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Some Mechanical Properties of Thermoplastic Rubbers and Blend with Gum Arabic.

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Abstract: *Polyblends of Styrene-Butadiene-Styrene (SBS) elastomer with ground Gum Arabic (G.A) Powder was made by Solution cast method by dissolving in toluene solvent. Tensiometer complex was used to monitor the deformational trend of the pure elastomer and the blend with Gum Arabic. The ultimate mechanical properties of the pure and blended samples were measured using Instron 4301 tensile testing machine. Results of the mechanical properties obtained following the deformation undergone by the test specimens were analyzed using a statistical analytical method, the 'analysis of the Covariance'(ANCOVA) method. Though the tensile set and stiffness of the pure elastomer were enhanced by the incorporation of Gum Arabic, the analysis showed that there was no significant difference in the tensile strength of the pure and blended samples. The result showed that the mechanical strength of the blended samples decreased as the particles sizes of the Gum Arabic within the matrix increased. Also viscoelastic model with a set of constitutive equations was predicted for the pure SBS elastomer.*

Keywords*: ANCOVA, Complex, Constitutive Equations. Polyblends,Tensiometer.*

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I. Introduction

The majority of compounds used by today's rubber industry are based on blends which combine the best features of the rubber blends constituents. Elastomeric blends are used extensively to produce vulcanized products and thermoplastic elastomers. Most elastomeric physical and rheological properties are achieved through blending [1]. A well-known example is the tyre tread compound, where the high abrasion resistance conferred by the use of the polybutadiene rubber (BR) is desirable, but the poorroad holding and rib-tear properties are not. Thus, blends with natural rubber (NR) or Styrene-Butadiene rubber (SBR) are usually employed to achieve the best compromise of traction and wear [2]. Elastomeric blends are of central importance to the rubber industry. Almost all the vulcanized rubber in the industrial and transportation applications are used as blends[3]. Moreover, the science and technology of blend is advancing at a rapidrate. This is particularly true in the case of blends for the production of thermoplastic elastomers.

Thermoplastic rubber polymers are resistant to the attack by most aqueous reagent, normally, have good electrical insulating properties and have excellent low temperature properties. They are soluble in many hydrocarbon solvents, and are thermoplastic at elevated temperatures. A measure of solvent resistance and high temperature strength can be imparted by mechanically mixing them with insoluble polymers such as polypropylene or by chemical cross linking. This informed the idea of embarking on this research; being that Gum Arabic is equally insoluble in hydrocarbon solvents, possesses good adhesive quality and readily available in large quantity locally in Nigeria. The behavior of thermoplastic rubber in adhesives is dependent on;

(i) The morphology and geometry of the submicroscopic end block phase as it is dispersed in the rubber matrix and

(ii) The compatibility of added ingredient with the two phase present[4].

The phase morphology of the thermoplastic elastomers blended with other polymers can be controlled by the viscoelastic difference between the two. The fineness of the dispersion of the minor phase in an extensive mixing process conditions; shear rate and temperature are important variables since it is unlikely that both polymers in the blends will exhibit the same response. Good compatibility or solubility are not necessarily desirable for effective property modification [5].

Gum Arabic, also known as Acacia gum, or Meska is a natural gum made of hardened sap from two species of acacia tree, Senegalia (Acacia) Senegal and Vachellia (Acacia) Seyal [5]. Gum Arabic with molecular formula $C_{26}H_{34}N_2O_{13,}$ is water soluble. Nigeria is the 2nd largest producer of Gum Arabic in the world after Sudan, with average production of 20,000 metric tons of all grades of Gum Arabic. Gum Arabic has a wide range of

industrial uses especially, in areas of feeds, textiles and pharmaceutical industries. In textile industry, it is used for fabric stiffening and as a binder for textile printing gums; it is also used in plastic industry.
The test samples were analyzed to ascertain the mechanical properties. The blended material samples were deformed under static tension to determine their stiffness, elastic strength and resilience. The presence of the Gum Arabic within the SBS elastomer matrix was expected to increase the stiffness (spring constant) with loading. Dimensional stability is one of the most important properties of solid materials, e.g. glass, ceramics, cement, metal, polymers; hence, it is an important consideration in choosing a polymer to use in the manufacture of an item. No one wants a plastic telephone receiver which sags after sitting in its cradle for several weeks, or a cartyre that develops a flat spot if parked on one position for too long, or clothes made from synthetic fibres which becomes baggy and deformed after short period of wear. Creep testsprovide a measure of this tendency to deform and are relatively easy to carry out. Creep is the time dependent relative deformation under a constant force (tension, shear or compression)[6, 7]. The material behavior is governed by rheological equations of state or constitutive equations. [8, 9]This is a direct manifestation of viscoelasticity.

