

Reinforcement Effect of Nano Silica on some Mechanical properties of Side wall Tire Batch

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Abstract : The aim of this research it is to get the best pulp of the side wall as tire of the terms of the mechanical properties by using nanosilica. This is done by precipitation from water glass by adding hydrochloric acid.

Many tests are conducting on nano silica such as the (XRD, SEM). The results of the tests nano silica prepared that it forms a spherical irregular, which has a high surface area of roughly sizes less than 100 nm exactly in the range of (48-91)nm are confirmed by SEM. With purity of silica is 100% and density of 0.3511 g/cm³. SEMs -amorphous structure at ($2\theta = 21^\circ$) is obtained by XRD and after being modified by a coupling agent (APTS) a crystalline peak appears.

Nano silica which is used as filler using the following percentages of (0.02,0.04,0.06,0.08,0.1,0.2,0.4,0.6,0.8,1) pphr to the rubber recipe consisting of styrene butadiene rubber(SBR)/ Natural Rubber(RSS) with the optimum constant percent of carbon black (50pphr). Get a perfect blend of nano silica with rubber, it was added modified nano silica worker couplings on the same percentage of nano silica, which protects the surface of the polar silica and reacts with the rubber matrix.

Achieve the effect of nano-silica on some mechanical properties of the side wall tires (hardness, Sp. Gravity, fatigue, Resilience, compression) are Required. Each percent of the addition of nano silica and modified nanosilica act as a filler and it increases the rigidity and flexibility recipe, and then concluded that the specific gravity property and hardness will increase. At 0.02pphr Nano silica, and an increase in the compression starts after that to decrease with nanoparticles increase to 0.04 pphr but at the addition amounts 0.8 pphr of modified nano silica give the good compression loss. At 0.02pphr of nanosilica, increase in the resilience appear then begins to decrease with nanoparticles increase to 0.04 pphr. At 0.8 pphr of nano silica increase the resistance to crack growth up to 1783 cycles. As the amount of Increase result in increased pressure in the recipe which creates cracks.

Key Words : composite materials, Mechanical Properties, silica.

General Introduction:

Composite materials are material made of two or more substances that make up the physical or chemical properties differ materially, that when combined, produce with different characteristics of individual material components. The individual components remain separate and distinct within the final structure. New materials may be the preferred method for many reasons: Common examples include materials that are stronger, lighter and less expensive when compared to traditional materials¹⁹.

Rubber compounding is essentially the science (and art) of selecting and combining ingredients to produce useful characteristics sufficient to perform satisfactorily under the conditions in which the end product is intended to be used. Perspective, the major factors influencing compounding decisions are cost, processing, and properties, not necessary that three factors are in the same importance **1**. In the rubber industry, the problem of selecting the basic raw materials for the preparation of a specific commercial product is usually assigned to the compounder². This background is necessary since some of the processes involve chemical reactions, of which vulcanization is the most important besides the reflected properties which is an engineering materials matter. In addition, chemical and physic mechanical tests are necessarily applied to the final product for evaluating and analyzing the proper selection of the basic raw materials on one hand, and to evaluate and analyzing the final product properties as recommended on the other hand. Making a balance between them is the mission of the compounders and researchers, besides concerning with production of a serviceable product at a reasonable cost. So, compounding can be defined as a process of mixing the basic raw materials in a certain recipe to obtain desired processing characteristics, ultimate properties of the finished product, and the cost control **3**. The object of compounding is to select the most suitable combination of materials in their correct proportion and to determine the treatment that the chosen combination shall undergo in the processes of mixing, forming, and vulcanization so that the finished rubber product is of the required quality and is produced at the lowest possible cost. The operations of mixing, forming, and vulcanizing are the essential fabrication steps **4**. A tire compounder often specializes in one component of a tire, for example, sidewall **12**, The Sidewall compounds consist of natural rubber, SBR along with carbon black and a series of oils and organic chemicals. This part protects the casing from side scuffing, controls vehicle–tire ride characteristics, and assists in tread support **16,11,12**. An amorphous silica consists of silicon and oxygen atoms arranged in a tetrahedral structure of a three-dimensional lattice with silanol groups (Si-OH) present inside and on the surface.

Silica are considered to be a highly polar, reactive filler and the tendency for filler agglomeration rise, because they have a number of surface silanol groups (Si-OH) **15**.

