

Physicomechanical Properties of α -Cellulose-Filled Styrene-Butadiene Rubber Composites

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ABSTRACT: In this research, the influence of adding α -cellulose powder to styrene-butadiene rubber (SBR) compounds was investigated. Physicomechanical properties of SBR- α -cellulose composites, including tensile strength, elongation, Young's modulus, tear strength, hardness, abrasion, resilience, and compression set, before and after ageing, were determined and analyzed. Young's modulus, hardness, and compression set increased and elongation and resilience decreased with increasing α -cellulose loading in the composites, whereas tensile strength, tear strength, and abrasion resistance initially increased at low α -cellulose concentration (5 phr), after which these properties decreased with increasing α -cellulose content. Lower loadings of α -cellulose (5 phr) showed better results than higher loadings,

given that tensile strength, tear strength, and abrasion resistance increased at low α -cellulose concentration. Theoretical prediction of elastic modulus was carried out using rule of mixtures, Hashin, Kerner, and Halpin-Tsai equations. Calculated results show that these equations are not suitable for accurate prediction for the work carried out. However, these models can be used with confidence for the prediction of elastic modulus because experimental results are higher than the calculated values. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 96: 2203–2211, 2005

Key words: elastomers; additives; mechanical properties; composites; rule of mixtures

INTRODUCTION

Cellulose, an abundant natural polymer, is the major cell-wall material in land plants and their main structural component. Native cellulose is found in both hard and soft woods (~ 50% cellulose) and cotton (~ 95%), which are the major sources for conversion to a wide variety of useful products including fibers, textiles, paper, and various derivatives. These latter form useful plastics, fibers, films, emulsifiers, and thickening agents.¹

The idea of using cellulose fibers as reinforcement in composite materials is not a new or recent one. In the past, composites, such as coconut fiber/natural rubber latex, were extensively used by the automotive industry. However, during the 1970s and 1980s, cellulose fibers were gradually substituted by newly developed synthetic fibers because of better performance. Since then, the use of cellulose fibers has been limited to the production of rope, string, clothing, carpets, and other decorative products. Over the past few years, there has been a renewed interest in using these fibers as reinforcement materials in

the plastics industry. This resurgence of interest has arisen because of the increasing cost of plastics, and also because of the environmental aspects of using renewable and biodegradable materials.²

In the rubber industry, treated cellulose fiber has been widely commercialized as a short-fiber reinforcement for rubber compounds over the past 25 years. This product is based on wood cellulose that has been specially treated to disperse easily in rubber compounds during mixing and bond to the matrix rubber during vulcanization. During processing of the composite rubber stock before vulcanization, the fibers become aligned by the flow. Control can be exercised over this orientation process to optimize the reinforcement potential for stiffening and strengthening the rubber stock in both its green and cured states to meet particular application requirements. These materials are of interest beyond business considerations as a benchmark for defining the breadth of short-fiber composite applications in the rubber industry. They demonstrate the potential of low-modulus, low-cost fibers as ideal reinforcements for the soft matrices typical of most rubber formulations. The main application areas for short-fiber composites are in hoses, belting, solid tires, and pneumatic tire components. Miscellaneous application areas include rubber roofing, dock fenders, seals, and diaphragms.³

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In addition to cellulose fibers, which are used in a wide variety of polymers, α -cellulose fibers have also been used to reinforce thermosets, especially melamines.^{4,5} A search of the literature showed no publication on cellulose/ α -cellulose powder to use as a filler, especially in elastomers. Therefore, in the present study, the grade of cellulose powder having the highest content of α -cellulose of all the reported grades of cellulose powder was selected to study its performance as a filler for rubber compounds. The effects of filler loading on both curing characteristics and physicomechanical properties of rubber composites are discussed.

