

# Studying the Effect of Silica on Some Physico-Mechanical Properties and Morphological Behavior of Bagasse Filled Styrene Butadiene Rubber

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**Abstract:** Currently, the incorporation of ground cellulosic bagasse as a supplement reinforcing filler in rubber composites is considered to be one of the favorite means that could potentially assist in avoiding the utmost problematic environmental pollution due to the ashes left after burning of agricultural wastes. In addition, it may act in reducing the cost simultaneously with preserving the rubber vulcanizates physico-mechanical properties. In this study, a systematic work was implemented by preparing a series of mixes based on fixed concentration of 25 phr from natural ground bagasse powder (GBP) on a two roll mill to investigate the effect of different quantities (0 – 80 phr) of fumed silica (Si) on the cure characteristics, physical and mechanical properties of Bagasse-Styrene Butadiene Rubber (B-SBR) composites. A grinding machine was used, to obtain GBP with a selective grain size distribution ranging from about 20-180  $\mu\text{m}$ . As well, the interfacial adhesion between SBR and bagasse was improved by adding 2.5 phr of maleic anhydride (MA), as a compatibilizer. It was obvious that values for both tensile strength and modulus at 100% elongation have recorded a significant increase with the increase in Si addition till reaching an optimum concentration of 60 phr, and then a gradual decrease in values takes place with further Si addition. Similarly, hardness recorded a gradual increase in values with the increase in Si content. On the other side the abrasion loss and degree of swelling values are enhanced by showing a marked decrease in values with the increase in silica content. It was evident that modified SBR brings well physico-mechanical properties and wear resistance. Thereby, it's worth derived that the overall performance of Si filled SBR composites is well preserved in the presence of bagasse powder. The prepared modified SBR composite samples were also characterized by scanning electron microscope (SEM), which reveals a behavior confirming the improved mechanical properties.

**Keywords:** Bagasse, Mechanical Properties, Maleic Anhydride, Silica, Styrene Butadiene Rubber

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## 1. Introduction

Lately, biodegradable friendly, natural products have gained the attention of worldwide scientific researchers as people become more aware of environmental conservation. The superb properties like biodegradability, renewability, recyclability, low production cost and absence of associated health hazards in addition to their well competition in terms of strength to weight of material were the utmost motivation for the employment of numerous biomass residues (i.e., rice husks, maize stalks and sugarcane bagasse) generated from agro-businesses as reinforcing fillers in various polymeric materials [1].

In recent times, few studies were reported using natural

fibres from bagasse as a source of reinforcement filler for both natural and synthetic rubbers. Accordingly, by investigating the utilization of bagasse in powder form instead of ash form, an interesting idea arose to examine its role as a complementary reinforcing filler for silica-filled SBR vulcanizates. Sugarcane bagasse is composed mainly of cellulose (41.0-55.0 wt%), hemicellulose (20.027.5 wt%), lignin (18.0-26.3 wt%) and others (~ 7.0 wt%) attributed to inorganic materials [2]. Abdelwahab and Helaly [3] investigated the effect of adding various concentrations of modified bagasse to SBR formulations with carbon black and silica (hisil) as commercial fillers already used in industry. They observed that the better retained values of the physico-mechanical properties and equilibrium swelling were

obtained for the rubber vulcanisates containing 50 phr modified bagasse in comparison with those containing carbon black, hisil, and untreated bagasse. Khalaf E S A et al [4] investigated the effect of different concentrations (i.e. 10-50 phr) of modified bagasse powder incorporated into carbon black reinforced SBR formulations. They reported that an improved mechanical properties and equilibrium swelling values for SBR vulcanisates were recorded at an optimum concentration of 30 phr from modified bagasse powder.