Vulcanizing Agents: Sulfur is most famous Vulcanizing agent. This was readily available in the form of powder and urea are packed in polyethylene bags. Sulfur greatly improves the properties of raw rubber which is sticky and soluble in solvents extent. With the addition of rubber to transform Alkipriteetmnotacky product, tough, flexible **5**.

This study article some of the mechanical properties such specific gravity and hardness, specific gravity tests were carried out by Densitron according to Archimedes principle. it was weighed in air and in water. The specific gravity is calculated by the following equation:-

$$\text{Specific gravity} = \frac{\text{Weight in air}}{\text{Weight in air} - \text{weight in water}} \times \text{specific gravity of water}$$

Resilience : it is one of the characteristic features of the rubber as the ability to show the energy used to deform returns , when deformed rubber, the energy that is not due mechanical as energy they dissipate as heat inside the rubber and rubber ability to represent damping, as the aftershocks associated damping the following relationship:

$$R\% = 100\% - \text{Damping}\%$$

The compressibility a measure for portability material rubber to regain their original form after the demise of the distorted power and thus the sample recovered thickness completely original then it is compressibility 0% but if material rubber did not regain its original form after the demise of acting force, the compression surface will be 100% and so the few compressibility give a numerical value the good high compressibility of the surface gives a few the numerical value **13,14**. And compressibility are calculating according to the equation:

$$C\% = [(t_0 - t) / t_0]$$

Where C represents the compressibility, t_0 = primary thickness, t = final thickness

The Fatigue life occurs when the rubber part is exposed to the cyclic stresses. The rubber fails in fatigue from the growth of one or more cracks that develop from small defects during frequent rubber deformation. The

crack is completely spread through the rubber material, in severe cases. The growth of the crack occurs slowly at first, and may increase rapidly as its volume increases **17**. As a result of many rubber products repeatedly overloaded in service, for example, tires, fatigue and cutting growth deserve great attention **12**. it can be defined hardness and resistance to indentation under specific conditions. This test is conducted on rubbers in accordance with ASTM D1415-687.

Experimental Techniques

1-Introduction:

It describes the particular choice of materials, and material properties, silica (SiO₂) Although a recipe on the side wall tire. And rubber processing technology, roads samples preparation and methods of inspection and testing of mechanical properties.

2-Materials:

The materials used in this research are: Water glass, silica, (3-Amino propyltrimethoxysilane), toluene. The rubber batch materials are Styrene Butadiene Rubber (SBR 1502), Natural Rubber (RSS), zinc oxide, stearic acid, castor oil, sulfur, Rineset, Carbon black (N 375), (N-phenyl-p-phenylenediamine)(CTP-100), (N-(1,3-dimethylbutyl)-N-phenyl-p-phenylenediamine)(6PPD). Which are supplied by Babylon tire company.

3- The recipe Formulation of Tire side wall.

The preparation of rubber nanocomposite material for tire tread supporters requires many mechanical tests which are suitable for application conditions such as (fatigue, Resilience, hardness, Sp. Gravity, compression). another physical tests are necessary under aging intemperture, ultraviolet. The suitable recipe for these applications require the processes to improve these parts by preparation of some reinforcement materials as silica and mixing a different ratio of these reinforcement with the recipe for provide a laboratory samples of various recipes for mechanical tests by using the standard moulds according to the ASTM. The standard specifications used in this work are those of the tires company of Babylon for the tire industry which is described in the **Table (1)**.

Table(1)the standard specification for the tire industry(IW8035)

the specification for the tire industry (IW8035)	
Viscosity (moony viscous)	8-16
Cure time (min)	2.2-3.1
Scorch time (min)	0.7-1.5
Torque (N.m)	Min22
Hardness (shore A)	52-60
Sp. Gravity	1.108-1.114

The basic ingredients of recipe formulation used in this work are based on the basic formulation of passenger tire side wall. The master batch is prepared from Styrene Butadiene Rubber (SBR) and Natural Rubber (RSS) with addition of some of materials (Such as zinc oxide, stearic acid, castor oil, sulfur, Rineset, Carbon black (N 375), N-phenyl-p-phenylenediamine) (CTP-100) (N-(1,3-dimethylbutyl)-N-phenyl phenylenediamine) (6PPD). After this is done to improve the recipe by reinforcement with modification nano silica of nano silica as ratio shown in **Table (2)**.

