Study the Tensile Characteristics of Elastomer Composites Reinforced with Alumina and Precipitated Silica Particles

Dr. Jawad K. Oleiwi
Materials Engineering Department**,** University of Technology/ Baghdad.
Dr. Mohammed S. Hamza
Materials Engineering Department**,** University of Technology/ Baghdad.
Mayyadah S. Abed
Materials Engineering Department**,** University of Technology/ Baghdad.
Email:.mayyadah82@gmail.com

ABSTRACT

   Styrene butadiene rubber (SBR) and natural rubber (NR) composites reinforced with alumina (Al_2O_3) and precipitated silica (SiO_2) particles had been prepared separately by calendaring process. It had been studied the adding effect of the alumina and silica particles separately at different ratio (5, 10, 15, 20, and 25 pphr*) on the tensile characteristics of natural rubber NR and styrene butadiene rubber SBR. The results show that the tensile characteristics increase with the increase loading level of the both reinforcing particles. The maximum values of the tensile characteristics were for natural rubber as compared with styrene butadiene rubber, and the reinforcing effect of silica particles higher than of alumina. The results indicate that the maximum tensile strength at break, modulus of elasticity, and elongation percentage at break were (70MPa, 18, 350%) respectively for natural rubber reinforced with (25 pphr) of SiO_2.

Keywords: Natural rubber (NR), styrene butadiene rubber (SBR), composite, elstomer, silica, and alumina.

دراسة خصائص الشد لمتراكب مطاطي مقوى بدقائق من الالومينا والسليكا المترسبة كيميائيا″

الخلاصة
   تم تحضير متراكبات من المطاط الستايرين بيوتاداين (SBR) والمطاط الطبيعي (NR) والمقواة بحبيبات من الالومينا (Al₂O₃) والسليكا المترسبة كيميائيا″ (SiO₂) و بصورة منفصلة بطريقة الدرفلة. حيث تمت دراسة تاثير اضافة دقائق الالومينا و السليكا والمضافة بصورة منفصلة عند نسب مختلفة (5 و 10 و15 و 20 و25 جزء لكل مئة جزء من المطاط) على خصائص الشد للمطاط الطبيعي و مطاط الستايرين - بيوتاداين. حيث بينت النتائج بان خصائص الشد تزداد مع زيادة نسب اضافة مادة التقوية و ان اعلى خصائص شد تم الحصول عليها هي للمطاط الطبيعي مقارنة بالمطاط الستايرين -بيوتادايين, وان تأثير التقوية بدقائق السليكا اعلى من تاثير اضافة الالومينا.

حيث ان اعلى قيمة تم الحصول عليها من مقاومة الشد عند القطع ومعامل المرونة عند استطالة 100 %و النسبة المئوية للاستطالة هي كانت (350% ,70 MPa, 18 MPa) على التوالي للمطاط الطبيعي المقوى بدقائق السليكا وبنسبة (25 pphr).

pphr*: part per hundred rubber.

INTRODUCTION

Elastomer or rubber is amorphous polymer which shows very large strains when subjected to stress and which returned to their original dimensions when stress is removed. There is properly no more complex and widely used class of materials than rubber compounds because of their unusual properties flexibility, extensibility, resiliency and durability, more over their ability to absorb particle filler like carbon black, silica and clay in amounts exceeding their own weight means that the range of properties. The addition of various chemicals to raw rubber, to impart desirable properties is termed rubber compounding of formulation. Typical ingredients include cross linking agent (curatives), reinforcements, antidegredants, process aids, and colorants [1, 2].

Component based on rubbery materials play a very important part in engineering process and products. The most important applications of rubber is in the transport sector with tires and related products which represent nearly (70%) from rubber production. Next in importance is the application in belting for making flat, V-conveyor and power transmission belts, and in the hose and sealing industry. Other important special applications of rubber are in the load bearing pad for bridges, coated fabrics , rain wear, foot wear, pipes and tubing , tank lining for chemical plants and oil storage, gaskets and diaphragms, rubber roller, toys, balloons, sport goods and wide variety of mechanical products [3 - 4].The aim of current research is study the effect of type and loading level for reinforcing particles Al2O3 and SiO2 on the tensile characteristics (tensile strength at break, modulus of elasticity, and elongation percentage at break) of natural rubber and styrene-butadiene rubber separately.