Reinforcing fillers are added to rubber compounds to enhance their physical properties. Nowadays, silica is proposed to be the most frequently used reinforcing white filler for rubbers. Natural and synthetic silica are two available varieties that can be used in different industrial applications. Precipitated silica and fumed silica are the two synthetic silicas currently used in elastomer technology, depending on their method of manufacture [5]. A tire must have many necessities, especially reduced rolling resistance, improved abrasion resistance, heat build-up of rubber tires during driving, and improved wet traction [6-8]. Currently, synthetic rubbers are typically used in pneumatic tires. According to the survey and statistics during the driving process of the car, the rolling resistance of the tire accounts for 20-30% of the total energy consumption of the car. Furthermore, the rolling loss of tread is about 50% of the total energy consumption of tire [9]. Owing to its distinctive characteristics and in order to meet the urgent demands in an extensive range of applications in rubber and particularly in the tire industry, silica has offered a multiple of applications including such products as heavy service tire treads, passenger tire treads, shoe soles, engine mounts, wire coats, cables, and any application where a colored or transparent rubber is desired. Furthermore, due to its outstanding properties mentioned earlier, silica is considered prominent filler for reinforcing rubber composites required for the production of high-performance passenger tires [6, 8].

Creation of strong filler-filler interactions and adsorption of polar materials by hydrogen bonds were generated, due to the presence of strong intermolecular hydrogen bonds between many hydroxyl groups on the silica surface. This phenomena can cause a poor dispersion of silica in a rubber compound, and consequently they can be tightly aggregated [10, 11]. Many researches were reported earlier about the modification of rubber in the rubber molecular chains such as natural rubber (NR) grafted by maleic anhydride (MAH) [12].

In the present work, the influence of different concentrations of silica on the properties of bagasse-styrene-butadiene rubber (B-SBR) compounds was investigated. The morphological behaviors of the silica-filled compounds were also characterized by using a SEM, in order to provide a full agreement with the derived mechanical properties.

## 2. Experimental

### 2.1. Materials

The synthetic rubber used in this study was SBR (23.5% styrene content, ML (1+4) of 52±3 at 100°C), delivered from Bayer AG (Germany). The employed maleic anhydride

(MA) was obtained from Merck KGaA Ltd., Germany. Hydrophobic fumed silica AEROSIL® R 972 (SiO<sub>2</sub>) was supplied by Frankfurt am Main (Germany), specific surface area of silica was 110 ± 20 m<sup>2</sup>/g. The activator system comprised zinc oxide (ZnO, purity 93-96%) and stearic acid (SA, purity 98%). The phenolic antioxidant namely, 2, 2'-Methylenebis (4-methyl-6-tert-butylphenol) (C<sub>23</sub>H<sub>32</sub>O<sub>2</sub>) was brought from Taiwan. The processing lubricant oil used is Mobil 1 and the used solvent is toluene as a commercial grade. Whereas, the curing system consisted of commercial products of sulfur (S, minimum purity 99.5%) that was used as received and dibenzothiazyl disulphide (MBTS), as vulcanization accelerator was provided by Farbenfabriken Bayer AG kusen lever (Germany). Bagasse used in this study was obtained from local sugarcane mills after processing to extract sugar and liquor. Firstly, it was dried at 80°C for 48h, then ground and sieved into different particle sizes. The drying step was repeated before its incorporation into silica filled SBR rubber mixes [13].

### 2.2. Experimental Techniques

#### 2.2.1. Bagasse Powder Processing

The local milled bagasse fibres were subjected to surface modification, through performing an alkali treatment (5%NaOH) for 6 hours at 60°C followed by neutralization with Hcl solution. Then the fibre bundles were thoroughly rinsed with water and dried at 80°C for about 24 h in an oven. A deluxe-standard Retsch SK 100 laboratory mill operating with rotor speed of 2850 rpm at 50 Hz, was used to grind the chemically treated and dried bagasse to obtain a very fine bagasse powder. The ground bagasse was dried as aforementioned [13], before its incorporation into the rubber mixes. Grain size distribution of the ground bagasse powder was examined using mechanical vibratory sieve shakers with certified standard US. Mesh, which reveals that the received powder grain size distribution is ranging from 20 µm to 180 µm and that 98.06% of the particles passed the 180 µm sieve. Bagasse fibres (before grinding) and bagasse powder (after grinding) are clearly seen in (Figures 1(a) and 1(b)).



