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Effect of Wool Fiber on Structural and Mechanical Properties of Styrene-Butadiene Rubber Copolymer

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Abstract. In this work, a blend of styrene-butadiene rubber (SBR) filled with wool fiber (WF) was successfully prepared through compression molding using tetramethyl thiuram disulfide (TMTD) as a catalyst. The WF mixing to SBR with the proportion varying from 25 to 150 phr concerning the total weight of SBR. The effect of adding WF on the mechanical properties was examined. Also, the morphology and chemical structure properties were examined by used a scanning microscope (SEM) and Fourier infrared (FTIR), respectively. The results showed that the tensile strength and the elongation at the breaking stage are gradually reduced with the adding of WF. This may be due to WF are blended with the rubber randomly. The SEM images of WF/SBR blends showed some voids in the SBR matrix and weak adhesion between WF and SBR interface. The FTIR results revealed that the reaction of WF with the SBR matrix occurred to confirm the WF grafting SBR.

Keywords: SBR, Wool, FTIR, SEM, Tensile strength, elongation at break.

1. Introduction

Different kinds of fillers with SBR have been studied during the last few years to improve their mechanical and physical characteristics. Recently, the employing of natural organic fillers is the most crucial factor because of their lightweight, environment-friendly, and cost-effective [1, 2]. SBR is the most crucial synthetic rubber (elastomers) and is the most commonly used replacement for the NR. The SBR is a highly random copolymer consisting of two different types of monomer units includes butadiene and styrene, in 23 percent as raw materials that have come from oil at the distillation process in the oil refineries. SBR is prepared by the addition of copolymerization of one part of styrene to three parts of butadiene based on the weight, the structure of SBR is nearly 80% trans around the double bond, as shown in Figure 1 [3].

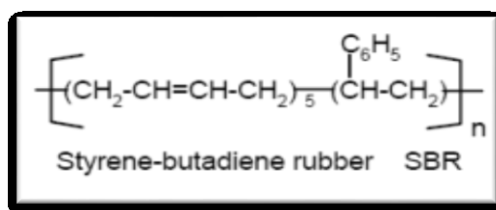


Figure 1. The structure of SBR[3].



Macro SBR and micro SBR are different in their structure, and their differences can have essential effects on the characteristics of the polymer. Macro-structural variables include:

1. Monomer ratio, including the possibility of additional monomers.
2. Average molecular mass.
3. Molecular mass distribution.
4. Linearity or otherwise of the molecular structure.

Micro-structural variables include:

1. Cis-1, 4/ trans-1, 4/ vinyl1, 2 group ratios in the butadiene segment as shown in Figure 2.
2. Distribution of styrene and butadiene units, the arrangement of styrene and butadiene units can have random, partially-block, or block character.

The properties are also influenced by styrene content; with increasing the styrene content, the rubbers are more rigid and less rubbery mutual [4].

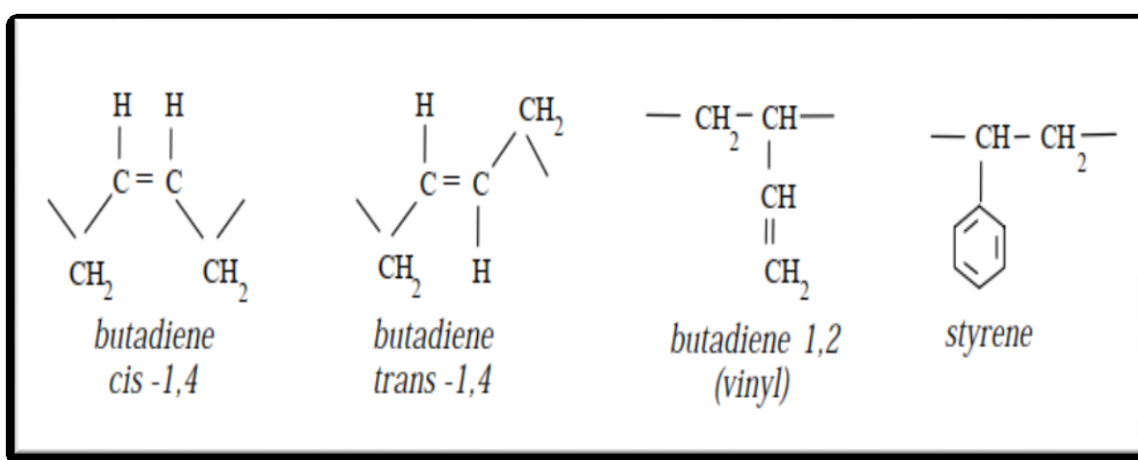


Figure 2. Structural units of styrene-butadiene [4].