Table(2) Shows The composition of the rubber nanocomposites

compounding ingredients (pphr)	Pure composite (pphr)	SBR/RSS/Silica nanocomposite (pphr)	SBR/(Silica with capouling agent)
Styrene Butadiene Rubber (SBR)	50	50	50
Natural Rubber (RSS)	50	50	50
Rineset	0.15	0.15	0.15
Stearic acid	1.0	1.0	1.0
Zinc oxide	5.0	5.0	5.0
Wat	2	2	2
CTP-100	0.13	0.13	0.13
Antiozonant(6PPD)	3.25	3.25	3.25
DOP oil	7.0	7.0	7.0
Sulfur	1.8	1.8	1.8
Carbon black (N-370)	50	50	50
Accelerator (CBS)	1.0	1.0	1.0
Silica	0	(0.02,0.04,0.06,0.08,0.1,0.2,0.4,0.6,0.8,1)	
modification of nano silica	0	0	(0.02,0.04,0.06,0.08,0.1,0.2,0.4,0.6,0.8,1)

After preparation of the samples from the original recipe and make mechanical tests were compared to the results of tests as shown in **Table (3)**, however, the specifications described in **Table (1)**.

Table (3) Mechanical tests for master batch without silica

Properties	Value
Viscosity (moony viscous)	13.17
Cure time (min)	2.22
Scorch time (min)	1.17
Hardness (shore A)	54.5
Sp. Gravity	1.113
Torque (N.m)	24.70
Rebound resilience %	38.8
Fatigue cycle	1120
Compression %	0.046

4- Models preparation:

4-1 Model for preparing samples for vulcanization properties of the tire side wall Batch:

In this test model is prepared before vulcanization process. The sample is cut with diameter 2mm and thicknesses 6mm and placed in the device (Oscillating disc rehometer) and the vulcanization get inside the device. In this test, the properties of cure time, scorch time, torque and viscosity were studied.

4-2 Model for preparing samples for fatigue :

Template that is used to prepare of fatigue test samples contains 12 cavities of equal dimensions with thickness 6.5mm. The mould is heated at 160 °C and the recipe put in the mould, then the mould is covered and placed in a hydraulic press for vulcanization in hydraulic piston at temperature 160°C for a period of 15 min and pressure 4 MPa. The sample are taken off from the mould and left 24 hour for cooling before testin.

4-3 Model for preparing samples for Hardness and Sp. Gravity and Resilience:

Template that is used for the preparation of these models has the following dimensions 200 * 180 mm and consists of 9 circular holes with diameter 45mm and equal volumes. The mould is heated and the appropriate amount of recipe is put in the mould, and then the mold is covered and placed in a hydraulic press for vulcanization at pressure 4MPa and temperature 160°C for 15min. The samples are taken off from the mould and left for 24hr for cooling before test.



4-4 Model for preparing samples for compression :

To prepare the test sample compressibility conducting following these steps We are heating the mold at a temperature of 160oC temperature for 15 minutes. We put the sample within the mold cavity and then cover and press. t the mold is covered and placed in a hydraulic press for vulcanization at pressure 4MPa and at a temperature of 160oC degree heat for 30 minutes. Samples extracted from the mold and leave for 24 hours before the test and the sample dimensions are 30mm in diameter and 12mm thickness.

5-Samples Preparation

The hand-made wooden molds use measurements and dimensions according to the American Society for testing and Materials (ASTM). Table (4) shows the dimensions of the samples and forms.

Table (4): shows the dimensions of the samples and forms.

	Standard	Samples and dimensions	Test
3	ASTM D 1415		Hardness & Speasific Gravity & Resilience
4	ASTM D 395		Compression

Results and Discussion

1-Analysis results of nano-silica:

1-1 Scanning Electron Microscopy (SEM):

Figure (1-1) explained the different magnification of silica that are measured microparticles and nanoparticles surface. The results of these analyzes showed that observed in spherical nanoparticles diameters in the range between(48-91)nm and significantly agglomeration by this examination. This agree with the results of research⁸.