(a) Before grinding



(b) After grinding

**Figure 1.** Bagasse before grinding (a) and after grinding (b).

### 2.2.2. Rubber Formulations and Compounding

The rubber was firstly masticated, by passing it twice through a laboratory two-roll mill of outside diameter 470 mm and working distance 300 mm, speed of the slow roll is 24 rpm and friction gear ratio of 1.4:1 for one and a half-minute. The masticated rubber will indeed facilitate the vulcanization process, by making the rubber softer and enabling for an ease mixing and addition of all ingredients. The order of constituents addition into the masticated SBR starts with maleic anhydride, followed by bagasse and the band formed around the roll mill takes about three and a half-minute. Then the rest of accurately weighed additives including activators, reinforcing silicon dioxide, processing oil, accelerator, antioxidant and finally the sulphur vulcanizing agent were added in a normal sequence, with mixing stage of about 8-10 minutes and fixing the rolls temperature at about 60°C according to ASTM D-3182. The overall mixing time was about 13-15 minutes and a three to four cut was made every half-minute alternatively from each side, in order to obtain homogenous sheeted rubber mixes with thickness of about 2 mm. The sheets obtained from compounded SBR mixes were left for at least 6 hrs, before being compression moulded (i.e vulcanized) at  $152 \pm 1^\circ\text{C}$  under pressure of 10 ton in an electrically heated hydraulic press at an optimum curing time derived from the reograph. Finally, the moulded rubber sheets were left for at least 6 hrs before the tensile testing, according to ASTM D-412.

The group of mixes employing different concentrations of Si ranging from 0 to 80 phr at fixed concentration of bagasse; 25 phr used in this work, were shown in Table 1. The size of GBP in all gum formulations were ranging from 20 to 180  $\mu\text{m}$ .

**Table 1.** Formulations of B-SBR composites with different concentrations of Si [phr].

Formulations code	B25	Si20	Si40	Si60	Si80
SBR	100	100	100	100	100
MA*	2.5	2.5	2.5	2.5	2.5
GBP	25	25	25	25	25
ZnO	5	5	5	5	5
Stearic acid	2	2	2	2	2
Silicon dioxide (AEROSIL® R 972)	0	20	40	60	80
Processing oil	10	10	10	10	10
MBTS**	1.5	1.5	1.5	1.5	1.5
A. O (Cyanox)***	1	1	1	1	1
S	2.5	2.5	2.5	2.5	2.5

\*Maleic anhydride \*\* Mercapto benzthiazyl disulphide

\*\*\*2, 2'-Methylenebis (4-methyl-6-tert-butylphenol).

### 2.2.3. Rheology Measurements

The Oscillating Disc Rheometer (ODR) model 4308 from Zwick, Germany was used to measure the cure characteristics of rubber mixes, according to ASTM D-2084.

### 2.2.4. Measurement of Mechanical Properties (ASTM D-412)

Zwick Tensile Testing machine (1445), Germany, was used

in measuring the mechanical properties such as tensile strength, elongation at break and modulus M 100, at a cross-head speed of 500 mm/min. A set of five dumbbell-shape specimens with the standard width of 4 mm and length of 115 mm, were cut from the vulcanized sheets along the mill grain direction to be used in tensile testing. The thickness of the test specimens was determined by a graduated gauge to one hundred of mm. Shore 'A' hardness tester of the type HGIB, produced by the VEB Thuringer was used to measure the hardness of the samples, according to ASTM D-2240. The abrasion resistance for rubbers and elastomers are determined according to standard test method DIN 53516. The instrument used in finding the abrasion wear is —Din abrader.

### 2.2.5. Measurement of Equilibrium Swelling

The swelling process was performed by immersing the specimens in toluene at room temperature  $25 \pm 2^\circ\text{C}$ , for 24 hours. The equilibrium swelling was carried out by calculating the variation of apparent percentage of change in mass expressed as the increase percentage of the original mass, according to D471-97 (1998). The swelling percent was calculated according to equation 1:

$$\text{Swelling percent} = [(d-w)/w] \times 100 \quad (1)$$

Where,

w: the original weight of the sample;

d: the final weight of the swelled sample.