Theory

Styrene butadiene rubber (SBR) and natural rubber (NR) have excellent physical and chemical properties that make them as candidates for heavy duty applications in different areas as tire tread [5-7]. There is no alternative materials In most applications for rubber products due to amazing high deformability and a memory of its original unstressed state and will return to its original dimensions when external forces are removed (in other words; natural shape memory material) due to high molecular mass molecules that can be crosslinked together to forma network and entanglements [8-11].

The raw rubber is usually reinforced with carbon black and carbon nanotubes and inorganic fillers like clay, oxides like silica, alumina and titania, and metallic particles [12-15]. The smaller the size of the particles, the larger is the interface where interactions between polymer molecules and fillers can generate new properties [16]. The enhancement of mechanical properties (stiffness and elongation at break) due to adding the stiff fillers depending on the filler volume fraction, filler shape factor (aspect ratio), the interactions and dispersion between filler and rubber, surface modification, and the chemical nature of the rubber [17].

There are different methods to produce rubber composites: mechanical mixing, solution blending, melt compounding, and latex compounding [dr. saad]. In this

research, the mechanical mixing by calender (two rod mill) was used to prepare rubber composite.

Tensile test is widely used to provide the designer with information about material strength and maximum elongation and others. This test is performed by applying uniaxial force with continuous increasing to obtain simultaneously elongation in specimens [18].

The common values which are calculated from tensile curve for rubber material are:

1. Tensile strength at break (MPa).

2. Modulus of elasticity (MPa); at 100%, 200%, or 300% elongation (Mod 100, Mod 200, or Mod 300); because the rubber behaves elastic behavior up to break.

3. Elongation percentage at break.

Experimental work
Mixing

The preparation of rubber composite for application of NR and SBR require many mechanical tests which simulate conditions that these parts are subjected to such tension. Rubber composite preparation includes processing technology consists of compounding, mixing by two rod mill (calender), shaping; generally molding, and vulcanization. Rubber is always compounded with additives: vulcanization chemicals (sulphur), usually reinforcing fillers, antidegredants, and oil or plasticizer. It is through compounding that specific rubber vulcanizate obtain its characteristics. This work based on standard practice ASTM D3182 for rubber material, equipment and procedures for mixing [19]. The basic ingredients of recipe formulation which used in this work represent in table (1). Table (2) and (3) show some characteristics of rubbers and reinforcing particles respectively [2,20]. Table (4) illustrates the mixing process.

Compression Molding and Vulcanization

Vulcanization or curing are two exchangeable terms used to describe the process whereby rubbers are reacted with chemicals, usually in the presence of heat, to convert the uncured state into the generally accepted "rubbery" or "elastic" state. NR, SBR, IR, IIR, EPDM, and NBR and others react with sulphur and sulphur bearing chemicals to achieve this [21, 22].

After rubber compounds had been properly mixed on the mill and shaped into sheets, the sheets were left for a certain time before vulcanized in clean polished and lubricated molds to execute hot compression molding and vulcanization; the final products are obtained at the end of the vulcanization period. During vulcanization, the following changes occur:

1. The long chain of rubber molecules becomes cross-linked by reactions with the vulcanization agent to form three dimensional structures. This reaction transforms the soft weak plastic-like material into a strong plastic product.

2. The rubber loses its tackiness and becomes insoluble in solvent and more resistant to deterioration normally caused by heat, light and aging processes.

In general, the vulcanization process implied that the vulcanization press was primarily heated by an electric source which were (140, 150 and 170 ºC) for the moulds with dimensions (300*300*6 mm), (40 mm diameter*70 mm height), and (28 mm diameter * 13 mm height) respectively. The empty mold was brought to vulcanization temperature in the closed press and held it for at least 20 minutes before

the unvulcanized sheets were inserted. The press was capable of exerting a pressure of (90 MPa) on the mould surface during the period of vulcanization which were (15, 20, 45 min) for the above moulds respectively. However, the sheets were vulcanized at different temperatures range and periods according to ASTM D 3182 depending on the recommended test [19].

Tensile Test

After specimens' preparation is performed, tensile test is done. This test is executed according to ASTM D 412 [23] for (24) different samples at 20 ºC. The dumbbell samples are shown in figure (1) before and after test. The applied force is (500 N) with pulling velocity of device is (20 mm/ min). The used tensile machine is (INSTRON 1195).