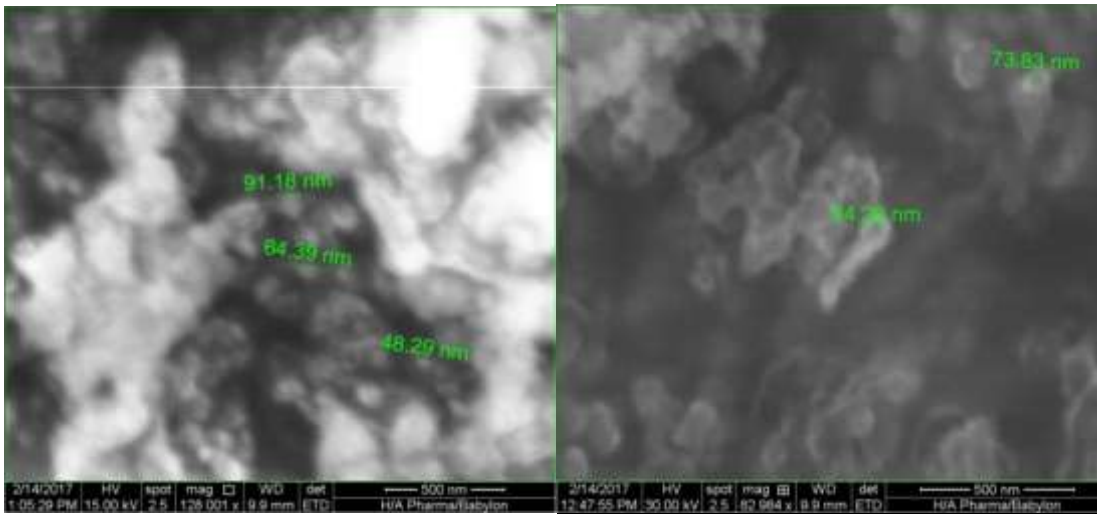


Fig. 1-1 SEM micrographs images of precipitated SiO₂

1-2 X-Ray Diffraction Analysis (XRD)

Of the test X-ray powder nano silica at the corner of diffracted (10° to 50°), a crystalline peak appeared weak and which refers to the installation of the semi-amorphous ($2\theta = 21^\circ$), figure (1-2a). Oadhaoho sodium chloried peak (by-product), apparently with a very low concentration ($2\theta = 25.69$, $2\theta = 31.574$) Results of neutralizing water glass with hydrochloric acid and disappear this big laundry where the figure (1-2b). This refers to the preparation of amorphous material which agrees with the results of research⁹.

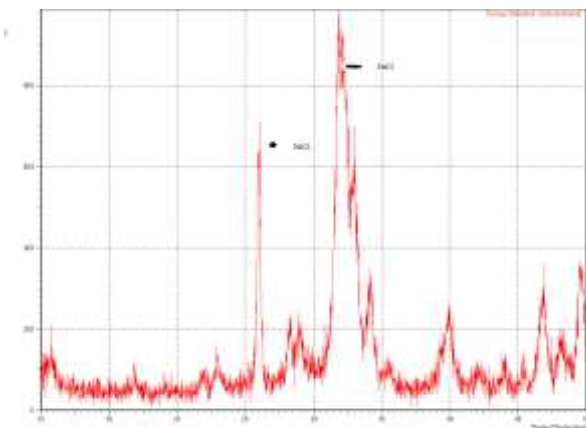


Fig. 1-2aX-Ray diffraction analysis of precipitated SiO₂ with amorphous peak (before washing)

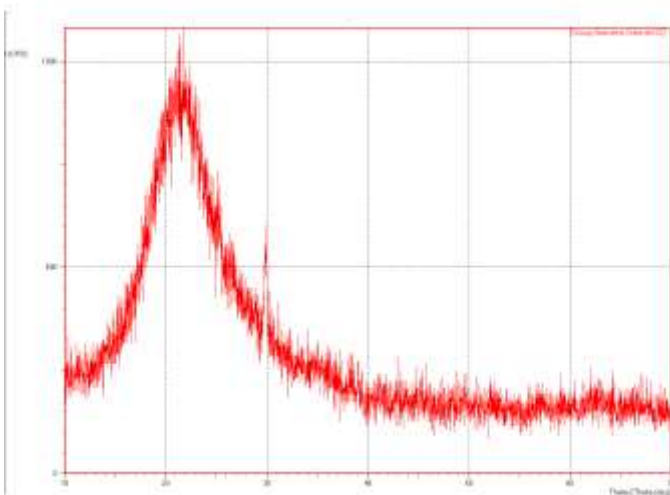


Fig. 1-2b X-Ray diffraction analysis of precipitated SiO₂ (after extra washing)