THEORETICAL

The field of composite material behavior can be studied from two perspectives: micromechanics and macromechanics. The goal of most micromechanics approaches is to determine the elastic moduli or stiffness of a composite material in terms of the elastic properties of the constituent materials. Most of the analytical models presented⁶⁻⁸ presume the idealization that there is perfect adhesion between the phases and that the particles are spherical and evenly dispersed. Some of the earlier attempts in modeling composites were performed by Einstein and Guth.⁶ Guth and Smallwood extended Einstein's theory to explain rubber reinforcement.⁶ Both of these attempts have proved to be applicable, but only at low concentrations of particulate. Thus, the focus will be on the newer works, separated into two approaches, defined as either a mechanics of materials or an elasticity approach. In the mechanics of materials approach some simplifying assumptions are made, the most significant of which is that the strain in the matrix is equal to the strain in the particulate. With this assumption the most simplistic of all methods of predicting the moduli of a composite, known as the rule of mixtures, can be obtained:

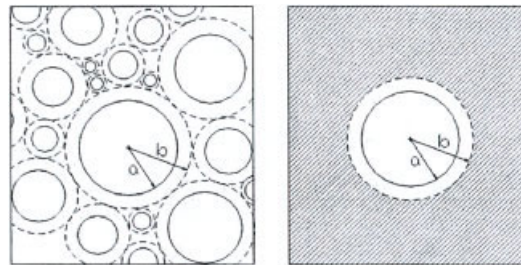


Figure 1 Composite spheres model (left) and three-phase model (right).

$$E = E_1\phi_1 + E_2\phi_2 \quad (1)$$

where ϕ is the volume fraction; subscript 1 denotes the matrix or continuous phase and subscript 2 denotes the particulate or filler phase.

In general, the rule of mixtures is regarded as the upper limit of the elastic modulus. The absolute lower bound on elastic modulus can be obtained, assuming equal stress in the matrix and particulate:

$$E = \frac{1}{\frac{\phi_1}{E_1} + \frac{\phi_2}{E_2}} \quad (2)$$

The upper and lower limits on elastic moduli represent the most widely used relationships produced from the mechanics of materials approach. Although other expressions have been presented using various assumptions, most of the attention has been given to the elasticity approaches.

The composite spheres model, introduced by Hashin, consists of a graduation of sizes of spherical particles fixed in a continuous matrix phase (Fig. 1). In line with this model, Hashin and Shtrikman developed the bounds for the shear and bulk modulus. The resulting bounds on the Young's modulus are:

Lower bound

$$E = \frac{9 \left[K_1 + \frac{\phi_2}{\frac{1}{K_2 - K_1} + \frac{3\phi_1}{3K_1 + 4G_1}} \right] \left[G_1 + \frac{\phi_2}{\frac{1}{G_2 - G_1} + \frac{6(K_1 + 2G_1)\phi_1}{5G_1(3K_1 + 4G_1)}} \right]}{3 \left[K_1 + \frac{\phi_2}{\frac{1}{K_2 - K_1} + \frac{3\phi_1}{3K_1 + 4G_1}} \right] + \left[G_1 + \frac{\phi_2}{\frac{1}{G_2 - G_1} + \frac{6(K_1 + 2G_1)\phi_1}{5G_1(3K_1 + 4G_1)}} \right]} \quad (3)$$

where K is the bulk modulus and G is the shear modulus.

Upper bound

$$E = \frac{9 \left[K_2 + \frac{\phi_1}{\frac{1}{K_1 - K_2} + \frac{3\phi_2}{3K_2 + 4G_2}} \right] \left[G_2 + \frac{\phi_1}{\frac{1}{G_1 - G_2} + \frac{6(K_2 + 2G_2)\phi_2}{5G_2(3K_2 + 4G_2)}} \right]}{3 \left[K_2 + \frac{\phi_1}{\frac{1}{K_1 - K_2} + \frac{3\phi_2}{3K_2 + 4G_2}} \right] + \left[G_2 + \frac{\phi_1}{\frac{1}{G_1 - G_2} + \frac{6(K_2 + 2G_2)\phi_2}{5G_2(3K_2 + 4G_2)}} \right]} \quad (4)$$

These relationships are applicable when $K_1 < K_2$ and $G_1 < G_2$.

