containing 40 wt% Halfa and R'tem SBRM4 had the highest micr-hardness value of 9.14. However, the composite made with 40 wt% Halfa had the highest micro-hardness value of 9.92. Also, the composite made with 40 wt% R'tem had a highest value of 10.23. Similar behavior was also seen in a previous study carried by our group using a combination of Halfa and Esbat [16]. This is in a good agreement with other studies, which showed that the addition of NFs to polymers helps improve the hardness [31-33]. The filler's capacity to disperse stress and withstand deformation under pressure is the reason leads to the increase in hardness [32]. On the other hand, poor bonding between the filler and the polymer matrix negatively affects the composites' hardness [33]. In general, a number of variables, including the fibers' intrinsic hardness, the matrix material, the filler content, the fiber-matrix contact, the composite's overall density and homogeneity may affect the hardness of the composites [32].

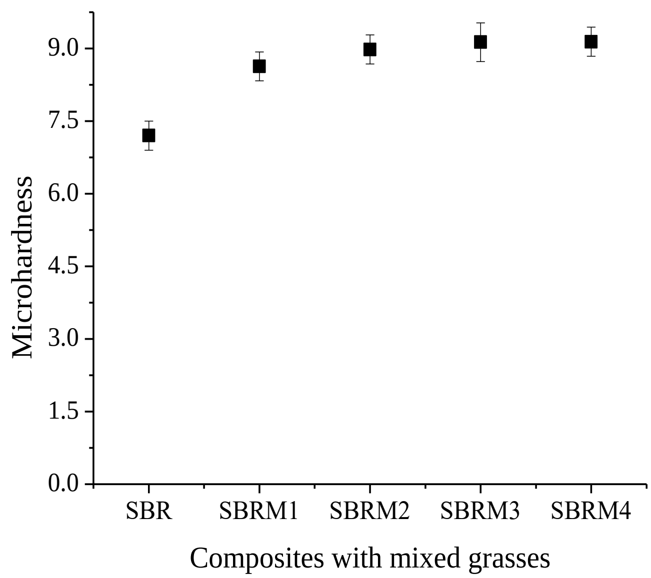

Figure 4. Micro-hardness of pure SBR and composites prepared with Halfa and R'tem.

It must be emphasized that utilizing these studied grasses individually or in combination as reinforcing filler will enhance the value of these native grasses and provide new possibilities for the production of inexpensive and environmentally friendly PMCs. Using them individually or in combination appears to produce composites with enhanced mechanical properties. For instance, Halfa appears to be relatively better than Esbat and R'tem in reinforcing SBR. Moreover, mixing Halfa and Esbat appears to be considerably better than individual Halfa or Esbat to produce composites with decent strength. On the other hand, individual Halfa or Esbat) appear to be better than mixing them together in producing composites with relatively better impact strength and hardness [16]. Contrary, compared to Halfa or R'tem alone, mixing Halfa and R'tem were less effective as reinforcing filler for the SBR. The optimum amount of these grasses that might result in composites with good mechanical properties (in both cases, individual or combined) is between 20 and 30 wt% relative to polymer.

Morphological properties

The optical micrographs of the composites prepared with Halfa and R'tem are shown in Figure 5. It is clear that a better interfacial adhesion was observed by composite containing 20 wt% of Halfa and R'tem content.

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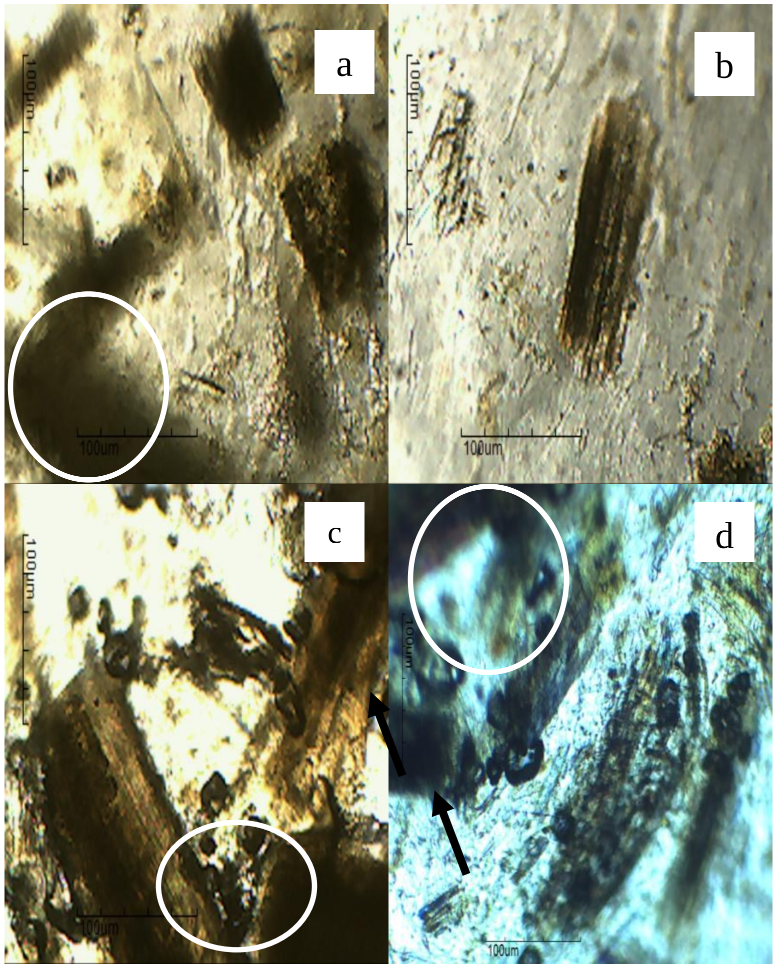

Figure 5. Optical micrographs of composites with mixed grasses content of a) 10%, b) 20%, c) 30% and d) 40%.