Wool (W) is a biopolymer that has several properties, and it is natural, cheap, abundantly obtainable, renewable, and 100 % biodegradable materials that do not contain any chemical materials or other environmental pollutants [5]. Also, W is known as a useful source that has many nutrients and organic substances and has an excellent absorption capacity and water retention, which prevents the drying of the soil. The hydrophilic nature of wool (W) is associated with the presence of polar functional groups (carboxyl, hydroxyl, and amine group) [5].

Thus, the effective use and recycling of animal waste convert them into high-value outputs that significantly impact and lead to an eco-friendly environment [6]. The wool does not accumulate in nature, which indicates the presence of natural degradation for it. The proteolytic enzymes are a group that can hydrolyze the insoluble keratins more than the other proteases, which are called keratinases. These enzymes can be produced from insects and generally through microorganisms. The keratinases produced from the dermatophyte are the best genera *Microsporum* and *Trichophyton* and bacteria of the genera *Bacillus* and *Streptomyces* [7].

The cost of SBR production is higher than the cost of wool production; so, the blends of SBR and wool will decrease the cost of products and become appropriate for industries. This research is the first challenge that shows the SBR modified by WF in which W and SBR are attached using tetramethyl thiuram disulfide (TMTD) as the catalyst.

Thus, the recycling of W in synthetic rubber of styrene-butadiene rubber (SBR) might decrease the production price, enhance the biodegradation property and plant growth, which contributes to the improvement of the environmentally friendly and sustainable agriculture. The information gathered from this research will help researchers and industrialists to select materials with optimized properties for agricultural applications.

2. Experimental

2.1. Materials and methods

2.1.1. Materials. The used materials in this work are the supplied wool from animal waste obtained from sheep, the synthetic rubber type of SBR 1502 was provided by Babel Tires Factory (SBR 1502, Shell Chemical Company), and the urea fertilizer that is obtained from the General Company for Southern Fertilizers, Iraq. The curing agent, TMTD, is supplied by Al-Kiiubar Company, Saudi Arabia. The suppliers of stearic acid and zinc oxide (ZnO) were Acidchem-International CO., Malaysia, and ChemTAL (Malaysia) SDN-BHD, Malaysia.

2.1.2. Preparation of wool. Sheep wool of 750g was taken and thoroughly cleaned from dirt, washed with distilled water to remove suspended dust, and dried under the atmospheric condition. Wool fibers were then manually cut into tiny pieces using scissors.

2.1.3. Preparation of the wool/styrene-butadiene blends (W/SBR). The blends were prepared by mixing the WF to SBR with the percent alter from 25 to 150 phr concerning the total weight of SBR, as illustrated in Table 1. First, a specified amount of rubber (SBR) was passed through the mill with two-roll (Comerio Ercole Busto Arsizio, Italy) for softening. The accelerators (TMTD), zinc oxide (ZnO), and stearic acid were added to SBR. After that, the WF was slowly added to this blend until a homogeneous mixture was obtained. The specimens were prepared by vulcanization of previous mixtures in a compression molding (Moore company, England) under a temperature of 145 C and a pressure of 3.5 Mpa, and for 45 minutes.

Table 1. Formulation of W/SBR blends in wt%.

Materials	A0	A1	A2	A3	A4
SBR	100	100	100	100	100
W	0	25	50	100	150
ZnO*	3	3	3	3	3
Stearic acid*	2	2	2	2	2
TMTD*	3	3	3	3	3

*In phr (parts per hundred rubber) from the total of W/SBR blend; TMTD=Tetramethylthiuram disulfide.