The three-phase or spherical inclusion model, introduced by Kerner, is similar to the composite spheres model except that it focuses on a single inclusion in a homogeneous phase. The single spherical particle is

encapsulated by a spherical matrix phase, which itself is embedded in a homogeneous phase. Both models stipulate that the encapsulated sphere has a constant a/b ratio. The advantage of this model is that there is an exact solution. The resulting Young's modulus is

$$E = \frac{9 \left[K_1 + \frac{\phi_2}{\frac{1}{K_2 - K_1} + \frac{3\phi_1}{3K_1 + 4G_1}} \right] \left\langle G_1 \left\{ 1 + \frac{\phi_2}{\phi_1} \left[\frac{15(1 - \nu_1)}{8 - 10\nu_1} \right] \right\} \right\rangle}{3 \left[K_1 + \frac{\phi_2}{\frac{1}{K_2 - K_1} + \frac{3\phi_1}{3K_1 + 4G_1}} \right] + \left\langle G_1 \left\{ 1 + \frac{\phi_2}{\phi_1} \left[\frac{15(1 - \nu_1)}{8 - 10\nu_1} \right] \right\} \right\rangle} \quad (5)$$

The Halpin-Tsai semiempirical equations represent yet another way to predict composite properties:

$$E = \frac{E_1(1 + \zeta\eta\phi_2)}{1 - \eta\phi_2} \quad (6)$$

where

$$\eta = \frac{\frac{E_2}{E_1} - 1}{\frac{E_2}{E_1} + \zeta} \quad (7)$$

and $\zeta = 2$ (for a first approximation).

Some additional mechanical relationships are presented below for Young's modulus and the Poisson's ratio of a composite, as given by Budiansky⁹:

$$E = \frac{9KG}{3K + G} \quad (8)$$

$$\nu = \frac{3K - 2G}{6K + 2G} \quad (9)$$

and for each constituent

$$K_n = \frac{E_n}{3 - 6\nu_n} \quad (10)$$

$$G_n = \frac{E_n}{2 + 2\nu_n} \quad (11)$$

where $n = 1$ or 2 (1 denotes continuous phase, 2 denotes filler phase).

EXPERIMENTAL

Materials

Table I shows the materials, their suppliers, and amounts used in this study. All materials were used as supplied. The α -cellulose used in this work is based on cotton and has a particle size of $150 \mu\text{m}$. Table II shows the chemical composition of cellulose powder (Ariacel-A150) used in this study.

Compounding and cure characteristics

Mixing was carried out on a laboratory-size two-roll mill according to ASTM D3182. Cure characteristics of

TABLE I
Formulation of the α -Cellulose-Filled SBR Composites

Material	phr ^a	Manufacturer
SBR 1502	100	BIPC, ^b Iran
Zinc oxide	5	Gostar Jam, Iran
Stearic acid	1	Minko, Malaysia
Carbon black (N 330)	30	Ahwaz Factory, Iran
α -Cellulose	0, 5, 10, 15, 20, 25	Aria Cellulose, Iran
Parrafin wax	2.0	Rose Polymer, Iran
CBS ^c	0.8	Flexsys, Belgium
Sulfur	1.4	RPC ^d Iran

^a Parts per hundred rubber.

^b Bandar Imam Petrochemical Company.

^c *N*-Cyclohexyl-2-benzothiazyl sulfenamide.

^d Razi Petrochemical Company.

mixes (i.e., scorch time t_2 and cure time t_{90}) were determined using a model 100 rheometer (Hiwa Engineering Co., Tehran, Iran) according to ASTM D2084.

Testing

Various rubber compounds were compression molded into sheets, at 150°C according to their respective t_{90} values. Dumbbell and "Die C" crescent test pieces were cut from the sheets. Tensile and tear tests were carried out on a TS5 tensometer (Santam, Johannesburg, South Africa) according to ASTM D412 and ASTM D624, respectively, at a crosshead speed of 500 mm/min. Test for hardness was carried out using a Shore type A durometer according to ASTM D2240. The other physicochemical tests were: resilience (ASTM D 1054), compression set (ASTM D395), and abrasion (DIN 53516). All tests were carried out at room temperature (25°C).

To study the ageing property of the rubber composites, samples were aged in an air oven at 70°C for 24 h. Then samples were conditioned at ambient temperature for at least 16 h before testing.