### 2.2.6. Scanning Electron Microscopy (SEM)

An FEITM Inspect model high-resolution imaging, with advanced chamber vacuum technology, England was used to carry out the Scanning Electron Microscopy (SEM) analysis. The samples were mounted on a standard specimen stub, subjected to high resolution Schottky field emission. An operating accelerating voltage of 200 V – 30 kV, Probe current:  $\leq 200$  nA, continuously adjustable and with magnification: 14 to 1000000 x.

## 3. Results and Discussion

### 3.1. Rheological Properties

Table 2 shows the rheological characteristics of styrene-butadiene rubber mixes that were measured at  $152 \pm 1^\circ\text{C}$ , for a period of 30 minutes. From the rheological data it can be seen that, as the concentration of  $\text{SiO}_2$  is increased from 0 to 80 phr the scorch time and optimum cure time were increased. The rate of curing is also dependent on the  $\text{SiO}_2$  content of the different mixes. Thus, an increase of  $\text{SiO}_2$  content will result in a slight decrease in the rate of curing.

The increase in the scorch time and optimum cure time and the decrease in the rate of curing indicate that, the vulcanizing accelerators could be adsorbed on the surface of  $\text{SiO}_2$ , consequently, the effective concentration is lower as compared with the unfilled SBR.

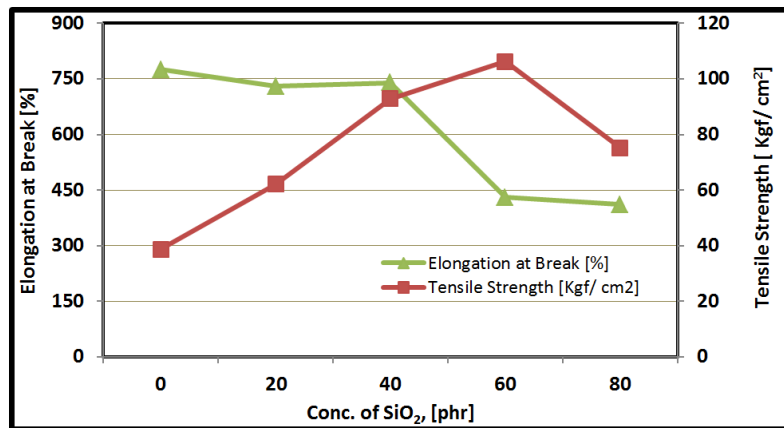
**Table 2.** Overall properties of B-SBR composites with different concentrations of Si.

MIXTURE CODE			B25	Si20	Si40	Si60	Si80
Rheometric Properties	T <sub>S2</sub>	[min]	1.1	1.1	1.6	1.8	3.6
	T <sub>C90</sub>		19.29	17.4	19	19.6	21.5
	R	[1/min]	0.22	0.34	0.27	0.32	0.22
Physico-mechanical Properties	Tensile Strength	[Kgf/cm <sup>2</sup> ]	38.75	62.1	92.93	106.3	75.20
	Modulus 100%		10.2	16.83	17.34	32.12	29.06
	Elongation	%	775	730	740	430	411
	Hardness	'A'	49.3	45.1	55.8	61.2	72.6
	Abrasion	%	-	45.3	32.7	24.4	22.1
Swelling Properties in:	Toluene	%	235.04	232.35	195.5	142.8	92.52