2-Effect of Nano Silica and Modified Nano Silica with Coupling Agent on the mechanical Properties of Recipe:

The effect of nanosilica and modified nano silica particles on the mechanical properties of tire wall side recipe of constant carbon black (50pphr) shows:

1-Hardness property :

Show that **Fig.(1)** increases hardness with small amounts of both nanosilica and modified nano silica property. it up to the maximum value at 0.8pphr of silica As a result of silica, which operates as a filler that enhances the recipe and makes them resistant to the forces of to the applied value of high hardness and this is in line with the increase in the elastic modulus as shown in **fig.(1)**.But note through the form **fig.(1)** when we add small amounts of modified nano silica gives the best value of the hardness is due to the reason for this lack of conglomerate rubber granules between the chains .This agree with research **3,6,12**.

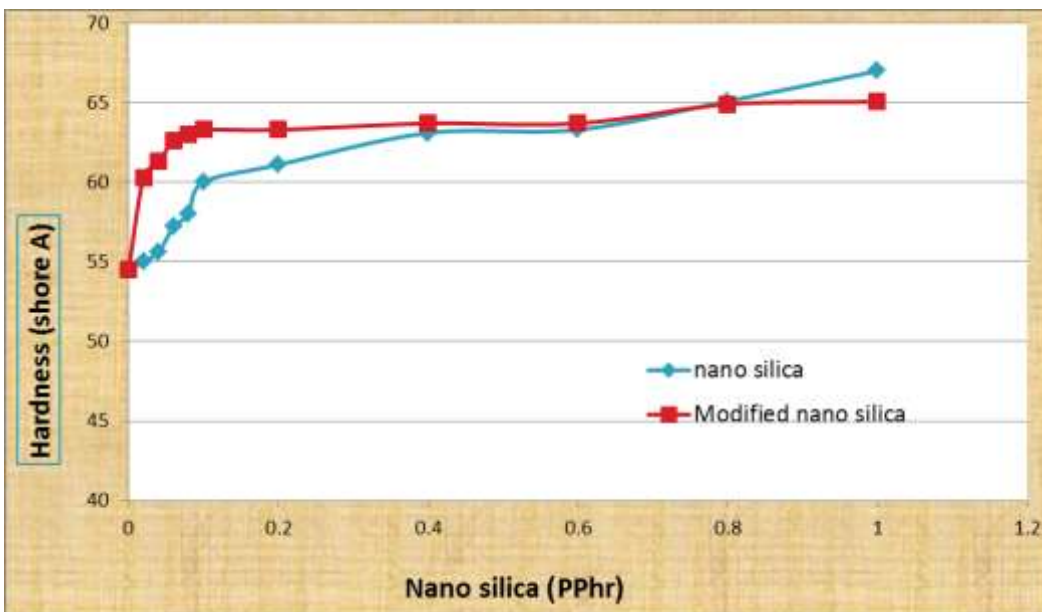


Fig. (1) Effect of nano silica and modified nano silica on hardness of recipe

2-Specific Gravity property

In each addition percent for each of the nano silica and modified, and works as a filler silica as I mentioned before, so it increases the unit volume weight, leads to increased quality and number density as show in **fig.(2)**.This agree with research **6,18**.