Predicting elastic moduli

For the prediction of Young's modulus, rule of mixtures [eqs. (1) and (2)], Hashin equations [eqs. (3) and (4)], Kerner equation [eq. (5)], and Halpin-Tsai

TABLE II
Chemical Composition of Ariacel-A150

Cellulose Purity (%)	99.8
α -Cellulose (%)	95–98
Moisture (%)	2.0
Ash (%)	0.2
pH	6.0

TABLE III
Calculation of α -Cellulose Volume Fraction in Compound with 5 phr α -Cellulose Content

Ingredient	Amount (gr)	Density (gr/cm ³)	Volume (cm ³)	Volume fraction
SBR	100	0.94	106.4	—
Zinc oxide	5	5.47	0.9	—
Stearic acid	1	0.86	1.2	—
Carbon black	30	1.80	16.7	—
Parrafin wax	2	0.90	2.2	—
CBS	0.8	1.28	0.6	—
Sulfur	1.4	2.04	0.7	—
α -Cellulose	5	1.40	3.6	0.027

equations [eqs. (6) and (7)] relationships were used. The Young's modulus of α -cellulose was considered to be equal to the Young's modulus of cotton fibers (i.e., 50 MPa). The Poisson's ratio of matrix and α -cellulose were assumed to be 0.48 and 0.35, respectively. Volume fraction calculations of α -cellulose are presented in Tables III and IV.

RESULTS AND DISCUSSION

Cure characteristics

Figure 2 shows the effects of α -cellulose loading on scorch time t_2 and cure time t_{90} , obtained from a Hiwa 100. It can be seen that scorch time increases with increasing α -cellulose loading, whereas cure time is not significantly affected. The increase of scorch time could be attributed to the acidity of α -cellulose. Given that curing chemical reactions take place in a base media, the addition of any material that increases the acidity of system may cause the time of initiation of curing chemical reactions (i.e., scorch time) to increase.

Tensile strength

The addition of α -cellulose gradually increases the tensile strength until a maximum is attained at 5 phr, as shown in Figure 3. Further increases lead to a gradual decrease in tensile strength.

The initial increase in tensile strength at low α -cellulose concentration (5 phr) could be attributable to

TABLE IV
Volume Fraction of α -Cellulose in Compounds with 10–25 phr α -Cellulose Content

α -Cellulose content (phr)	Volume fraction
10	0.052
15	0.077
20	0.100
25	0.122

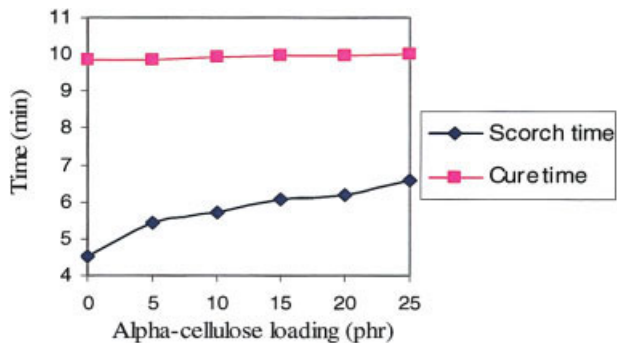


Figure 2 Cure characteristics of SBR- α -cellulose composites.

improved interfacial bonding between α -cellulose and the matrix. α -Cellulose particles have a higher modulus and therefore act as a reinforcing filler, although the reinforcing capability is limited and falls far short of the high-modulus reinforcing fillers such as carbon black.¹⁰ On the other hand, for small amounts of filler, good embedding may be obtained and an increase in tensile strength occurs.¹¹ Also, the interaction between the filler and rubber matrix plays a very important role in increasing tensile strength.¹² Above 5 phr α -cellulose content, agglomeration and thus particle-particle interaction of the α -cellulose account for the observed decrease in tensile strength.¹⁰ As the filler loading increases, it can be anticipated that filler particles and aggregates in synchrony will not be dispersed and wetted efficiently by the rubber matrix.^{10,13} These inherent defects can act as stress concentration points and, consequently, decrease the tensile strength of the vulcanizates.^{13,14}

Elongation at break

The incorporation of α -cellulose into styrene-butadiene rubber (SBR) composites reduces the elongation at break (Fig. 4). This is a common observation, given that many researchers¹²⁻¹⁵ also reported a decrease of elongation at break with the addition of more filler to the polymer matrix. With increasing α -cellulose load-

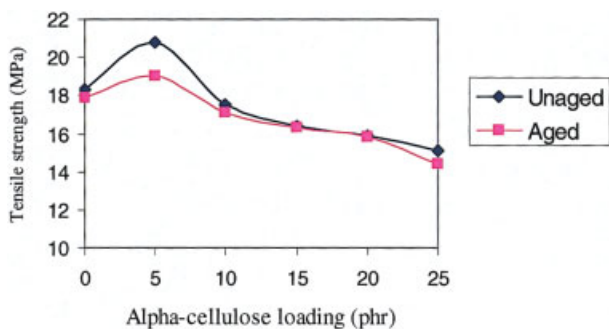


Figure 3 Tensile strength of SBR- α -cellulose composites.