II. Materials/Methodology

Styrene-Butadiene-Styrene rubber (SBS) (Shell Chemical Company USA), Gum Arabic-(Bauchi State, Nigeria), Toluene Solvent (Eagle Scientific company Ltd, Aba, Abia State, Nigeria). Paraffin Oil (M&B Nig. Ltd, Aba, Abia State, Nigeria). Instron Testing Machine Model 4301, tensionmeter, (Instrumentation Laboratory, Department Polymer and Textile Engineering, Federal University of Technology, Owerri).

2.10 Preparation Test samples, Pure SBS Elastomer, Gum Arabic powder and blend of SBS and Gum Arabic.

Elastomer film test samples of pure SBS rubber were prepared by solution film casting.Gum Arabic was ground to fine powder and sieved to achieve different particle sizes. Specified quantities of the different particle sizes of Gum Arabic were thoroughly mixed with the solution of SBS dissolved in toluene solvent. Homogeneity and evenness of Gum Arabic particle distribution within the elastomer matrix was achieved through constant and rigorous stirring of the mix for 12 hours before casting on petri-dish. The film cast was allowed for a period of 4 weeks at room temperature for the solvent to completely evaporate.

5g of Styrene-Butadiene-Styrene (SBS)Elastomer was dissolved in 30ml toluene solvent and stirred intermittently with the aid of a glass rod for twelve hours until complete dissolution was achieved. The homogeneous solution obtained was casted into films onPetri-dish by allowing it to evaporate over a period of four weeks at room temperature. The dried SBS cast was conditioned in an oven at 50^0C for 48hrs. The film was weighed on a weighing balance at hour's interval, until constant weight was achieved, which was an indication that the SBS cast was completely solvent-free. Samples were then stored in a desiccator containing silica gel for further period of 24hours, this was done to eliminate the presence of any traces of moisture that may be present in the cast samples.

2.20 Preparation of Gum Arabic Test sample.

Gum Arabic was dried properly on an oven at 50^0C and ground to smaller particles with the aid of an electric grinder (blender). A set of calibrated sieves was used to sieve the ground Gum Arabic to obtain particle size of 0.30mm, 0.1mm, 0.075mm and 0.050mm.

2.30 Solution Blending of SBS and Gum Arabic

5g of SBS was dissolved in 30ml toluene solvent (as in 2.10 above) in 5 different beakers. Into each of the 5 beakers, 5g, 4g, 3g, 2g and 1g weight of grounded Gum Arabic (of differentparticle size) were introduced and stirred properly to evenly distribute the Gum Arabic particles in the SBS matrix. The blend was repeated for all the Gum Arabic particle sizes, (0.30mm, 0.1mm, 0.075mm and 0.050mm). The film cast obtained were allowed to evaporate for two weeks at room temperature and conditioned in an oven for 48 hours at 50^0C. The samples were weighed at 2hours interval to achieve consistency in weight. The films were thereafter cut into equal strips (2cm×5cm) sizes and according to ASTM specifications: BS2782, ASTMD638 and ASTMD790.

2.40 Determination of the Ultimate Mechanical Properties of Pure SBS and SBS/GA Blended samples.

The casted film strips of each of the pure SBS and SBS/GA blends test pieces used for the ultimate mechanical properties tests, were prepared in accordance with ASTM standard specifications as explained in section 2.30 above. The width and thickness of each specimen were measured with the micrometer screw gauge and the cross-sectional area at the point of minimum cross-section were calculated as follows:

$$A = \frac{M}{DL} = \text{B x T} \qquad (1)$$

Where:A=cross-sectional Area, M= Mass,D=density,L=length,B=Breadth,T=Thickness.

The results obtained were used to analyze the following mechanical properties: Tensile set before at and after break; stiffness, elastic strength and resistance of both the pure SBS and blend of SBS/GA Samples was calculated using the equation:

Tensile set at break = $\left(L - \frac{L_o}{L_o}\right) x\ 100\%$ (2)

where L=Length of the joined test pieces after 10mm sets

L_o=Original distance between bench marks.

2.50. Determination of creep for pure SBS sample (Long Term Test Using Tensiometer)

Six test samples from pure SBS film cast were cut out with equal dimensions [1cm×7cm]. Each of the test pieces was clamped to the tensiometer.To each of the test samples varying constant weights were attached. The exact time of loading of the weights and the instantaneous deformations were recorded. Under the constant stress or load, the materials continue to deform indefinitely, this phenomenon of containing or time-dependent deformation of the material under constant loads for a period of three months and the temperature was maintained at a constant level (room temperature). The deformation was recorded at regular intervals of two days or more. For more accurate reading, the deformation was measured along the meter-rule, with the aid of a travelling microscope which was set at a stationary position from the commencement of the creep test, the deformation of the material were properly monitored;also, the stress relaxation and elastic hysteresis energy dissipated were obtained.

