Effect of Zinc Oxide Level on Tensile Properties of a NR/SBR Composite

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Abstract

The aim of this work is studying the effect of zinc oxide level on tensile properties of the compounds. Since the compounds consist of natural rubber (NR) and styrene-butadiene rubber (SBR) with ratio (50:50).This work included studying the replacing of conventional zinc oxide by nano-zinc oxide as an activator has small particle size and large surface area in comparison with conventional zinc oxide so as to improve tensile properties and reduce the amount of zinc oxide inside the compounds. In this work, two groups of compounds are prepared:

- Six compounds have conventional zinc oxide as an activator with concentrations (0,2,4,5,6,8 phr (part per handred)).
- Nine compounds have nano-zinc oxide as an activator with concentrations (0,0.2,0.4,0.6,0.8,1,1.2,1.4,1.6 phr).
- The compounds were prepared by two-roll mill and laboratory press. Dumbbells (test samples) of tensile test are prepared by Wallace. Test Specimens Cutting Press. Tensile test carried out by Monsanto T10 Tensometer. Crosslink density tested by swelling the samples of the compounds by toluene and using Flory- Rehner equation. The results refer to the maximum values of tensile properties of a NR/SBR blend with conventional zinc oxide at (5 phr) of zinc oxide level. The maximum values of tensile properties with nano-zinc oxide at (1.2 phr) of zinc oxide level. The replacing conventional zinc oxide by nano-zinc oxide reduces the cost of the compounds by reducing the amount of zinc oxide inside the compounds and improves the tensile properties.

Keywords: Tensile Properties, Zinc Oxide, Natural Rubber(NR),and Styrene-Butadien Rubber (SBR).

تأثير اوكسيد الزنك على خواص الشد لمتراكب من المطاط الطبيعي (NR) ومطاط ستايرين-بيوتادين SBR

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الخلاصة

الفكرة الأساسية للبحث هي التعرف على تأثير تركيز اوكسيد الزنك على خواص الشد Tensile properties (قوة الشد Tensile Strength،معامل المرونة Elastic Modulus،والاستطالة Elongation) لعجنات مطاطية مؤلفة من المطاط الطبيعي (NR) والمطاط الصناعي (SBR) بنسب متساوية.وللتعرف على إمكانية استبدال اوكسيد الزنك الاعتيادي باوكسيد الزنك النانوي كمادة منشطة ذات حجم دقائق اصغر ومساحة سطحية اكبر (مقارنة باوكسيد الزنك الاعتيادي) ولغرض استثمار هذه الخواص في تحسين خواص الشد ولتقليل كمية اوكسيد الزنك في العجنات المطاطية. حضرت في هذا البحث مجموعتين من العجنات المطاطية والتي يمكن تصنيفها بالصورة التالية:

- ست عجنات كان العامل المنشط فيها هو اوكسيد الزنك الاعتيادي وقد أضيف للعجنات الست بتراكيز مختلفة هي phr 8,6,5,4,2,0(جزء من مائة من وزن المطاط)).
- تسع عجنات كان العامل المنشط فيها هو اوكسيد الزنك النانوي وقد اضيف للعجنات التسع بتراكيز مختلفة هي (phr1.6 ,1.4,1.2,1,0.8,0.6,0.4,0.2, 0).

أجريت عملية تحضير العجنات باستخدام عصارة ذات رولة مزدوجة ومكابس مختبرية.تم تحضير نماذج فحص الشد باستخدام جهاز(Wallace Test Specimen Cutting Press). أجريت عملية فحص خواص الشد باستخدام جهاز التنسومتر (Monsanto T10 Tensometer). كثافة الترابط (Crosslink Density) تم فحصها لجميع العجنات باستخدام معادلة فلوري-رينر عن طريق فحص الانتفاخ وباستخدام مذيب جيد هو التولوين.

بينت نتائج الفحص ان التركيز الأمثل لمادة اوكسيد الزنك الاعتيادي لعجنات مطاطية مكونة من المطاط الطبيعي (NR) والمطاط الصناعي (SBR) (بنسب متساوية) وللحصول على خواص شد افضل هو (5 phr). كما أشارت الى ان التركيز الأمثل لمادة اوكسيد الزنك النانوي لعجنات مطاطية مكونة من المطاط الطبيعي (NR) والمطاط الصناعي (SBR) (بنسب متساوية) وللحصول على خواص شد افضل هو (1.2phr).ان استبدال اوكسيد الزنك الاعتيادي باوكسيد الزنك النانوي يساهم في تحقيق مجموعة من المميزات ابرزها تحقيق جدوى اقتصادية من خلال تقليل كلف العجنات اضافة الى تحسينه خواص الشد وهذه النتيجة تتضح من خلال مقارنة فحوصات العجنات التي تم تحضيرها لتبين لنا تلك المميزات .