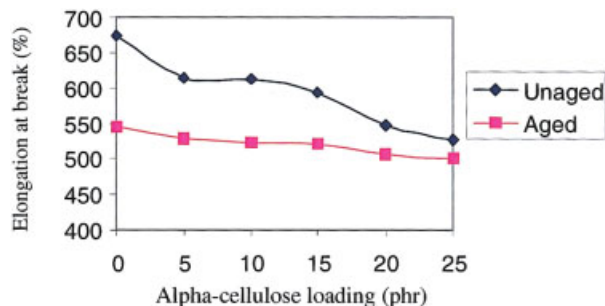


Figure 4 Elongation at break of SBR- α -cellulose composites.

ing, the stiffness and brittleness of the composite increased gradually, with a concomitant decrease in the elongation at break.¹³ In other words, with the enhancement in rigidity, the ductility of composites decreases; consequently, the composites break at lower elongation.¹⁴ Incorporation of fillers that have poor adhesion to the polymer matrix seems to cause interruption in the alignment process of the chains. When filler loading is increased, more weak interfacial regions between the filler surface and the rubber matrix are formed. Because cracks travel more easily through the weaker interfacial regions, the composites fracture at a lower degree of elongation with increasing filler content.¹⁴ Also, as filler loading increases, a higher restriction to molecular motion of the macromolecules is expected. In other words, the addition of more α -cellulose tends to impose extra resistance to flow and lead to lower resistance to break.¹⁵

Elastic modulus

Figure 5 shows the relationship between α -cellulose loading and Young's modulus of SBR composites. It can be seen that Young's modulus increases with increasing α -cellulose loading. The addition of rigid and stiff particulate filler would increase the modulus of the composites because of the introduction of restrictions on the mobility of the polymer molecules.^{14,15} Elastic modulus is affected by several factors such as

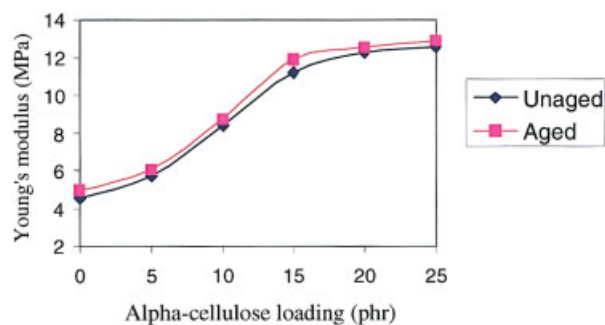


Figure 5 Young's modulus of SBR- α -cellulose composites.

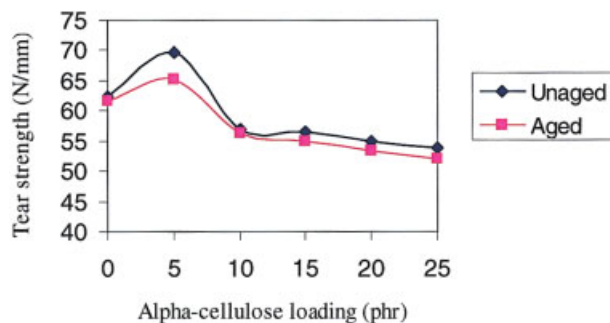


Figure 6 Tear strength of SBR- α -cellulose composites.

surface reactivity, which determines the polymer-filler interaction; aggregates; size and shape of particles; and structure and filler particle dispersion in rubber.¹²

Tear strength

The effect of α -cellulose loading on the tear strength of SBR composites is shown in Figure 6. It can be seen that the dependency of tear strength on filler loading is very similar to that of tensile strength, as reported by several researchers.^{10,13} Both tensile and tear strength typically indicate the resistance of polymer chain network to fracture.¹⁶