III. Results and Discussion

TABLE 3.1 summarizes the results of the finding on the deformational behaviour of the various blended samples and the pure SBS samples when subjected to varying tension (stress). The results show that the quantity of Gum Arabic incorporated into the pure elastomer and the particle size of the Gum Arabic affected the mechanical properties of the resultant materials. The mechanical strength of the blended samples decreased as the particle size of the Gum Arabic incorporated increased in the order: Blend with 0.05mm G.A> blend with 0.075mm G.A > blend with 0.10mm G.A > blend with 0.030mm G.A. It was equally observed that the pure SBS sample exhibited lower tensile set after 15 minutes and at break than the samples blende with gum Arabic. Also it was noticed that the higher the quantity of gum Arabic, the higher the tensile set. This behavior is an indication that the presence of gum Arabic in the blend retards or restricts the movement of the molecules of the elastomer from returning to its normal undistorted amorphous or cross-linked position after 15 seconds extension or at break. It was equally observed that the finer the particle size of the incorporated gum Arabic, the higher the tensile set of the materials.

In the region of small deformations, fillers have much the same role as crystallites, especially if the filler surface is wetted by the polymer. Since the gum Arabic particles are not soluble and are retained as reinforcing granules to the SBS elastomer matrix. The main response of a partially crystalline material to an external stress is borne by the amorphous fraction. In materials of low crystallinity, say less than 30% crystallinity, the crystallites function as temperature – dependent cross – link [8, 9].

Table 3.1: Results of Some Deformational Behaviour of Pure SBS Samples and Blend of SBS/Gum Arabic Samples.

S/No.	Sample Type	Particle Size of Gum Arabic (mm).	Tensile Stress (N/m^2)	Tensile Strain (%)	Modulus ((N/m^2)
1	Pure SBS	-	322	11.90	27.06
2	SBS/G.A. Blend	0.050	29.60	26.02	1.14
3	SBS/G.A. Blend	0.075	20.50	24.43	0.84
4	SBS/G.A. Blend	0.10	16.90	21.55	0.78
5	SBS/G.A. Blend	0.30	14.50	20.22	0.73

Table 3.2: Results of Tensile Set @15 Seconds and @ Break for Pure SBS and Blends of SBS/G.A.

S/No.	Sample Type	Particle Size of Gum Arabic (mm)	Tensile Stress @ 15 sec. (%)	Tensile Stress @ Break (%)
1	Pure SBS	-	14.62	14.86
2	SBS/G.A.	0.050	14.71	14.89
3	SBS/G.A.	0.075	14.76	14.93
4	SBS/G.A.	0.10	14.79	14.96
5	SBS/G.A.	0.30	14.79	15.07

The presence of gum Arabic particles in the matrix exhibited increase in stiffness (spring constant) with loading and a moderate increase in damping, i.e. fractional energy loss per loading cycle.

From the results obtained, as contained in TABLES 3.1 and 3.2; the presence of sum Arabic particles within the SBS elastomer matrix exhibited increase in stiffness (spring constant) with loading and a moderate increase in damping i.e. fractional energy loss per loading cycle.

In the stress – strain relationship, the linear portion of the curves give an indication of the stiffness of the materials under investigation which is measured by the modulus of elasticity, E. since E = σ/ε, it is obtained from the linear portion of the stress – strain curve by measuring the slope of the straight line. The result showed that the incorporation of gum Arabic into SBS elastomer increased the stiffness or modulus. Also that the blend simples with larger particles of gum Arabic had higher values of stiffness or modulus than samples with smaller particle sizes.

Elastic strength being measured as the strength (stress) at the proportionality limit, that is, the stress marking the end of the elastic and linear range of the stress - strain curve; within the limit of experimental errors, it was observed that the elastic strength of the pure SBS elastomer was higher than the elastic strength of the blend samples [12].

The elastic resilience, which accounts for the capacity, of the materials under investigation to store energy elastically; was obtained by measuring the area under the stress – strain curve, in accordance with equation:

$$U = {}^{\sigma\varepsilon}/_{2} = {}^{\sigma 2}/_{2E} \qquad (3)$$

Where U = strain energy stored per unit volume.
σ = Elastic stress
ε = Elastic strain
E = Elastic modulus

The results obtained showed that the blended samples possessed poorer elastic resilience that pure SBS samples. The residence of the blend samples varied according to the particle size of the Gum Arabic incorporated in the following order: pure SBS elastomer samples > samples blended with particle size of G. A of 0.050mm> samples blended with particle size of G.A of 0-075m > samples blended with particle size of G.A of 0.110mm > samples blended with particle size of G.A of 0.3mm. The incorporation of sum Arabic with the pure SBS elastomer enhanced the stiffness of the blend, but reduces its resilience.