Table (1) The Recipe Formulation.

| Item	Material	Loading level (pphr)	Loading level (pphr)
1	SBR	100	0
2	NR	0	100
3	Zinc oxide (activator)	5	5
4	Stearic acid (activator)	2	2
5	Antioxidant	1.5	1.5
6	Antiozonant	1.5	1.5
7	Process oil	5	5
8	Carbon black (N339)	25	25
9	Precipitated silica or Alumina Filler	(0, 5, 10, 15, 20, 25) *Variable*	
11	Silane Coupling agent (PSAS)	----	----
12	Sulphur	5	5
13	Accelerator (TMTD)	1.7	1.7
14	Retarder (MBTS)	0.1	0.1
15	Reclaim	5	5

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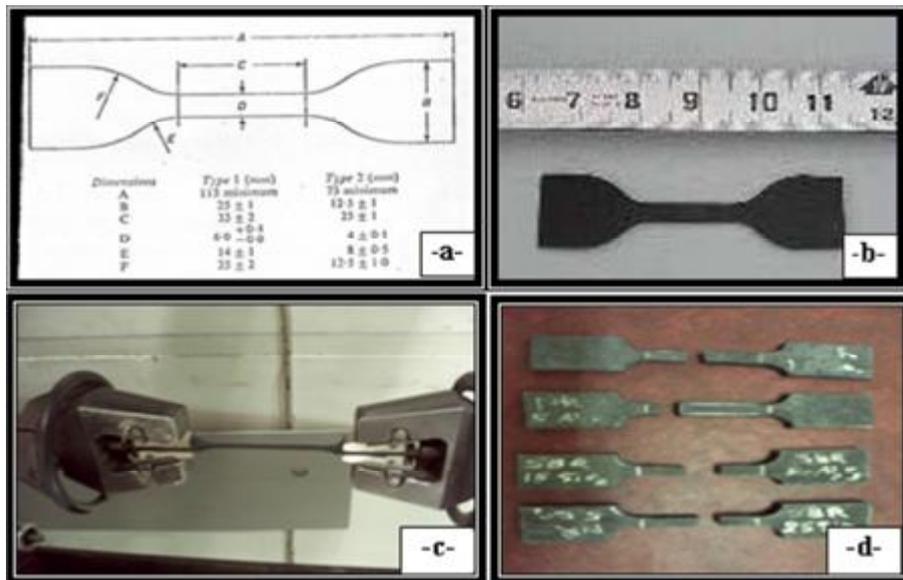
Figure (1): Tensile test specimens: a-Schematic specimens- and b- Before testing; c-During Testing; and d- After break.

Note: All ingredient of the table is constant except the filler adding amount.

Table (2): Some characteristics of SBR and NR.

Property	SBR	NR
Color	Yellow	Brown
Specific Gravity	0.92	0.934
T_g (°C)	- 55	- 70
Tensile strength at break (MPa)	12.4 - 20.7	17.23 - 25
Modulus of elasticity at 100% elongation (MPa)	2 - 10	4 - 15
Elongation% at break	500 – 1000	300 – 700
Origin	India Type 1502,	Malaysia, Type SMR

Table (3): Some characteristics of Al_2O_3 and SiO_2.

Property	*Al_2O_3*	*SiO_2*
appearance	Fine white powder	Fine agglomerate white powder
Specific Gravity	3.88	1.95
Surface area (m^2/g)	……	60
Particle size	30 µm	(20 – 25) nm
Origin	Germany, WGK	Germany, Bayer

Table (4): Mixing Steps.

Item No.	Description	Period (min)
	Master Batch	
1	Mastication of rubber by passing them through rolls several times with decreasing mill roll opening (1-0.5 cm) at 70ºC.	5
2	During whole operation, cutting of milled rubber diagonally, rolled or spiraled ,and passed into nip in horizontal and vertical state alternatively several times for homogenization.	3
3	Adding of Stearic acid and zinc oxide.	3
4	Adding of antidegredants, repeat item (2).	5
5	Adding the reinforcing fillers (Al_2O_3 or SiO_2) blended with coupling agent, and repeat item (2).	7
6	Adding the carbon black with process oil alternatively and repeat item (2).	3
	Final Batch	
7	Adding the pre-blending of sulphur ,accelerators and retarder	10
8	Decease mill opening to (0.3) cm	1
9	Sheeting the batch to a thickness of (0.7) cm.	1
10	Cooling the batch to room temperature rapidly to prevent pre-vulcanization.	5
Total	------	43

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Results and Discussion

Figure (2) shows the relationship between stress and strain in tension for SBR and NR at (0 pphr) loading level of reinforcing fillers. It is clear from this figure that the stress increases with the increase strain in a non-linear relationship and at different rate. The tensile strength of NR equal 19.68 MPa is more than the tensile strength of SBR which is equal 15.58 MPa; this is because of the stereoregularity of NR which is made it susceptible to crystallization upon straining [4]. The crystalline domains of NR restrict the free ends of flexible segments in rubber chains (act as Sulphur cross linking) which then impart the green strength and give vulcanized rubber high cut growth resistance at sever deformation even without reinforcement, but SBR is amorphous and uncrystallized upon straining [4].