Hardness

Hardness increases with increase in α -cellulose content, as shown in Figure 7. This result is expected because, as more α -cellulose is incorporated into the rubber matrix, the plasticity of the rubber chain is reduced, resulting in more rigid composites.¹³ The hardness is increased because the α -cellulose particles have relatively higher modulus than that of the rubber matrix.¹⁰ It is well known that the addition of filler in rubber compounding leads to a linear increase in a material's hardness. It is also known that the international rubber hardness degree (IRHD) is correlated with the elastic modulus.^{11,17}

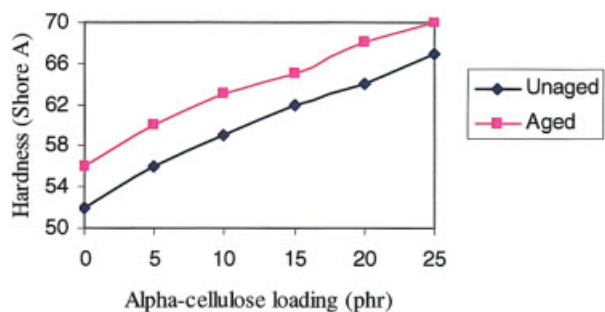


Figure 7 Hardness of SBR- α -cellulose composites.

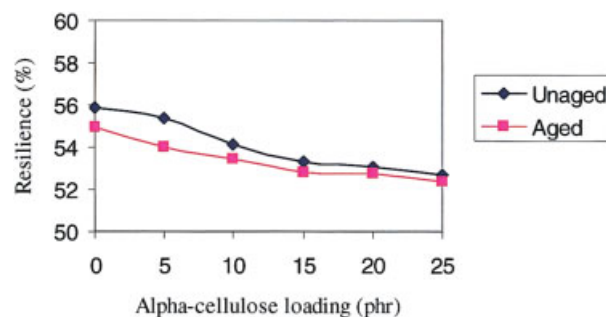


Figure 8 Resilience of SBR- α -cellulose composites.

Resilience

A gradual decrease in resilience, which is not so significant with increasing α -cellulose loading, is observed (Fig. 8). The decrease in resilience is explained by the α -cellulose particles acting as fillers that introduce a mechanism whereby the strain energy diminishes. This is caused by the decreased segment mobility of the matrix molecules arising from their interaction with filler particles. This will result in increased hysteresis.^{10,16} Thus hysteresis increases and, of course, the opposite trend is exhibited in terms of resilience. If a large amount of filler is used, the hardness achieved is high, but resilience decreases¹⁸ because there are more points of slippage between the filler and the matrix, and also because the filler tends to clump together so that particles touch one another instead of being totally embedded in the matrix.¹¹ The mobility of α -cellulose particles and slippage of chains attributed to applied stresses on vulcanizate increase the hysteretic behavior of the vulcanizate.¹⁶ Therefore, the resilience decreases with increasing α -cellulose loading.

Compression set

Figure 9 shows the relationship between α -cellulose loading and compression set of SBR composites. It can be seen that compression set increases with increasing α -cellulose loading. The increase is explained by the decrease in resilience with addition of α -cellulose in compounds; the reduction in resilience, in turn, means

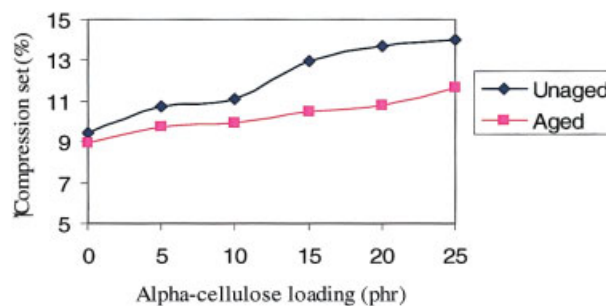


Figure 9 Compression set of SBR- α -cellulose composites.