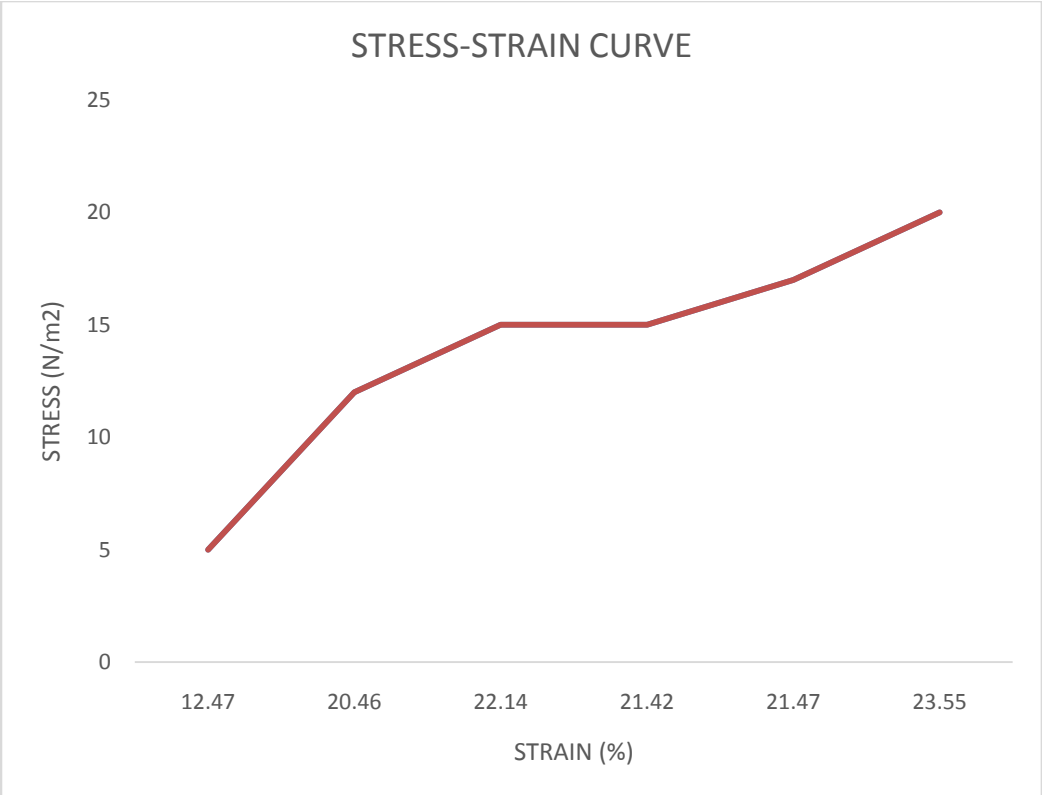

Figure 3.1 Variation of Stress with Strain of Pure SBS Elastomer

In the stress- strain relationship, the linear portion of the curve gives an indication of the stiffness of the material under investigation which is measured by the modulus of elasticity, E, using equation (4).This is

obtained from the linear portion of stress-strain graph, as shown in Fig. 3.1; by measuring the slope of the straight line. The result shows that the incorporation of gum Arabic into SBS elastomer increases its stiffness, which is the modulus. Also, that the blend samples with larger particle sizes of gum Arabic had higher stiffness (modulus) than samples with smaller particle sizes. Modulus E is given by:

$$E = {}^{\sigma}/_{\varepsilon} \quad (4)$$

Where, σ, is stress (N/m^2) and ε, is strain (%).

Elastic strength is usually measured as the strength (stress) at the proportionality limit, that is, the stress marking the end of the elastic and linear range of the stress-strain curve. Within the limit of experimental errors; it was observed that the elastic strength of the pure SBS elastomer is higher than the elastic strength of the blend samples.

The elastic resilience, which accounts for the capacity of the materials under investigation to store energy elastically; was obtained by measuring the area under the stress-strain curve. The result obtained showed that the blended samples possessed poorer elastic resilience than the pure SBS samples. The resilience of the blended samples varied according to particle sizes of gum Arabic in the following order: Pure SBS elastomer samples > samples with particle size 0.050mm > samples with particle size 0.075mm > samples with particle sizes 0.l0mm > samples with particle sizes 0.30mm.

Table 3.3 Variation of Modulus with Log Time (From the Creep Curve)

Modulus (MPa)	Log Time
2.161	0.11
1.879	1.05
1.594	2.05
1.309	2.41
1.023	3.25
0.738	4.05