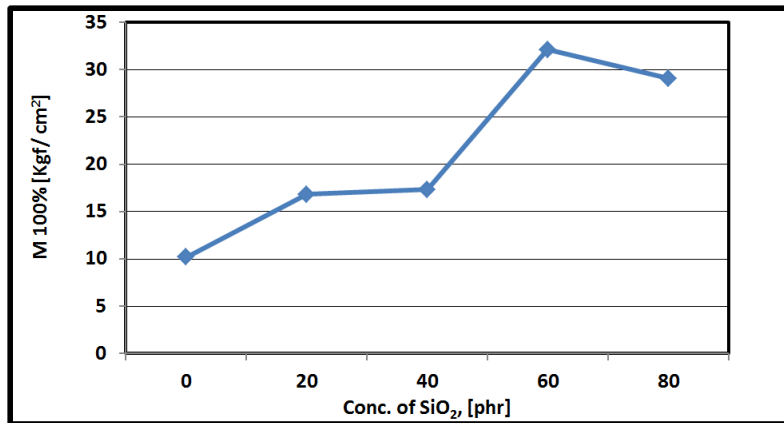
t<sub>C90</sub>= Optimum cure time at 90% of full torque development, t<sub>S2</sub>= Scorch time, CRI= Cure Rate Index=100/ (t<sub>C90</sub> - t<sub>S2</sub>).

### 3.2. Physico-Mechanical Properties

#### 3.2.1. Effect of SiO<sub>2</sub> Concentration on the Tensile Properties of B-SBR Composites



**Figure 2.** Effect of different SiO<sub>2</sub> concentrations on tensile properties of B-SBR vulcanizates.



**Figure 3.** Effect of different SiO<sub>2</sub> concentrations on modulus of B-SBR vulcanizates.

The tensile strength of B-SBR composites having different silica loadings are illustrated in figure 2. The figure shows that the tensile strength values increase with increasing content (20, 40 and 60 phr) of silica. The increase in tensile strength values is suggesting that there is a significant increase in the reinforcing level with the increase in silica content, until reaching an optimum silica loading of 60 phr. Whereas, further increase in silica content causes an obvious decrease in tensile strength values of the prepared composites because of the poor silica dispersion in the matrix and also due to the poor interaction of silica /SBR caused by the hydrophobic nature of

fumed silica particles, which resulted in silica aggregation at higher loadings and led to high filler-filler interactions as stated earlier by Agarwal [14] et al. The increase in filler loading has similar effect on modulus (M 100). As shown in figure 3, an increase in Si loading increases the modulus up to loadings of 60 phr for silica-filled vulcanizates, beyond which the modulus values tend to slightly decrease. This observation is indicating stronger filler elastomer interactions for silica filled vulcanizates. Tangudom [15] et al, had also reported in their study that the tensile modulus increased when the silica content increased. While the elongation at break show an

opposite trend for the composites by the obviously observed lower values, as seen in figure 2. This result is correlated well with the decreasing trend in swelling ratio values of the mixed composites, as obviously will be seen later in figure 7. It is reported elsewhere that reinforcing silica increases the elongation at break up to an optimum filler loading of elastomer, after which properties deteriorated due to agglomeration of silica [16]. Ismail [17] et al.; Arayaprane [18] et al., found that the elongation at break tended to steeply decline with increased silica content, although there were small peaks for the 40 phr silica-filled vulcanizates suggesting a good interfacial adhesion of the filler-rubber and proper wetting of the silica. In addition, a reduction in the elongation at break at the higher silica content indicates the restriction of movement in polymeric molecular chains due to the greater filler-filler interactions.

These tensile properties indicate that silica is capable of reinforcing the Bagasse-SBR composites. This is confirmed by the scanning electron micrographs of the fracture surfaces of silica-filled vulcanizates, which will be discussed in the next

section. Early optimum loading of silica-filled vulcanizates may be due to either formation of filler agglomeration at lower silica loading or other filler-related parameters, such as larger particle size and poor filler-matrix interaction.

### 3.2.2. Effect of $\text{SiO}_2$ Concentration on Hardness

The hardness (shore A) of B-SBR composites having different silica loadings are illustrated in figure 4. The increase in the silica content led to an increase in the hardness values. The better silica dispersion may lead to an increase in the composite's crosslink density, and as a consequence the softer matrix turns to a harder one. The enhanced hardness is suggested to be due to the higher crosslinking density. The incorporation of silica in the soft matrix reduces the elasticity of the SBR chains and as a consequence increases the hardness values, as an indication for a higher reinforcement [19]. Jacques [20] stated that an increase in the hardness accompanied with a dramatically reduction in the resilience will be generated by the addition of most of the particulate fillers into rubber, particularly with the more reinforcing fillers.