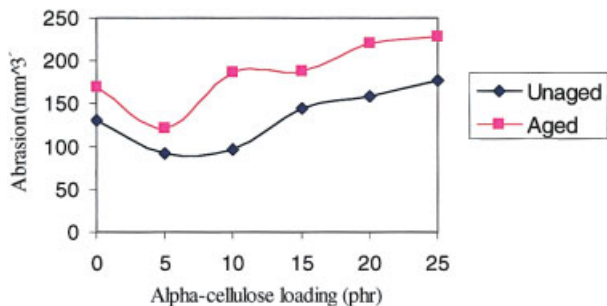


Figure 10 Abrasion of SBR- α -cellulose composites.

that the elasticity of the compound is reduced.¹⁰ Thus compression set that is proportional to elasticity is reduced with α -cellulose loading.

Abrasion

The addition of α -cellulose gradually decreases abrasion until a minimum is attained at 5 phr, as shown in Figure 10. Any further increase in α -cellulose loading leads to a gradual increase in abrasion. Thus abrasion resistance is initially increased at low α -cellulose concentration (5 phr). Above 5 phr α -cellulose content, abrasion resistance is decreased. It is well known that the abrasion resistance of filled rubber is basically determined by filler characterization, especially its morphology and surface reactivity.¹⁹ The lower filler-filler interaction leads to higher abrasion resistance.¹⁹ Therefore, at low concentration of α -cellulose, where there is better distribution of α -cellulose in the rubber matrix, the interaction between α -cellulose particles is minimum (i.e., the abrasion resistance is maximum).

Ageing

During ageing of rubber vulcanizate, a breakdown of the crosslinks—especially polysulfidic ones—occurs.²⁰ Polysulfidic crosslinks are broken into monosulfidic ones; thus, the number of polysulfidic crosslinks is reduced and that of monosulfidic crosslinks is increased.²¹ Excellent tensile strength, rebound resilience, and flex fatigue properties are obtained with polysulfidic crosslinks, whereas resistance to heat ageing and compression set are best with shorter crosslinks.²² Obtained results for unaged and aged samples follow similar trends with lower values for the latter (Figs. 3–10). For example, Figure 3 shows that tensile strength of unaged samples is higher than that of aged ones because of (1) the breakdown of polysulfidic crosslinks to monosulfidic ones, and (2) the formation of induced oxidative crosslinks that have lower strength than that of polysulfidic crosslinks.²¹ Subsequently, tear strength shows a trend similar to that of tensile strength being reduced after

ageing (Fig. 6). Also, Figure 5 shows that Young's modulus increases after ageing as a result of the presence of carbon black in the compound. Carbon black is a well-known trap for free radicals enabled to join macroradicals produced during ageing.²¹ Subsequently, elongation that inversely follows modulus decreases after ageing (Fig. 4). Furthermore, hardness, which has a direct relation to modulus, increases after ageing (Fig. 7). Finally, Figure 9 shows that compression set reduced after ageing because monosulfidic crosslinks, produced from the breakdown of polysulfidic ones during ageing, have a lower compression set than that of polysulfidic crosslinks.²²

Comparison between theoretical and experimental data for Young's modulus

Rule of mixtures

Figure 11 shows the comparison of predicted Young's modulus from rule of mixtures to experimental data for SBR- α -cellulose composites. It is expected that experimental values are within the lower and upper bounds of the rule of mixtures, although this is not observed. This model can be used in two ways: (1) to find an empirical correlation factor for the experimental data and (2) to use this model as a first approximation for predicting the elastic modulus with confidence.

Hashin equations

Figure 12 shows the comparison of predicted Young's modulus from Hashin equations to experimental data for SBR- α -cellulose composites. It can be seen that obtained results from this model are substantially similar to those from the rule of mixtures. A similar discussion is applied here.

Kerner equation

Comparison of predicted Young's modulus from the Kerner equation to experimental data shows a major departure from the experimental results (Fig. 13).

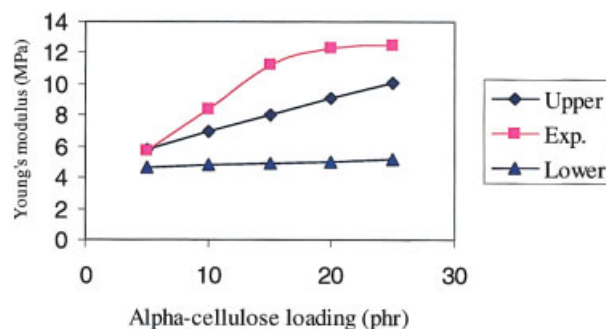


Figure 11 Comparison of predicted Young's modulus from rule of mixtures to experimental data for SBR- α -cellulose composites.