الكلمات المفتاحية:خواص الشد،اوكسيد الزنك ،المطاط الطبيعي (NR)،ومطاط ستايرين بيوتادين(SBR).

1-Introduction

Tensile properties include tensile strength , elongation at break point ,and elastic modulus at determined strain such as at strain equals 300% [1].

Tensile strength of a rubber compound is defined as themaximum tensile stress applied in stretching a specimen of rubber compound to rupture. The relation between tensile strength and the force which is affected on the sample of rubber as:

$TS = F_{BE}/A$ …..1

Where

TS=Tensile strength, the stress at rupture, MPa (Mega Pascal)

$F_{(BE)}$ =The force magnitude at rupture, MN (Mega Newton).

A=cross-sectional area of unstrained specimen, m^2.

Elongation is used to describe the ability of a rubber compound to stretch without breaking. It is equal to the difference between the final and initial lengths expressed as a percentage of the later[1].

The elongation can be represented as: $E=100(L-L_{\circ})/L_{\circ}$ ….2

Modulus at 300% equals to the stress at elongation equals to 300%.

Accelerator complex forming which is one of the main steps in the vulcanization process [2,3,4,5]. This complex reacts with sulphur to form an active sulphurating agent which interacts with allylic sites of the rubber chains to form crosslinks between the adjacent chains of the rubber. Zinc oxide reacts with stearic acid to form zinc stearate and liberate water molecule. The role of zinc ions (Zn^{++}) of zinc stearte in the vulcanization process increases the cure rate and increases the crosslink density. Zinc oxide influences on physical properties such as tensile properties by increasing the crosslink density. Since (tensile strength and modulus) tensile properties increase with increasing the crosslink density. Zinc oxide has negative effect on environment and human . Since the zinc release into the environment from rubber products during production, service life, disposal and recycling as its toxicity has detrimental effect on the living environment and organisms. In 1995, zinc and zinc oxide were placed on the second European list of priority hazard substances in the EU Risk Assessment Programme [6] In 2005,The US Environmental Protection Agency considers ZnO a toxic chemical for aquatic organisms and has detrimental effects on the living environment [7]. Reduction the cost of vulcanized products and negative effect on environment and human of using zinc oxide in rubber industry prompted the researchers to study the reduction of zinc oxide concentration in vulcanized products. In 2007, Sahoo et al. studied The effect of conventional zinc oxide and zinc oxide nano particles(30-70) nm as cure activators for natural rubber (NR) and nitrile rubber (NBR) and compared with conventional ZnO .They found that the tensile strength improved by 80% for NR when ZnO nano-particles used as cure activator instead of conventional ZnO. An improvement of 70% was in the case of NBR[8,9]. In 2009, Wang Jihu studied the effect of nano zinc oxide and conventional zinc oxide on cure characteristics and physical properties of NR/SBR blends. He concluded that tensile properties of blends improved when blends were added 3 phr of nano-ZnO as compared with 5 phr of conventional ZnO [10]. In 2012,G.N.Onyeagoro studied the effect of conventional zinc oxide level on physical properties of NR/NBR/ENR blends with different ratios . He found that zinc oxide concentration of 5.0 phr gave vulcanizates with optimum tensile strength and modulus[11].

2-Experimental part

2-1 Material

Natural rubber (SVR5 produced by Hoa Thuan CO. Vitnam**)** **,**Synthetic rubber (SBR1502, 23% Styrene,77% Butadiene, produced by Kumho CO. Koria),Conventional Zinc Oxide (purity =99%, Particle size=0.5-1µm,and surface area=3-5m^2/gm, produced by ChemTAL Sunnyjoint Chemicals CO. China),and

CTP-100 (produced by Shenyang Sunnyjoint Chemicals CO. China), Toluene produced by (Thomas Baker (Chemicals) PVT. Limited, Mumbai , India).

2-2 Compound recipe

Compound recipe are listed in table(1) with conventional zinc oxide and in table (2) with nano-zinc oxide .

2-3 Equipments

Two-roll mill, Electronic Balance, Laboratory compressor, Tensile test Mold,Wallace Test Specimen Cutting Press, Dial Gauge, and Monsanto T10 Tensometer.

2-4 Mixing Process

The mixing processes carried out in State Company for Tire Industry in Najaf, and according to ASTM D3182. The natural rubber, synthetic rubber and additives materials mixed on laboratory two roll mill (size 15 cm x 30 cm) .Starting temperature was 60^{O}C and the mixing is according to mixing schedule ,which is given in table(3). Total mixing time was kept to minimum to avoid sticking of the rubber compound on the mill rolls.