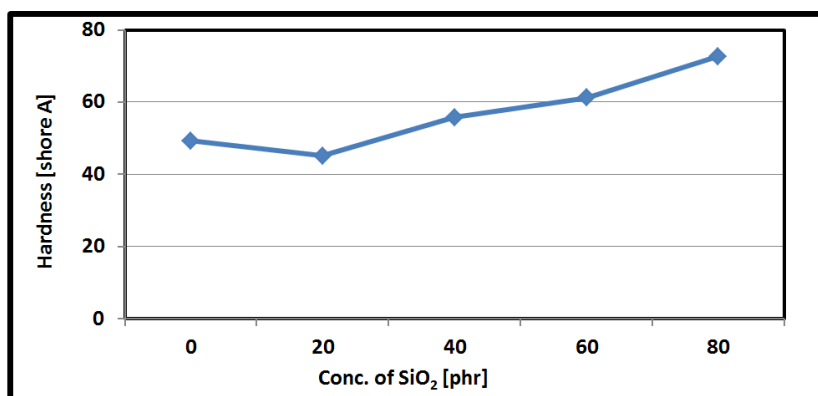


Figure 4. Variation of hardness for different  $\text{SiO}_2$  contents in B-SBR vulcanizates.

### 3.2.3. Effect of $\text{SiO}_2$ Concentration on the Abrasion Loss

Abrasion resistance was quite crucial when considering the operational life of the rubber material and was analyzed as weight loss during the abrasion test. Figure 5 shows variation of the abrasion loss with the filler content. The rise in silica concentration, accompanied with its good dispersion in the SBR matrix will reduce the filler-filler interaction, which in

turns leads to higher abrasion resistance. As a better reinforcement material, silica has a stronger interfacial relationship with the SBR matrix and the reduction of abrasion loss in the SBR composites was clearly evident [21]. All in all, the wear rate decreases with the increase in filler content indicating that the silica filler in the presence of fixed bagasse concentration show good resistance to wear.

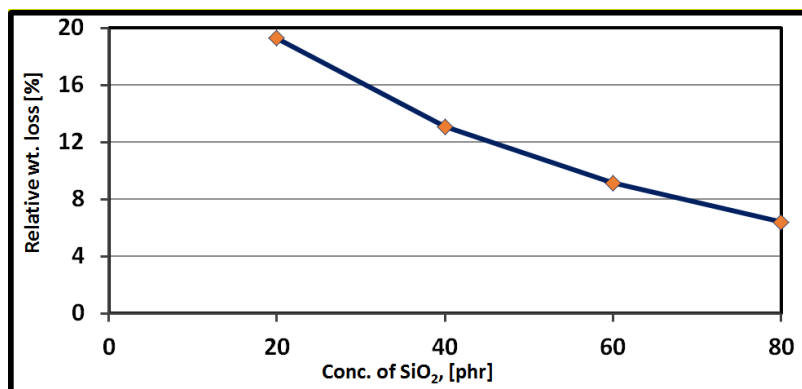


Figure 5. Variation of weight loss for different  $\text{SiO}_2$  contents in B-SBR vulcanizates.