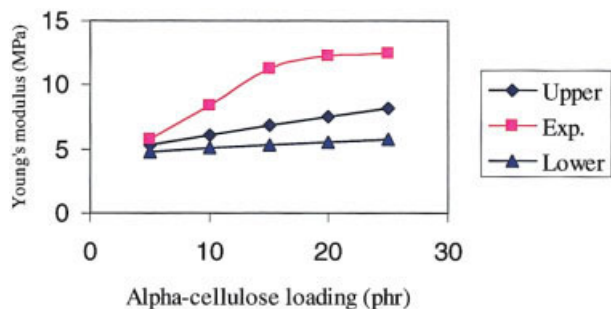


Figure 12 Comparison of predicted Young's modulus from Hashin equations to experimental data for SBR- α -cellulose composites.

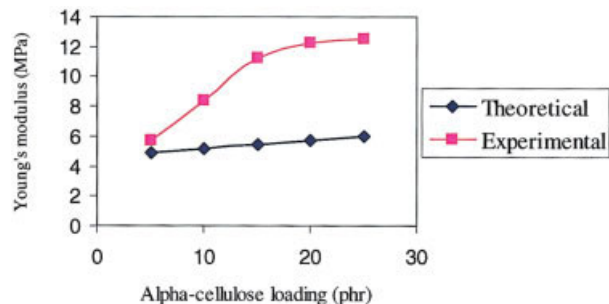


Figure 14 Comparison of predicted Young's modulus from Halpin-Tsai equations to experimental data for SBR- α -cellulose composites.

Halpin-Tsai equations

The comparison of predicted Young's modulus from Halpin-Tsai equations to experimental data shows that this model cannot accurately predict elastic modulus without any correlation (Fig. 14).

It can be concluded from the above discussion that theoretical models developed for particulate composites cannot be applied to predict the elastic modulus of these vulcanizates that are very complicated because of the existence of complex interactions between different ingredients in compound. This confirms the studies of Vilgis and Heinrich²³ on rubber reinforcement, which emphasized that no consistent model exists that may be used to explain rubber reinforcement. Also, as indicated in Eggers and Schummer,²⁴ these equations apply only to uncured systems. Once the rubber is vulcanized, these models no longer apply.²⁵

CONCLUSIONS

From this study, the following conclusions can be drawn:

1. Scorch time increases with increasing α -cellulose loading, whereas cure time is not significantly altered.

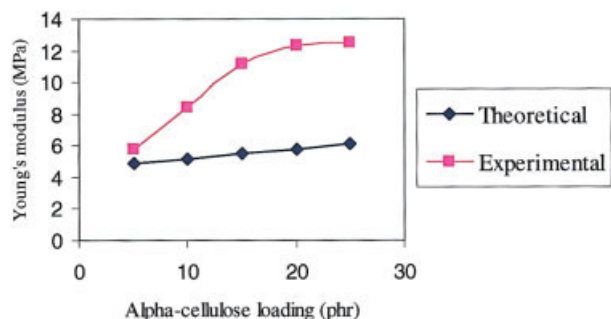


Figure 13 Comparison of predicted Young's modulus from Kerner equation to experimental data for SBR- α -cellulose composites.

2. The addition of α -cellulose gradually increases tensile strength until a maximum is attained at 5 phr. Any further increase leads to a gradual decrease in tensile strength.
3. The incorporation of α -cellulose into the SBR composites reduces the elongation at break.
4. Young's modulus increases with increasing α -cellulose loading.
5. The dependency of tear strength on filler loading is very similar to that of tensile strength.
6. Hardness increases with increasing α -cellulose content.
7. There is a gradual decrease in resilience that is not as significant with increasing α -cellulose loading.
8. Compression set increases with increasing α -cellulose loading.
9. Abrasion resistance is initially increased at low α -cellulose concentration (5 phr). Above 5 phr α -cellulose content, abrasion resistance is decreased.
10. Theoretical models can be used with confidence to predict elastic modulus as a first approximation or initial estimation of elastic modulus because the experimental data are higher than theoretical values.

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