The composite made with 20 wt% of Halfa and R'tem did not show any formation of filler aggregation, filler pullout, filler breakage, cracks, or bubbles. On the other hand, composites made with 10, 30, and 40% Halfa and R'tem content clearly exhibited the formation of filler aggregation and bubbles. Composites made with 10, 30, and 40 wt% of Halfa and R'tem displayed the formation of aggregation (Figures 5a, 5c, and 5d). Moreover, a composite made with 40 wt% Halfa and R'tem exhibited the presence of bubbles (see Figure 5d). Consequently, the mechanical properties of composites decreased noticeably when 10, 30, and 40 wt% Halfa and R'tem were used due to the formation of aggregation and the presence of bubbles. The formation of aggregation and the presence of bubbles may restrict the interaction between the filler and polymer matrix, resulting in poor interfacial adhesion, and therefore lowing the mechanical performance of the final composites [34].

Conclusion

Mechanical properties of SBR polymer were examined about the addition of local Libyan grasses combined (Halaf and R'tem) as a filler. Various composites made with 10, 20, 30 and 40 wt% filler content were produced. Additionally, a comparison study assessing the use of individual (from a previous study) and a mixture of these grasses has been accomplished. It is important to point out that composites made from these grasses, whether individually or combined, may result in good mechanical properties. The highest stress at break value was exhibited by the composite made with 20 wt% Halaf and R'tem together, which was similar to what observed Halfa was only used as the filler. The stress at break values of other composites was lower than that of SBR. Also, the addition of a mixture from Halaf and R'tem significantly decreased the elongation at break. As the amount of Halaf and R'tem increased, the elongation at break decreased significantly. However, the addition of Halaf and R'tem resulted in a considerable increase in the impact strength of SBR. Similar to composites made with Halaf or R'tem was used individually, adding more of Halaf and R'tem mixture had a considerable effect on the impact strength of SBR. Composites containing 20 wt% Halaf and R'tem together had the maximum

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impact strength. Likewise, micro-hardness was increased with the addition of more Halaf and R'tem. This was in a good agreement with the results obtained when Halaf or R'tem was used individually.

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المستخلص

يهدف هذا البحث إلى دراسة تأثير استخدام خليط مكون من نوعين من الأعشاب الليبية المحلية (الحلفا والرتم) كمواد مالئة معززة لمطاط ستايرين-بيوتادين (SBR). تم تحضير مركبات SBR بمحتوى مختلف من المادة المالئة (10، 20، 30، و40٪ وزنا) عن طريق عملية البثق. تم دراسة تأثير هذه الأعشاب على الخصائص الميكانيكية مثل قوة الشد والاستطالة عند الكسر وقوة التأثير والصلادة. أشارت النتائج إلى أن المادة المركبة المصنوع بمحتوى مادة مالئة بنسبة 20٪ وزنا أظهر أعلى إجهاد عند الكسر. وعلى النقيض من ذلك، فإن جميع المواد المركبة التي تحتوي على 10 و30 و40٪ وزنا من المادة المالئة كان لها إجهاد أقل عند الكسر من مطاط ستايرين-بيوتادين النقي. أشارت النتائج أيضًا إلى أن إضافة هذه الأعشاب قللت بشكل كبير من الاستطالة عند الكسر للبوليمر SBR. مع زيادة كمية المادة المالئة، انخفضت الاستطالة عند كسر ل SBRبشكل كبير. ومع ذلك، أظهرت مقاومة الصدم ل SBR زيادة ملحوظة مع إضافة نفس الأعشاب، حيث سجلت المركبات التي تحتوي على 20٪ وزنا من المادة المالئة أقصى قيمة لمقاومة الصدمات. وبالمثل، ازدادت الصلادة للمادة المركبة مع إضافة الأعشاب، حيث سجلت المادة المركبة التي تحتوي على 40٪ وزنا من الأعشاب أقصى قيمة للصلادة. أظهرت صور المجهر الضوئي عيوبا مختلفة في المواد المركبة المصنوعة من 10 و30 و40٪ وزنا من الاعشاب، بما في ذلك تكون تكتلات للمادة المالئة ووجود فقاعات. لم يلاحظ أيٌ من هذه العيوب في المادة المركبة المصنوعة من 20٪ وزنا من خليط الأعشاب، وهو ما يتوافق تماما مع نتائج الخواص الميكانيكية.

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