2.2. Characterization

2.2.1. Fourier transform infrared spectroscopy. The chemical structure for blends was investigated using FTIR (Alpha-Bruker, Germany). The vulcanized sheets were ground into tiny pieces (powder). After that, the plate and anvil tip was cleaned and allowed to dry before the placement of the sample. Then, enough quantity from the sample (powder) was put directly on the plate of a diamond crystal, and a pressure arm was placed on the sample and pressed. The wavenumber variation was in the range of 500–4000 cm^{-1} [8].

2.2.2. Morphological analysis. The SEM (VEGA/TESCAN-XMU type) was used to investigate the tensile fracture surfaces of samples to know the morphology of vulcanized blends and the distribution of wool in blends. Before the morphological analysis, the cross-section of the specimens was covered with a thin gold layer to avoid any electrostatic charge before scanning.

2.2.3. Mechanical test. Mechanical properties of samples (tensile strength) were inspected by using the T10 Tensometer (Monsanto, England) at room temperature, consistent with the ASTM D412-98 standards, which included crosshead rate fixed at 500 mm/min for all tested samples and the average of four tests were used.

3. Results and discussion

3.1. FTIR test of wool (W), pure SBR, and SBR grafting W (W/SBR blends)

FTIR results of W and pure SBR are presented in Figure 3a. The observed characteristic peaks of W in the FTIR spectrum are described in the following paragraph. The spectra of infrared absorption for W refer to characteristic bands of absorption mainly dedicated to the peptide bond ($-\text{CONH}-$), which represents the primary structural unit at the polypeptide chain. The atoms in $-\text{CONH}-$ vibrate to form spectral bands determined as I, II, and III amides [9]. The peak at 1632 cm^{-1} is referred to as the amide I, mainly associated with the carbonyl group $\text{C}=\text{O}$ stretch vibrations. The absorption band at 1513 cm^{-1} is assigned to the amide II band that corresponds to N-H bending with minor contributions of C-N and C-C vibrations of stretching. The peak at 1231 cm^{-1} is referred to as amide III, which is a more complex vibrational mode due to the combination of N-H bending and C-N stretching, with some contribution from C=O bending and C-C stretching vibrations [10, 11].

The peak at 1447 cm^{-1} is related to the bending of C-H. The methylene $=\text{CH}_2$ stretching vibrations at the peak of 2929 cm^{-1} [12]. The peak at 1390 cm^{-1} is referred to as $-\text{CH}_3$ symmetrical deformation [10]. The peak at 1032 cm^{-1} is referred to as the symmetric and asymmetric S-O vibration of the cysteine sulfinic acid [13]. The FTIR spectra for pure SBR are presented in Figure 3a. Absorptions with 742 and 834 cm^{-1} were referring to the cis- $\text{CH}=\text{CH}$ and CH out of the plane of cis- $\text{C}(\text{CH}_3)=\text{CH}$ bending vibrations, respectively [14]. The peak at 1373 cm^{-1} is referred to as symmetrical CH_3 [14]. The characteristic absorption peaks of benzene units were found in the peak of 1444 cm^{-1} . The peak at 1651 cm^{-1} is referred to as the stretching vibration of $\text{C}=\text{C}$. The peaks at 2854 and 2918 cm^{-1} are assigned to the C-H stretching vibrations [15]. FTIR spectra for W/SBR (A1, A2, A3, and A4) blends were presented in Figure 3b. The spectrum shows the appearance of many absorption peaks related to the structure of W. For A1, the spectrum shows the absorption peaks at 1640 , 1520 , and 1242 cm^{-1} correspond to the amide I, amide II, and amide III groups, respectively.

The peaks of the amide group are shifted to 1640 , 1522 , and 1243 cm^{-1} , respectively, for A2 blends, and are shifted to 1642 , 1522 , and 1243 cm^{-1} , respectively, for A3 blends, and are shifted to 1643 , 1524 , and 1244 cm^{-1} , respectively, for A4 blends as compared with the A1 blend. The absorption peak at 1038 cm^{-1} for A1 represents the S-O vibration; this peak is shifted to 1043 , 1044 , and 1048 cm^{-1} for A2, A3, and A4 blends, respectively, as compared with the A1 blend. The absorption peak at 3278 cm^{-1} for A1 represents the hydroxyl groups ($-\text{OH}$); this peak is shifted to 3279 , 3282 , and 3290 cm^{-1} for A2, A3, and A4 blends, respectively. Moreover, the FTIR band of SBR in A1, A2, A3, and A4 blends is clearly shown the cis- $\text{CH}=\text{CH}$ at 745 cm^{-1} for the A1 blend; this peak is shifted to 749 , 751 , and 753 cm^{-1} for A2, A3, and A4 blends, respectively, as compared with the A1 blend.