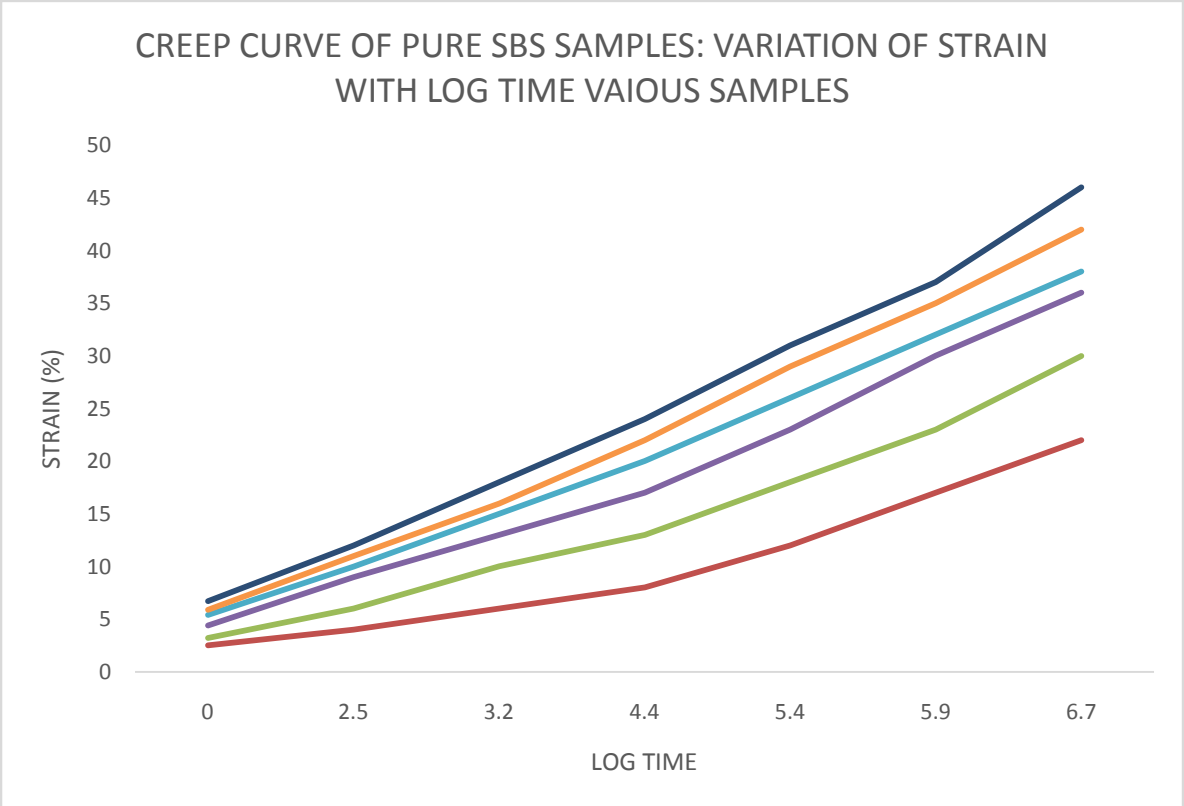

Figure 3.2 Creep Curve of Pure SBS Samples:

Variation of Strain with Log Time of the Various Samples.The creep curve as shown in Fig. 3.2 is the most common method of displaying the interdependence of stress, strain and time. If we take a constant strain section through the creep curves and replotting as stress versus time. The creep curves shown in Fig. 3.2 was used to obtain the data with which the Isometric curve was plotted as shown in Fig. 3.3. The data obtained served as a good approximation of stress relaxation in the material (samples) under investigation.Each of the

strain, ε, on the vertical axis was used to divide the stress producing them and the data as shown in TABLE 3.3 was used to plot a graph of Modulus against Time. This gave a good illustration of the time dependent variation of modulus. By taking a constant time section through the creep curves and plotting the stress against strain, produces a graph termed Isochronous curve.

Under a constant stress or load, materials continued to deform indefinitely; this phenomenon of continuing or time – dependent deformation of material under constant stress is called creep. Fig. 3.2 shows the result of the deformation recorded when six different samples of pure SBSelastomer films of the same dimensions were subjected to six different constant loads. It was observed that the higher the stress level, the materials deformed more rapidly; hence the strain rate, dε/dt, which is also called the steady state creep or minimum creep rate are higher. This creep rate determines the useful life of the material Kt^{-n}.

Table 3.4 Result of Stress Relaxation Experiment.

Stress σ x 10^8 (N/m^2)	Time t (min)	Original Strainε_o (%)	σ (x 10^7)/ε_o (x 10^7)	Log σ/ε	Log t
4.995	8.00	15.00	3.330	17.32	2.08
4.337	20.00	15.00	2.891	17.18	3.00
3.678	32.00	15.00	2.452	17.01	3.47
3.022	74.00	15.00	2.015	16.82	4.30
2.363	134.00	15.00	1.575	16.57	4.90
1.705	299.00	15.00	1.137	16.25	5.70