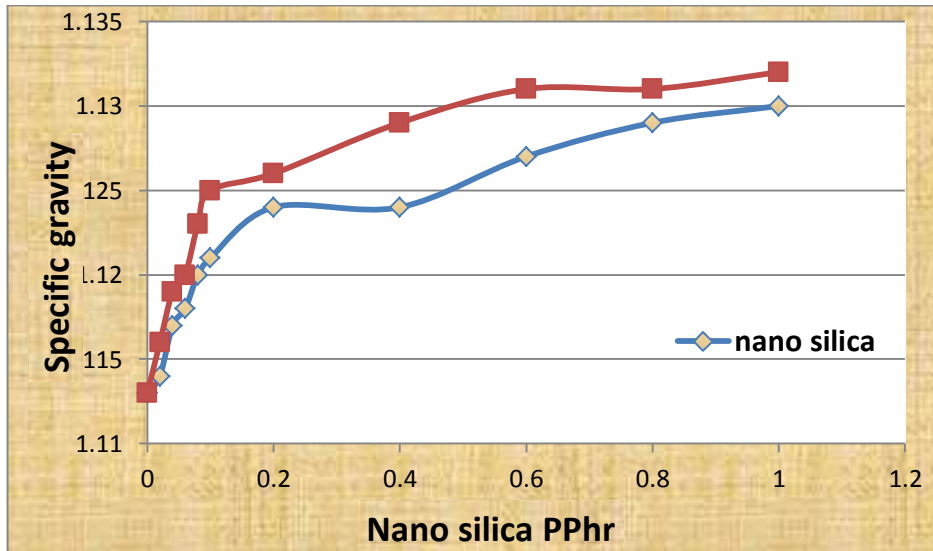


Fig.(2)Effect of nano silica and modified nano silica on specific gravity of recipe

3-compression

When adding small amounts of nano-silica and modified nano silica increased compressibility values of up to a maximum value of 0.080% at 0.02 pphr of silica and then begin to decline gradually to reach the value of 0.042% at 0.8 pphr of silica which is which is the best value for compression tires. The reason for this is that increasing the amount of additive caused the formation of linkages new picket inside the product and these entanglements restrict chains movement of rubber and thus reduce the resistance of compressibility. But note through the **figure(3)** when you add the modified nano silica given values of compressibility best result of the absence of bloc grained between rubber chains.

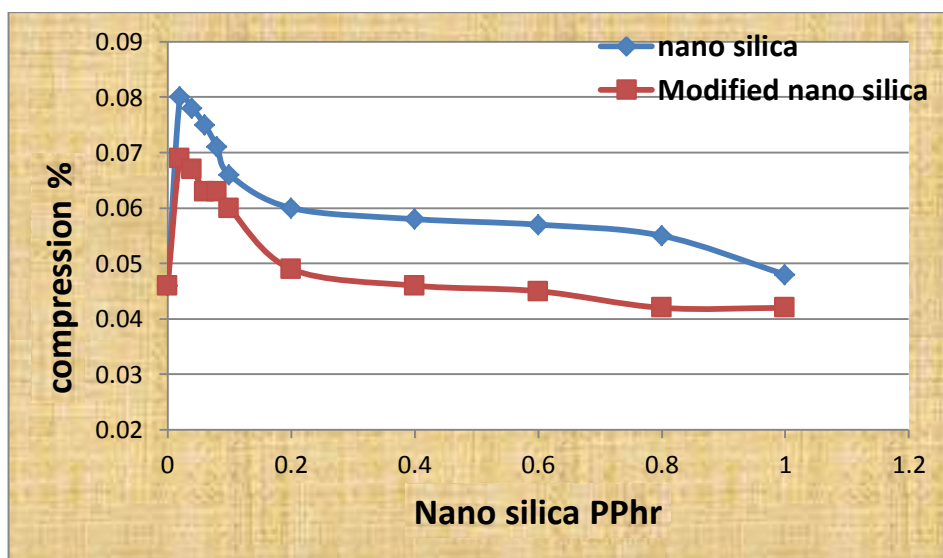


Fig. (3) Effect of nano silica and modified nano silica on of compression recipe

4-Fatigue property:

This property represents a material resistant to cracking growth. In small amounts from each of the silica nano and modified, as shown in the **figure(4)** silica act as a filler and it gives resistance to crack growth. But note of **shape (4)** when adding small amounts of nano-modified silica give us a longer lifespan for tearing materials up to the value of the 1800 cycle as a result of the lack of conglomerate Alhabayat between rubber chains. This agree with research **6**.