The absorption peak of benzene units was found in 1442 cm^{-1} for the A1 blend; this peak is shifted to 1443 , 1445 , and 1448 cm^{-1} for A2, A3, and A4 blends, respectively, compared with the A1 blend. The peaks at 2844 and 2914 cm^{-1} refer to the stretching vibrations of C-H for A1; this peak is shifted to 2844 and 2914 cm^{-1} for A2, 2844 , and 2914 cm^{-1} for A3 and 2844 and 2914.84 cm^{-1} for A4 blends [16]. The shifting and increasing in the intensity of these peaks with increasing WF loading have confirmed that some of the WF were crosslinked with the SBR phase [17]. A broadening of the amide groups and other bands can be observed with increasing the W content, and this shows that some interaction occurred between the W fiber and the SBR matrix [18]. The studies of FTIR give strong proof that there is an interaction between the W fiber and SBR matrix in the blends. The peaks of change in the shape, intensity, and position show the complexity of the blends [19].

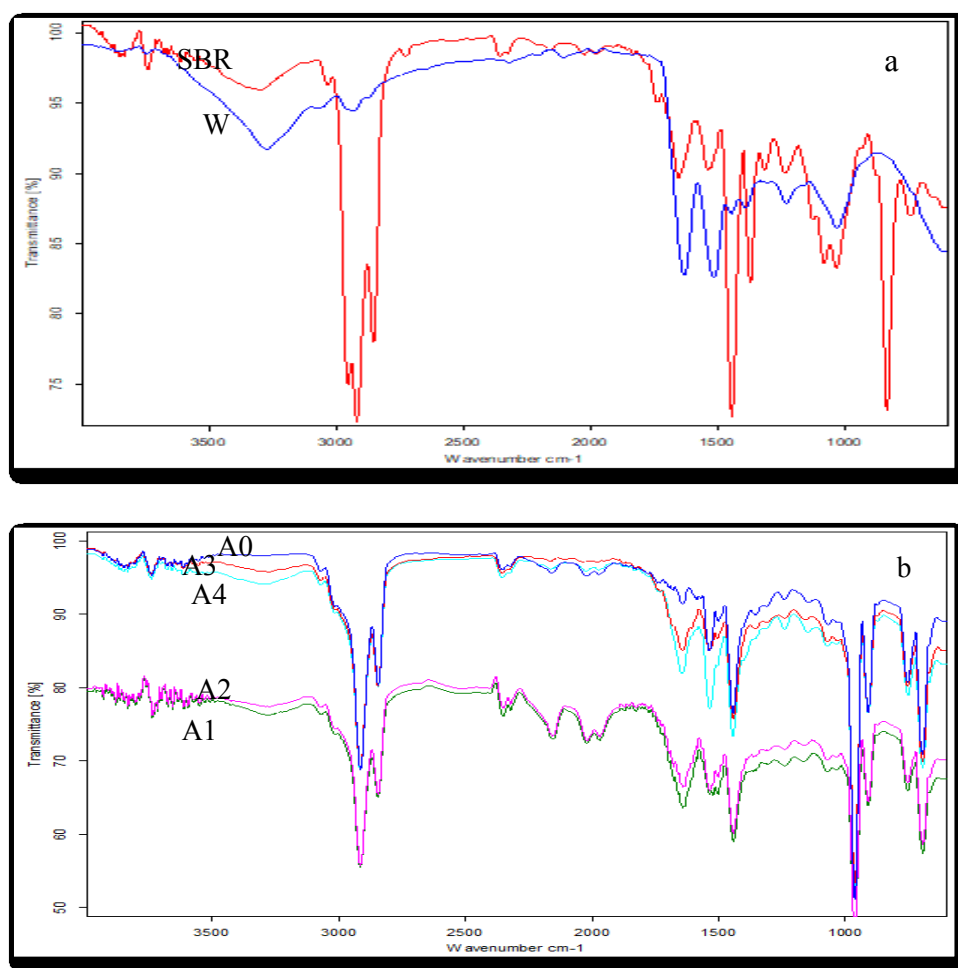


Figure 3. FTIR result of a) W and pure SBR b) W/SBR blends (A0, A1, A2, A3, and A4 blends).