Stress relaxation is a time – dependent change in stress at constant deformation and temperature. The stress relaxation behavior of polymers is extremely temperature – dependent, especially in the region of glass transition temperature. In the transition region, a plot of the logarithm of the tensile relaxation function ($\sigma_{(t)}/\varepsilon_o$), against logarithm of time, from TABLE 3.4, is nearly a straight line with a negative slope. At both higher and lower temperatures the slope becomes less steep. This behavior can be approximated by the empirical formula:

$$\frac{\sigma(t)}{\varepsilon o} = E_{rl(t)} = Kt^{-n} \quad (5)$$

Where, K and n are constants. For amorphous polymer, n may vary between 0.5 and 1.0; n, a dimensionless number, is a measure of the relative importance of elastic and viscous contribution to stress relaxation[13, 14]. From the graph, which produced a negative slope, the intercept, Log K, was obtained to be 18.3; hence the K – value was calculated to be 8.863 x 10^7 N/m^2. While the slope, n, was obtained to be 0.38. The value fell slightly below the theoretical expected value for amorphous polymers; likely due to experimental anomalies; and also due to the fact that the SBS elastomer is not completely amorphous. Also, the stress relaxation time τ_{rl} was calculated to be 177.19 seconds; within the limits of experimental errors.

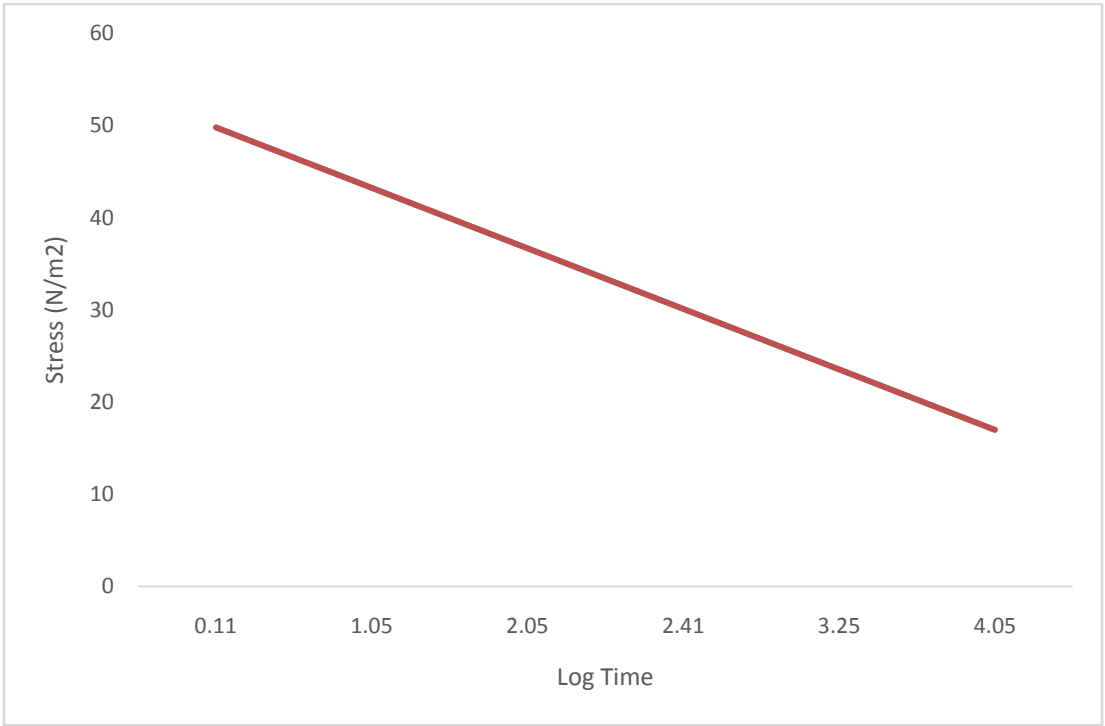

Figure 3.3 Variation of Stress (N/m^2) against Log Time (Isometric Curve).