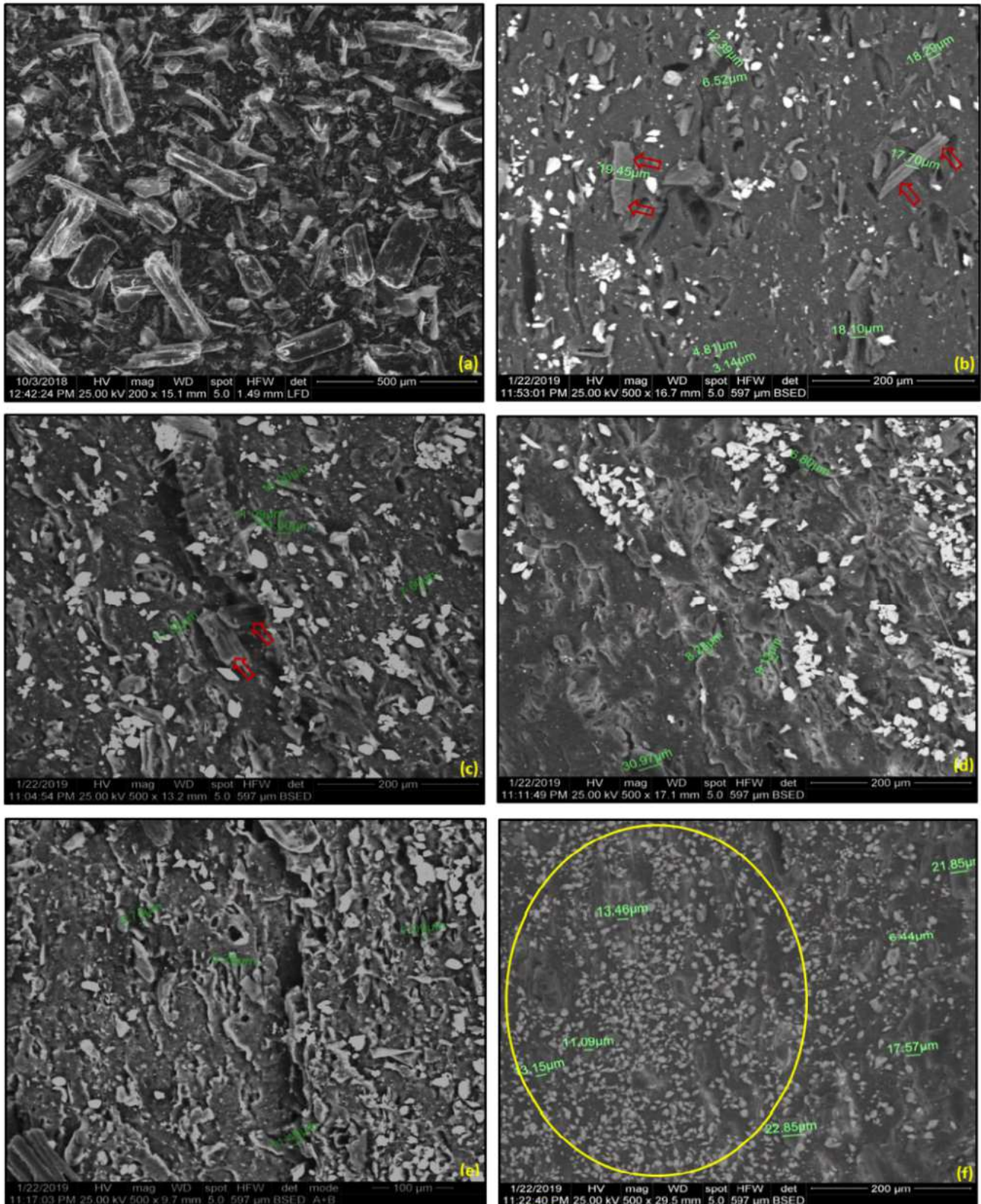


Figure 6. SEM micrographs of GBP (a), B-SBR unfilled composite (b), B-SBR vulcanizate loaded with 20 phr (c), 40 phr (d), 60 phr (e) and 80 phr (f)  $\text{SiO}_2$ .