Figures (3 and 4) show the relationship between stress and strain in tension for SBR reinforced with Al_2O_3 and SiO_2 respectively at different loading level (0, 5, 10, 15, 20 and 25 pphr). It is illustrated from these figures that the stress increases with the increase strain at different rates for different loading level and different reinforcing fillers Al_2O_3 and SiO_2, that is due to the very large coiled rubber chains which are pulled with stress direction by elastic deformation and no necking takes place until the rubber chains tear and specimen was broken down [24]. Also it can be seen from these figures that the modulus of elasticity and ultimate tensile strength increases with increase the loading level for both reinforcing fillers Al_2O_3 and SiO_2, this is because of *firstly*: reinforcing fillers Al_2O_3 and SiO_2 have breaking strength and tensile modulus higher than that for rubber matrix ;therefore these properties increase for composite, *secondly*: these fillers interfere with the rubber chains and decrease the crack propagation, and *thirdly*: these fillers impede dislocations motion in composite and pin them [25-29].
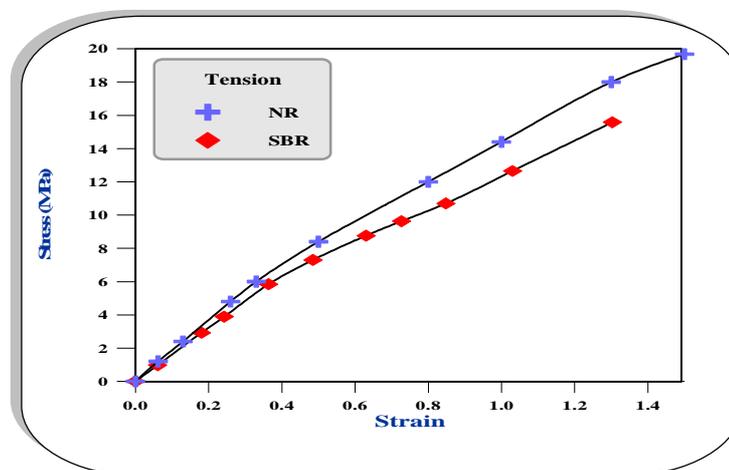

Figure (2): Stress vs. Strain in Tension for SBR and NR at Loading Level (0 pphr) of Reinforcing particles.
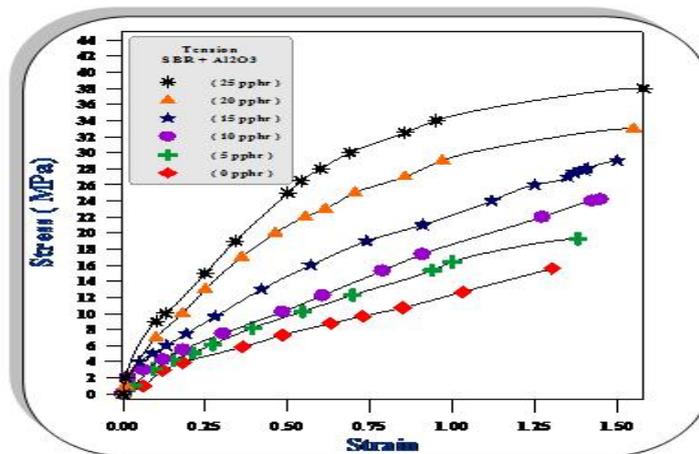
Figure (3): Stress vs. strain for SBR reinforced with alumina at different loading level.
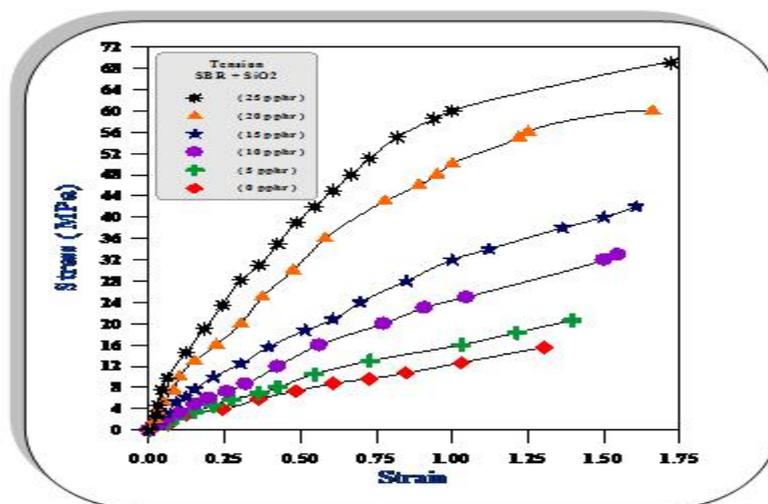