3.2. Morphology

The SEM was used to characterize the morphology of the W/SBR blends. Figure 4 shows the SEM images for the fractured surface of A0, A1, A2, A3, and A4 blends, respectively, at a magnification of 200X. A non-uniform dispersion of the WF component on the surface of SBR was clearly observed, which indicates the heterogeneous morphology of the blends that may be due to the nature of the W is hydrophilic. In contrast, the nature of the SBR matrix is hydrophobic. Also, an apparent void around the SBR matrix was observed, which indicates that the adhesion between SBR and W in the blend was weak [20, 21]. Moreover, it can be clearly seen that the scales of wool fiber are sharp. Depending on these properties, it could be suggested that the W/SBR blends utilizing the TMTD catalyst can be utilized in the application of slow-release fertilizer.

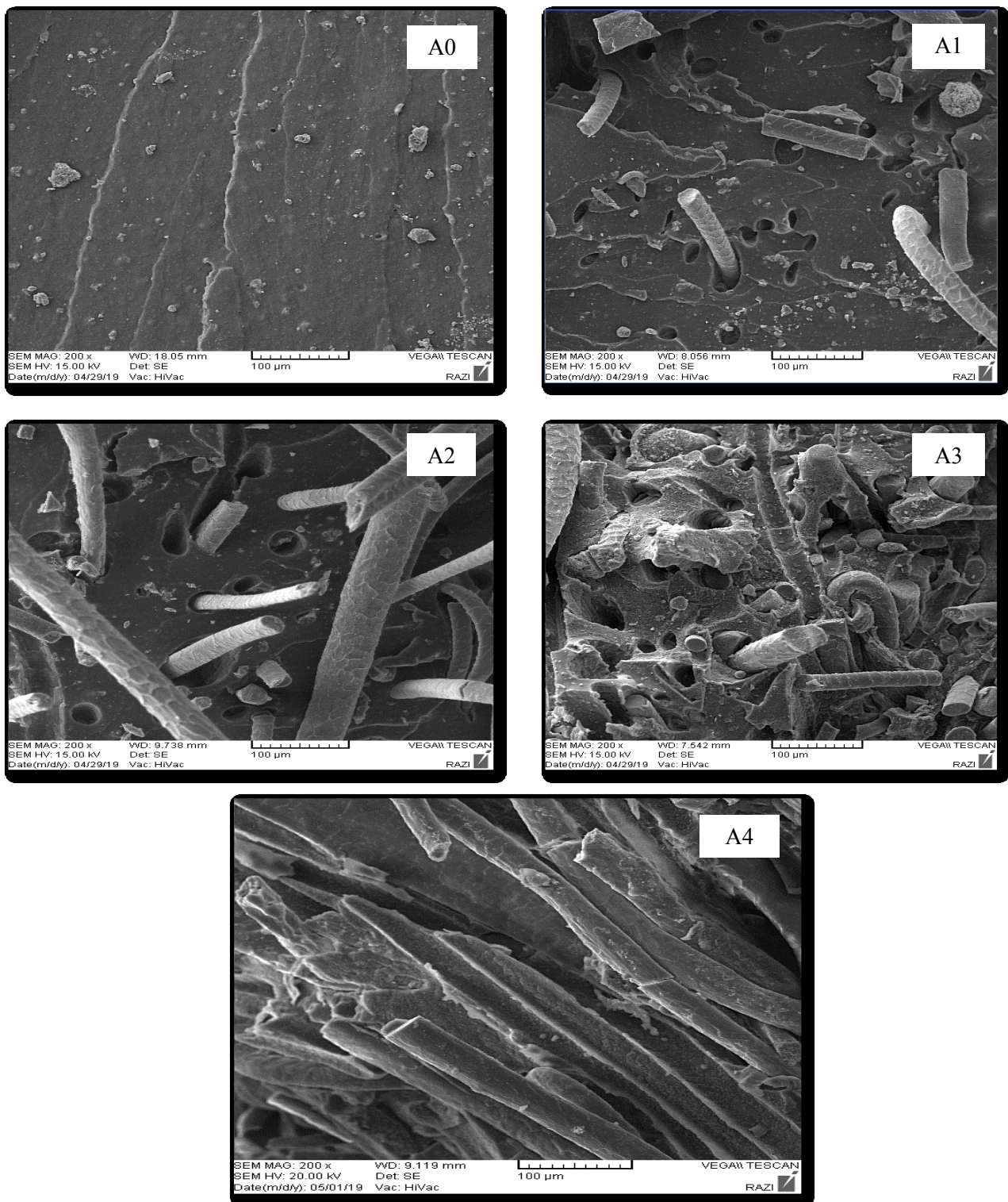


Figure 4. SEM of fracture tensile strength for A0, A1, A2, A3, and A4 blends.

3.3. Effect of W addition on the mechanical properties of SBR

Tensile strength and elongation at breaking were measured to estimate the effect of the incorporation of WF on the mechanical properties of the rubbers.

3.3.1. Tensile strength. The influence of WF load on the tensile strength of blends is shown in Figure 5, where the tensile strength is a function of WF content in a specimen, and it is increasing in the range from 25 to 150 phr of WF. The addition of WF to the rubber gradually decreases the tensile strength, and the maximum values were reached to 25 phr where they were about 1.997 Mpa. Further increase in WF loading leads to a slight decrease in the tensile strength. The increase in tensile strength with a low concentration (25 