### 3.3. SEM of Microstructure Fractured Surfaces

Figures (6a-6b) show the SEM micrographs of GBP and B-SBR composites prepared by direct mixing. The well

viewed bagasse fibres represented by the red block arrows were found to be somewhat homogenously dispersed throughout the unfilled SBR matrix, as seen in figure 6b. Figures (6c -6e) show that fracture surface of the 20, 40 and

60 phr Si/ B-SBR composite prepared by direct mixing is smoother than that of 80 phr Si/ B-SBR composite where several agglomerates can be seen (Figure 6f). The reinforcing silica was found to be well impregnated inside the SBR matrix, as clearly seen by the filler-rubber interaction in figures (6c, 6d and 6e), whereas a wide portion of agglomerates shown in figure (6f) reveals that the filler-filler interactions were found to be the predominant rather than the filler-rubber ones. Consequently, silica particles achieve better wetting and dispersion, in B-SBR matrix resulting in better mechanical properties. The improvement in mechanical properties of the prepared composites was mainly attributed to an effective load transfer, based on the homogeneous dispersion of fumed silica particles in the SBR matrix. On the other side, the aggregated silica fillers develop a stress concentration point which results in drop down of properties in the composite materials [22]. The SEM results were found to be in complete agreements with results discussed earlier in section of tensile strength properties.

### 3.4. Equilibrium Swelling Properties

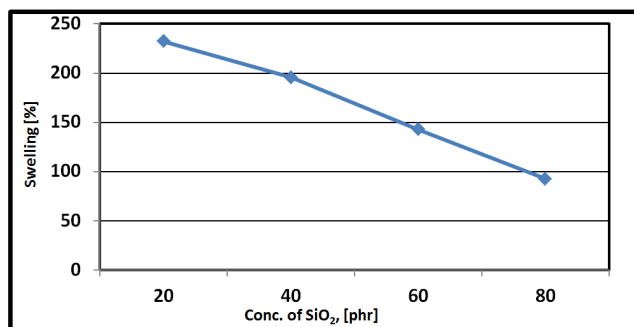


Figure 7. Variation of swelling behavior for different SiO<sub>2</sub> contents in B-SBR vulcanizates.

Rubbers are mainly used in manufacturing applications and are exposed throughout their lifespan to a range of chemical environments, thereby it should be a necessity to understand the interfacial interaction of the system through performing an analysis for the swelling characteristics of the composites. The effect of fumed silica loading and the existence of toluene as an aromatic penetrant were studied via B-SBR composites weight percent absorption. Table 2 shows the swelling properties in terms of weight percent uptake at different silica loading in B-SBR composites for toluene penetrant. The swelling ratio (i.e. weight-percentage absorption of toluene by fumed silica loading in B/SBR composites are clearly shown in Figure 7. As can be seen the equilibrium swelling of these composites decreases with increasing silica content. It is proved that the swelling resistance of the vulcanizates tends to be improved with increasing silica loading. This could be explained as a significance of the current pressure involved between the rubber polymeric chain network and the penetrant that acts to shrink the rubber chain network. In fact, the crosslinking density in rubber composites was dramatically increased with an increase in silica concentration, the matter that causes an increase in network elasticity. In addition,

crosslinks restrict the swelling induced extensibility of the rubber polymeric chains (i.e., macromolecular chains) and make it more difficult for solvent (penetrant) to disperse in the gaps between rubber content molecules and reduce the swelling percentage [23-25], thus combating the dissolution tendency [26]. As a result, the swelling decreases with network augmentation. Finally, lower composite swelling suggests that it was practically preferred to be used in industrial applications.

## 4. Conclusions

The performance of silica filled Bagasse-SBR vulcanizates has been investigated. Scorch time and optimum cure time were increased, whereas the cure rate of the silica-filled compound was decreased as the silica content increases. As expected, incorporation of silica increases the tensile strength and M100 up to the optimum filler loading of 60 phr. The elongation at break point also increases up to 40 phr filler loading and then undergoes an abrupt decrease on further loading. Results also indicate that the hardness undergoes a significant increase with filler loading, indicating its overall reinforcing ability. The abrasion is also improved with the addition of silica. As well, swelling shows significant decrease and SEM studies were found to be confirming for the tensile properties. Overall, the derived findings gave the currently used B-SBR system filled with the specified silica an advantage to be valorized in many industrial applications, and also it opens a wide gate for further deep investigations to appropriately address the compatibility of this system of fillers with different kinds of rubber.

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