Figure (4): Stress vs. strain for SBR reinforced with silica at different loading level.

Figures (5 and 6) show the relationship between stress and strain in tension for NR reinforced with Al_2O_3 and SiO_2 respectively at different loading level which are (0, 5, 10, 15, 20 and 25 pphr). It is clear from these figures that the stress increases with the increase strain at different rates for different loading level and different reinforcing fillers Al_2O_3 and SiO_2 that is due to the same reason that mentioned before for SBR. Also it can be seen from these figures that the modulus of elasticity and the ultimate tensile strength increases with increase the loading level for the both reinforcing fillers due to the dispersion hardening of the filler. This result agrees with other results Al-Mosawi A. I. *et al.* and Noraiham Mohamad *et. al.* [30,31]

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Figures (7 and 8) show the relationship between the tensile modulus and the loading level of reinforcing fillers Al_2O_3 and SiO_2 at 100% elongation i.e. (MOD 100) for SBR and NR respectively. These figures show that the tensile modulus increases with the increase the loading level of reinforcing fillers Al_2O_3 and SiO_2 in a non-linear relationship at different rate. For NR the increasing rate is higher than for SBR. The SiO_2 reinforcing fillers improve the tensile modulus more than Al_2O_3 reinforcing fillers.

From figure (7), the tensile modulus of SBR increased from 6.74 MPa at loading level (0 pphr) to 10 MPa and 13 MPa at loading level (25 pphr) for Al_2O_3 and SiO_2 respectively, which mean that SiO_2 reinforcing fillers increase the tensile modulus more than Al_2O_3 fillers, this is due to the strengthening mechanism as follows *firstly*: large particle strengthening for Al_2O_3 reinforcing filler which have particle size ≤ 30 μm, this filler tend to restrain movement of the matrix phase in the vicinity of each particle, whereas the matrix (rubber) transfer some of the applied load to the particles and bear fraction of it, and *secondly*: dispersion strengthening for SiO_2 reinforcing filler which have particle size ≤ 40 nm, this filler hinder or impede dislocation movement and require most of stress to bow dislocation in narrow space among particles compared with large particles of Al_2O_3 and the matrix bear the major portion of the applied load [1 and 32].

From figure (8), the tensile modulus of NR increased from 10.2 MPa at loading level (0 pphr) to 15 MPa and 18 MPa at loading level (25 pphr) for Al_2O_3 and SiO_2 respectively, which mean that SiO_2 reinforcing fillers increase the tensile modulus more than Al_2O_3 fillers due to the differences in particles size as mentioned before. This result is in good agreement with other workers [20, 33,34].
The relationship between the ultimate tensile strength and the loading level of the reinforcing fillers Al_2O_3 and SiO_2 are shown in figures (9 and 10) for SBR and NR respectively. From these figures, it can be seen that SiO_2 reinforcing fillers improve ultimate tensile strength more than Al_2O_3 reinforcing fillers in both matrices SBR and NR because of the particle size and mechanical properties difference which mentioned before. So that the ultimate tensile strength of SBR reaches to 33 MPa and 67.35MPa at loading level 25 pphr for Al_2O_3 and SiO_2 respectively. The ultimate tensile strength of NR reaches to 36 MPa and 70 MPa at loading level 25 pphr for Al_2O_3 and SiO_2 respectively. It can be seen clearly that strengthening by NR is more than SBR that due to the flexible segments of NR chains are not immobilized either in crosslinks or crystalline domains which induce upon straining, whereas SBR vulcanizate lacks self reinforcement i.e. strain induced crystallization, but this inadequacy is offset by the addition of reinforcing fillers i.e., carbon black, silica and alumina ….etc [2, 3 and 35].
Figures (11 and 12) show the relationship between the elongation percentage at break and the loading level of reinforcing fillers Al_2O_3 and SiO_2 for SBR and NR respectively. It could be observed from these figures that the elongation percentage at break increases nonlinearly with the increase of the increase of loading level of reinforcing fillers. This is due to the second role of filler, as well as reinforcement role, which is the voids filling that is naturally exist in rubber structure, then they diminished these discontinuous regions which act as stress raiser therefore break strength and strain (elongation) increase. The increasing percentages of SBR elongation from loading level (0 to 25 pphr) are 27.7% and 41.7% for Al_2O_3 and SiO2 respectively. Also the increasing percentages of NR elongation from loading