phr) is because of improving the interfacial bonding among wool fiber and the rubber matrix. Small quantities of WF can be well distributed, as shown in the SEM result, thereby increasing the tensile strength. Moreover, the interaction between the wool fiber and matrix plays a very significant role in increasing the tensile strength [22]. On the other hand, the increases of WF loading to be above 25 phr fiber content leads to a decrease in tensile strength. The reason may be due to WF is randomly blended with the rubber, and the irregular shape may lead to a decrease in the tensile strength of blends because of the inability of WF to support stress transfer from a polymer matrix [23]. The results of SEM show that the WF was agglomerated at high percentage loading, leading to a reduction in the bonding strength between the fiber and rubber chains. Another reason maybe because of the impurity in wool acts to separate rubber chains and thus weaken the cohesion between the rubber chains. This is coupled with agglomeration, lead to a rapid decline in tensile strength [24]. These observations are mainly attributed to the incompatibility of the blends where SBR was hydrophobic, and the WF was hydrophilic. The addition of WF in the SBR matrix makes voids in the SBR matrix. More wool fiber means increasing the number of voids in the SBR matrices, so act as a barrier to inhibit the formation of the chemical and physical crosslink between chains of rubber; thus, segregate the chains of SBR, which could explain the reduction in strength with increase WF load [25], as shown in SEM results in Figure 4.

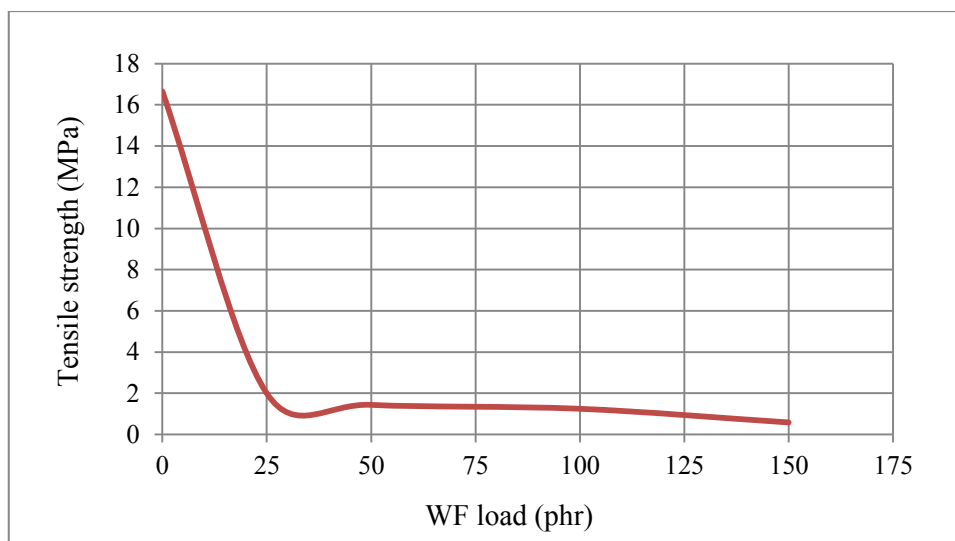


Figure 5. Tensile strength (Mpa) versus W content (phr) of W/SBR blends.