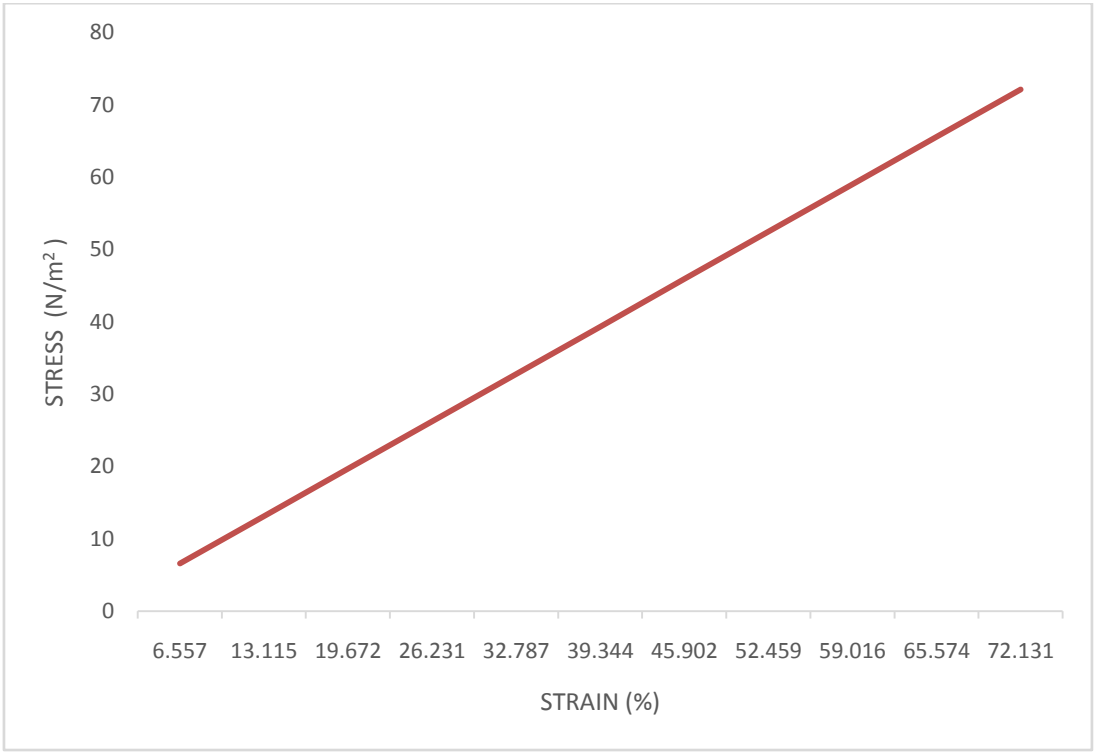

Figure 3.4 Variation Stress (N/m2) Versus Strain (%), Derived from the Creep Curve, (Isochronous Curve).

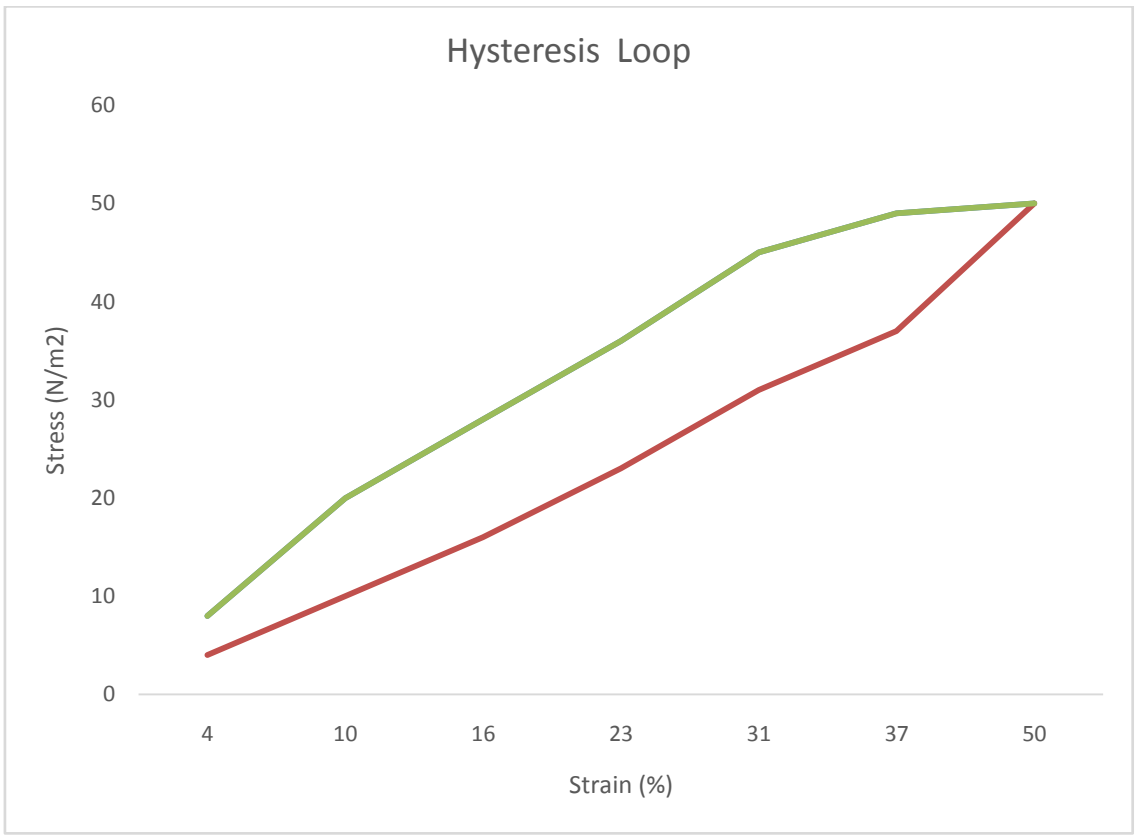

Figure 3.5 Plot of Loading and Off-Loading of Pure SBS Sample (Elastic Hysteresis Loop).