level (0 to 25 pphr) are 60 % and 200 % for Al_2O_3 and SiO_2 respectively. It can be noted from figures (11 and 12) that SiO_2 reinforcing fillers increase the elongation% at break more than Al_2O_3 reinforcing fillers for both matrices SBR and NR because the wider dispersion of SiO_2 more than Al_2O_3 due to the finer particle size of SiO_2.

CONCLUSIONS

• Tensile strength at break, tensile modulus, and elongation percentages at break increased with the increase loading level of reinforcing particles Al_2O_3 and SiO_2 and for the both rubbers SBR and NR. The maximum values of these properties were (70 MPa, 18 MPa, and 350 %) for NR reinforced with 25 pphr of silica respectively.

• SiO_2 reinforcing particles improve tensile properties nearly about (25-45%) more than of Al_2O_3 particles at the same loading level.

• The both reinforcing particles affect tensile properties for styrene butadiene rubber significantly more than NR, however natural rubber have better tensile properties.

  • The elongation of NR increased more than of SBR, so that the increasing percentages of SBR elongation from loading level (0 to 25 pphr) are 27.7% and 41.7% for Al_2O_3 and SiO_2 respectively. Also the increasing percentages of NR elongation from loading level (0 to 25 pphr) are 60 % and 200 % for Al_2O_3 and SiO_2 respectively.
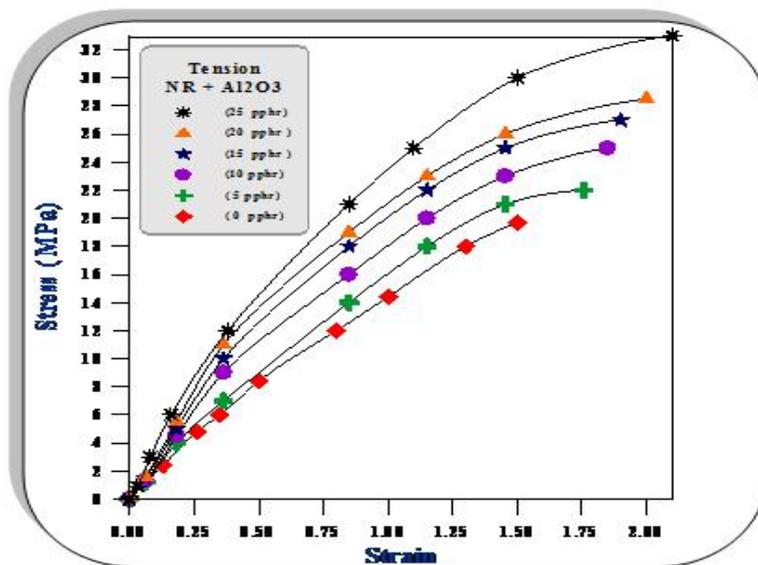