3.3.2. Elongation at break. The incorporation of WF to an SBR matrix decreases the elongation at break, as shown in Figure 6. It was noted that 25 phr of WF blend has a higher value of elongation at the break, which was higher by 40% than the other blends. During the application of the tensile stress on a sample, the region of stress concentration is appeared, leading to produce cracks in the sample [26]. With increasing WF content, the elongation at break decreases gradually, and this is related to a gradual increase in brittleness and stiffness of blends. In other words, the ductility of blends decreases

as the rigidity improved; thus, the blends break at lower elongation [27]. When the wool content increases, the interfacial regions between the wool and the matrix were formed, and it is increasing with the increase in the wool content, as shown in SEM results. As it is easier to travel through the cracks in these vulnerable regions, the fracture of blends will be a lesser degree of elongation, together with the increase of the WF [27]. Numerous parameters may affect the elongation at the break, such as the dispersion of wool fiber in a matrix and the adhesion between the WF and a matrix [28]. The wool fiber showed weak dispersion and adhesion in the SBR matrix due to the ability of wool to accumulate and form voids in the SBR matrix, as shown in the SEM results. Thus, this makes a regional stress concentration that leads to a decrease in the elongation at break [29]. It can be suggested that increasing the content of WF leads to a decrease in an elongation of blends as compared to the control sample (A0).

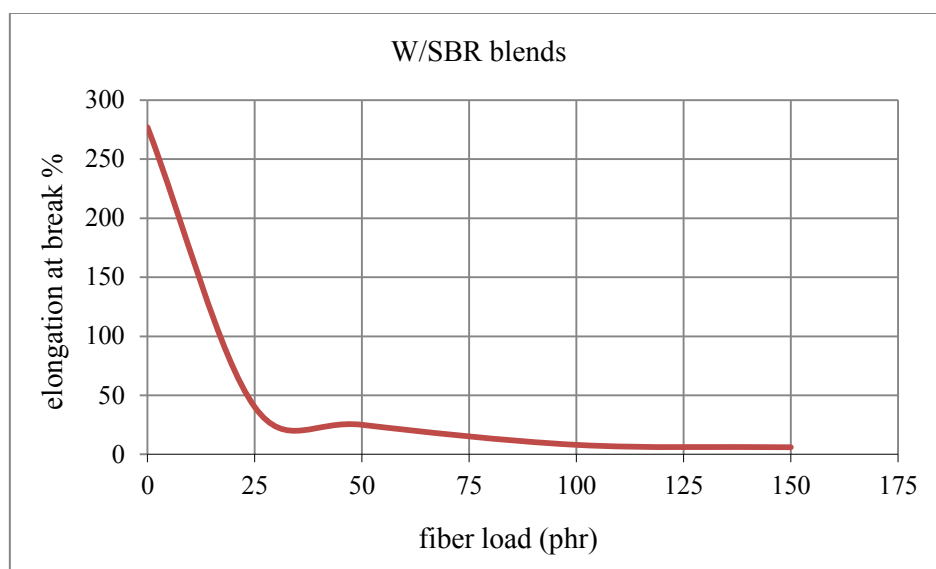


Figure 6. Elongation at break versus W content (phr) of W/SBR blends.

4. Conclusions

Styrene-butadiene rubber (SBR) was blended with the wool fiber (WF) at the loading range of 0-150 phr using tetramethyl thiuram disulfide (TMTD) as a catalyst. The results of FTIR showed a WF crosslinked with the SBR phase. The SEM results revealed the non-uniform dispersion of WF in the SBR matrix. An increase in the WF loading leads to decreased tensile strength and elongation at the break. Furthermore, a maximum tensile strength was observed at 25 phr of WF loading. The results of this study were believed to help in developing new ways to recycle keratin waste into beneficial products, and thus will reduce environmental pollution.

5. References

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