Fig. 3.5 shows the results of the loading and off – loading processes of pure SBS samples. During the loading process, the molecular chains are stretched to achieve alignment; and hence greater crystallinity was achieved in the elastomer. Heat given off by this ordering process more than counter acts the thermal effect due

to volume change on adiabatic stretching. As a result, rubber warms up on rapid loading. Rubber, however, have an exceptional property of negative thermal expansion in the direction of stretching[15]. Therefore, as the stretching rubber eventually cool down, it undergoes further elongation. In practice, the loading and unloading are not fully adiabatic, since there is always some exchange of heat with the surroundings, and the stress – strain diagram will assume the shape of a loop as shown on Fig. 3.5. The area enclosed by the loop is known as the elastic hysteresis loop, and represents the amount of elastic energy dissipated as heat during a loading and unloading cycle. If the material is loaded and unloaded on a continuous cycle, the total energy dissipated in a given period of time is the product of the area per cycle and the number of cycle. For a given relaxation process, the energy loss during a cycle is obviously a maximum at some frequency of loading. In automobile tyres, the frequency of loading increases with the speed of the vehicle. In the common type of tyres, the energy loss becomes a large value at high speeds such as 100 Km per hour. This would increase the heating effect in the tyres, reducing their lift – time as well as increasing the risk of a tyre burst at high speeds. Recent improvements such as fibre – glass reinforced polyester tyres yield a much larger relaxation time than the un–reinforced tyres for the same temperature. With this research, gum Arabic is believed to serve as a good reinforcement agent to serve the same purpose as fibre – glass, if properly incorporated and blended, so that tyre life and safety factors are improved.

The results of the mechanical properties of the pure SBS samples were compared with those of the samples of the SBS/G.A. blends, using the statistical tool of Analysis of covariance (ANCOVA) [10, 11]. The analysis of covariance wasused to measure one covariate in addition to the variate. The variate is the variable of primary interest, in this case 'strength', while the covariate is the variable of concomitant interest, 'percent composition of Gum Arabic'. The Analysis of covariance (ANCOVA) involved partitioning of the total sum of products[10, 11].Following the computation of the data using the ANCOVA analytical tool, the hypothesis drawn at the decision rule was:

Reject the null hypothesis if,

$F \geq F(\alpha, K-1, N-K-1)^{(10, 11)}$ (6)

Otherwise, accept the null hypothesis.

The decision of non-significant difference in strength of the blended and unblended (pure) samples was supported by the identical deformational trend of the pure and blended samples.

The graphs of the creep experiment (data) as shown in Fig. 3.1, which exhibited three stages of creep, each with a different slope. This is a possible indication of the presence of 3 – elements in the system.

The following constituted equations for each of the models were derived from the stress – stain relations:

$$\mu_3\sigma + \frac{E_2 d\sigma}{dt} = \mu_3(E_1 + \mu_1)\frac{d\varepsilon}{dt} + E_2\mu_1\varepsilon. \quad (7)$$

where μ= viscous constant, σ = the stress, E = Modulus constant, ε = Stains

$$E_1\,\mu_2\frac{d\varepsilon}{dt} = \mu_2\frac{d\sigma}{dt} + 2E_1\sigma \quad (8)$$

$$E_1\mu_2\tfrac{d\varepsilon}{dt} = 2\mu_2\mu_2\tfrac{d\sigma}{dt} + \sigma + E_1 \quad (9)$$

$$\mu_2\frac{d\sigma}{dt} + E_1\,\sigma = \mu_3(E_1 + E_2)\frac{d\varepsilon}{dt} + E_1\sigma - E_1E_2\varepsilon \quad (10)$$

$$\sigma\,(E_2 + E_1) + \mu_2\frac{d\sigma}{dt} = E_1E_2\,\varepsilon + E_1\mu_2\frac{d\varepsilon}{dt} \quad (11)$$

The SBS test piece under investigation could possess any of the constitutive equations arrived at above from the five proposed models. Equations 7 – 11 are the possible constitutive equations for each of the proposed models.

IV. Conclusion

Based on the findings arrived at following the research carried out, the following conclusion was reached:

(i) The pure SBS elastomer sample tested under creep experiment (constant stress with increasing strain), for a period of seventy (70) days, was found to show the behavior of a 3 – parameter element model.

(ii) During the loading and off – loading cycle of the pure SBS elastomer samples, a hysteresis loop of $24.56cm^2$ ($2.456 \times 10^{-3}\ m^2$) area, was obtained; which is an indication of the quantity of energy dissipated to the surrounding during such loading and off-loading cycle; whereas the hysteresis loop obtained for the blended sample is $12.4\ cm^2$ ($1.244 \times 10^{-3}\ m^2$). This shows that the incorporation of Gum Arabic into the styrene Butadiene styrene (SBS) elastomer led to increase in the heat energy retention in the material during usage; hence, capable of enhancing the serviceability and durability of the SBS elastomer.

(iii) The incorporation of the Gum Arabic into the SBS elastomer increased the stiffness and tensile set before and at break; but reduced the resilience of the material, meaning that the dimensional stability of the elastomer was enhanced.

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