Figure (5): Stress vs. strain for NR reinforced with alumina at different loading level.
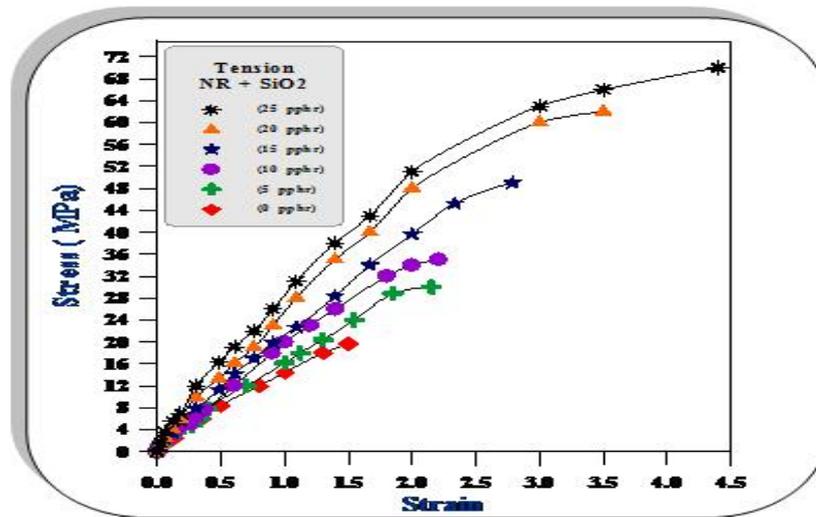
Figure (6): Stress vs. strain for NR reinforced with silica at different loading level.
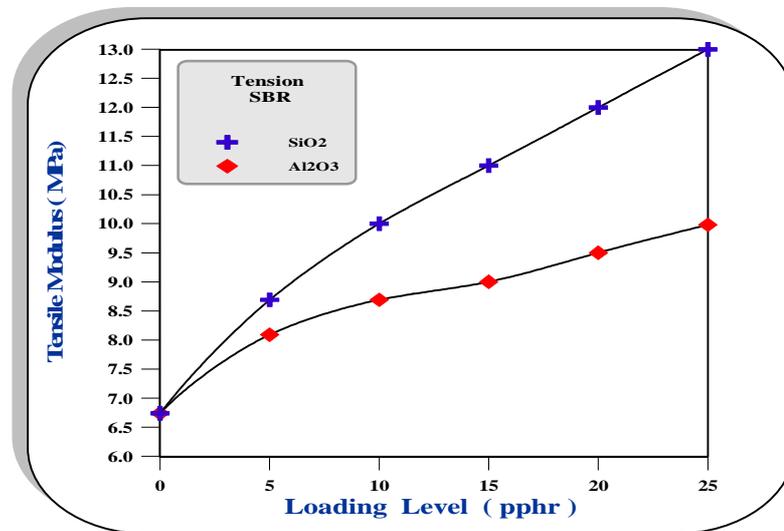

Figure (7): Tensile modulus vs. loading level of alumina and silica for SBR composite.
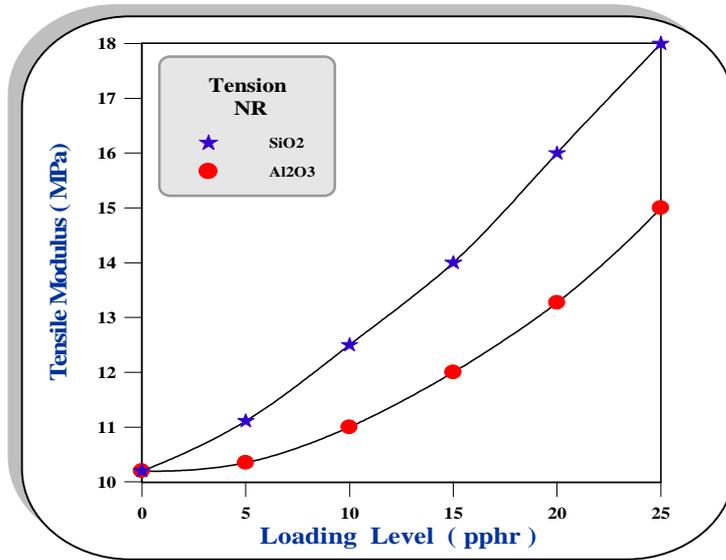
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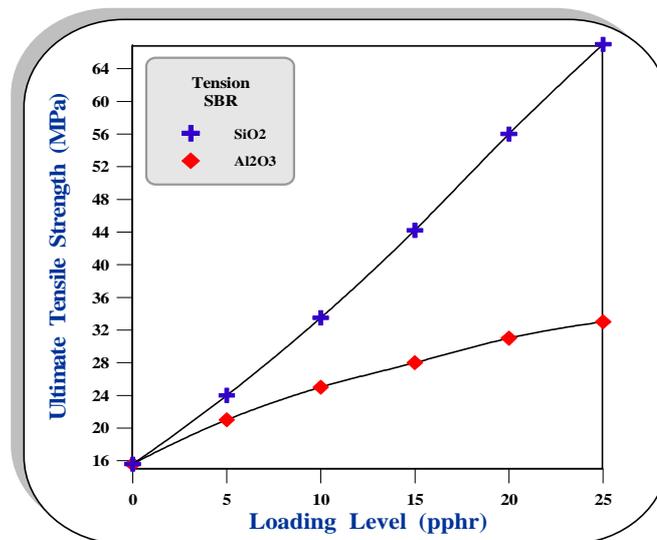**Figure (8): Tensile modulus vs. loading level of alumina and silica for NR composite.**