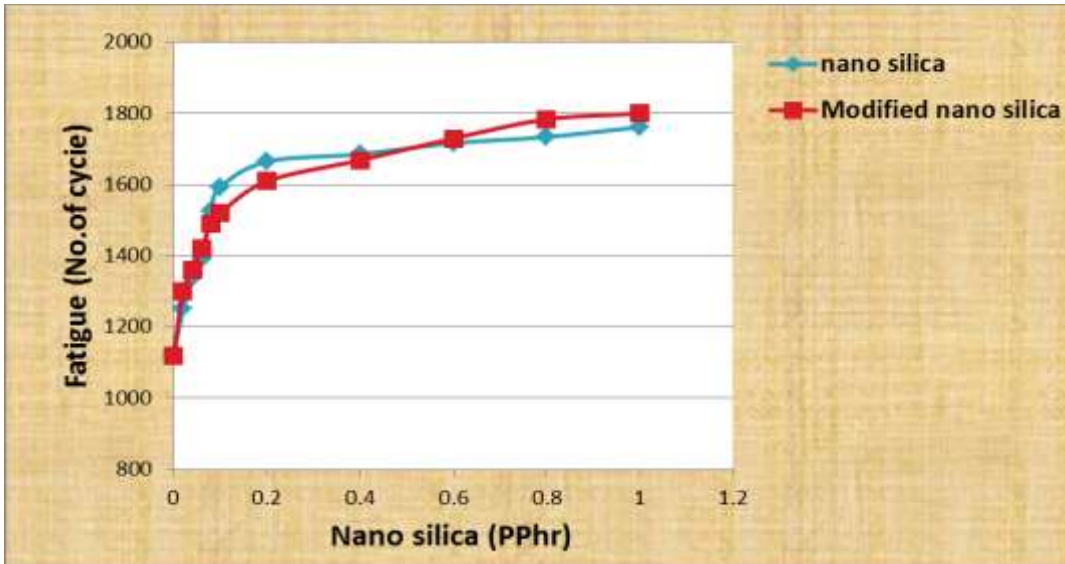


Fig. (4)Effect of nano silica and modified nano silica on fatigue property of recipe

5-Resilience

Show that **Figure (5)** an increase in the flexibility of the recovery. At 0.02 pphr both silica and nano-width modulation Nano jumps in values Besbtaatsrv silica as a filler to fill in the blanks and the dose and the lack of energy absorption. Then begins to decline, while Nano particals percent increase. After that it will be held and agglomerat form of surplus material on applied energy absorption work and dissipate as heat, and thus reduces the power of resilience. Rebound hardness characteristics associated with specific gravity flexible. But note of **fig.(5)** when adding small amounts of modified silica nano gives resilience best value to the lack of conglomerate granules between rubber chains.

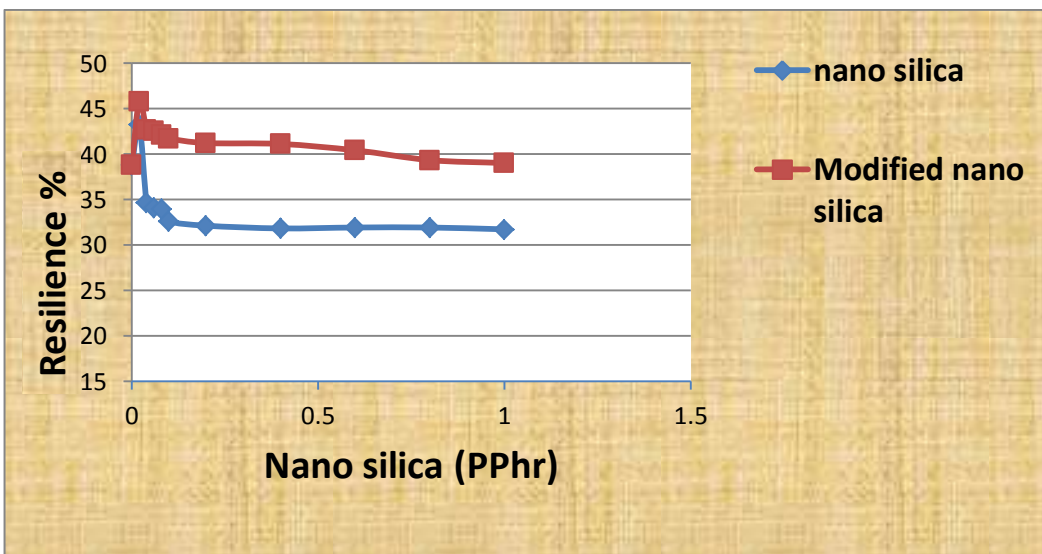


Fig.(5) Effect of nano silica and modified nano silica resilience of recipe

Conclusions:

In this study, the following conclusions are made:

1. High purity silica with the size of the granules (48-91)nm prepared by a chemical process low expensive.
2. Such a silica granules(48-91)nm improved mechanical properties recipe side wall of the tire.
3. This method is useful for the company for the manufacture of tires in the character of the side wall of the tire.
4. When adding small amounts of modified nano silica give good mechanical properties.

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