Figure (9): Ultimate tensile strength vs. loading level of alumina and silica for SBR composite.
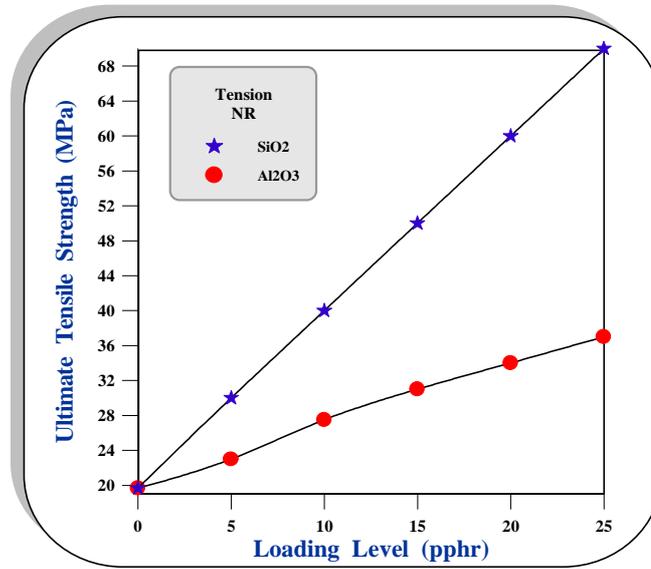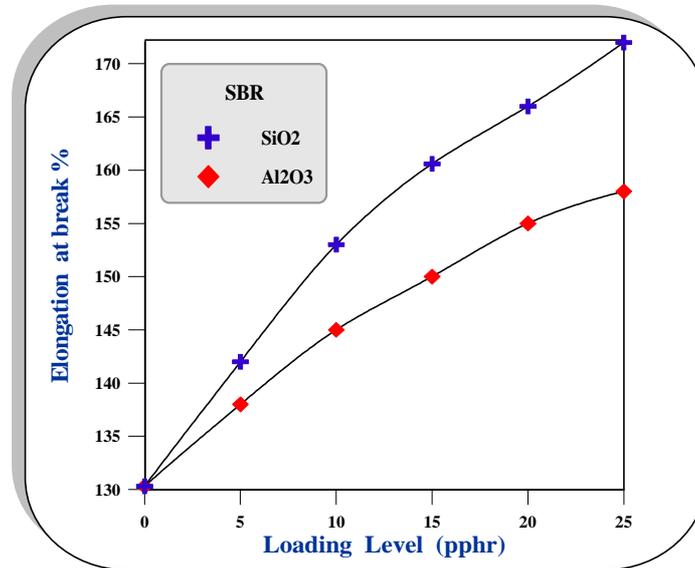
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Figure (10): Ultimate tensile strength vs. loading level of alumina and silica for NR composite.

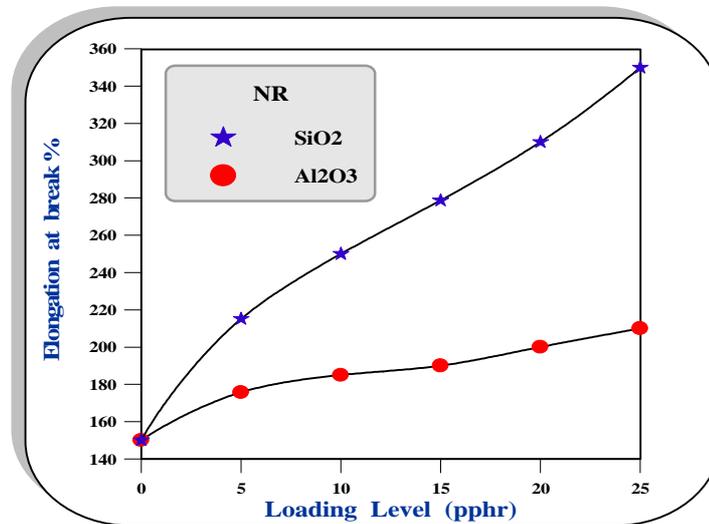**Figure (11): Elongation percentage at break vs. loading level of alumina and silica for SBR composite.**

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Figure (12): Elongation percentage at break vs. loading level of alumina and silica for NR composite.

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