2-5 Vulcanization process

The vulcanization process carried out in State Company for Tire Industry in Najaf, and according to ASTM D3182.The temperature for preparing the specimens of tensile tests is 150ºC. The pressure of compressor was determined as 3.5 MPa (500 psi) on the cavity areas during vulcanization. The time of vulcanization for the specimen of tensile tests is 45 minutes. The mold of tensile has two cavity sections, and they are similar in dimensions (150x150x2)mm.

2-6 Tensile test

The test carried out at 23±2 ºC and according to ASTM D412 . The dumbbell (test sample) was obtained by cutting the vulcanized sheets by Wallace Test Specimen Cutting Press. The thickness was measured of dumbbell by dial gauge. The dumbbell was placed in two grips of tensometer. The speed of moving grip was determined at 500 mm/minute. Data of the dumbbell (dimensions of dumbbell determined speed 500 mm/minute, number of dumbbell and the date of test) entered into the controlled board of tensometer. The values which are recorded, tensile strength, elongation, modulus at 100%,modulus at200%, and modulus at 300%.Three dumbbells for any compound were tested and took the average of the results**.**

2-7 Crosslink density test

Crosslink density test carried out by swelling method. Since the solvent ,which is used in this method is toluene.

1-the samples was Inserted on the cassette of Densitron for measuring mass density in unit (grams/cm^3).

2- Any sample (with dimensions 2×10×10 mm) was immersed inside 25 ml of toluene in closed bottle at 23º C.

3-The samples were taken out after 7 days. The surfaces were dried with filter paper. They were quickly weighed by using the electronic balance.

4-The specimens were weighed after 6 days at temperature 23º C[12,13,14].

The volume fraction of polymer (elastomer) in the swollen gel at equilibrium was calculated by equation(3) :

$$V_R = \frac{1}{1+\frac{\rho_p}{\rho_t\left(\frac{w_s - w_d}{w_d}\right)}} \qquad \ldots 3$$

Where ρ_p is mass density of sample, and ρ_t is mass density of solvent, W_s is the weight of swollen sample , W_d is the weight of the dried sample, and V_R is volume fraction.

5- Crosslink density (V_e) was calculated by Flory- Rehner equation [13,14].

$$V_e = \left[\ln(1 - V_R) + V_R + X_{NR/SBR} V_R^2\right] / 2V_\circ\left(V_R^{\frac{1}{3}} - \frac{1}{2} V_R\right) \qquad \ldots(4)$$

Where

V_e = Crosslink density (mole / cm^3).

$V_\circ$ = Molar volume of toluene =106.3 cm^3/mole.

$X_{NR/SBR}$=Parameter characteristic of interaction between the elastomer network and the swelling agent. This parameter was given by the average of the parameter characteristic of interaction for natural rubber(NR)-toluene and the parameter for synthetic rubber(SBR)-toluene with respect the ratio of weight of any rubber type to the total weight of all types of rubber in the compound [15].

X_{NR} for Natural rubber system –Toluene obtained by equation (5).

$$X_{NR}=0.44 + 0.18V_R \quad \ldots(5)$$

Where V_R is volume fraction. X_{SBR} for Styrene-Butadiene rubber -Toluene system was given by the equation (6) [16]:

$$X_{SBR} = 0.33 + 0.43V_R \quad \ldots(6)$$

The value of X_{NR+SBR} for blend of NR-SBR with ratio 50:50 is equal to average of X_{NR} and X_{SBR}.

$$X_{NR/SBR} = \frac{0.44+0.33}{2} + \frac{0.18+0.43}{2} V_R \quad \ldots(7)$$

$$X_{NR/SBR} = 0.385+0.305\, V_R \quad \ldots(8)$$

Table(1) Compound Recipe of (A) group with Conventional Zinc Oxide.

Material	A1	A2	A3	A4	A5	A6
SVR5	50	50	50	50	50	50
SBR1502	50	50	50	50	50	50
Conventional ZnO	0	2	4	5	6	8
Stearic acid	2	2	2	2	2	2
TMQ	1	1	1	1	1	1
6PPD	1.5	1.5	1.5	1.5	1.5	1.5
Carbon black N660	50	50	50	50	50	50
Paraffinic oil	10	10	10	10	10	10
Iraqi kaolin	0	0	0	0	0	0
CBS	0.75	0.75	0.75	0.75	0.75	0.75
Sulfur	3	3	3	3	3	3
CTP-100	0.2	0.2	0.2	0.2	0.2	0.2

Table (2) Compound Recipe of (B) group with Conventional Zinc Oxide.

Material	B1	B 2	B 3	B 4	B 5	B 6	B 7	B 8	B 9
SVR5	50	50	50	50	50	50	50	50	50
SBR1502	50	50	50	50	50	50	50	50	50
Nano-ZnO	0	0.2	0.4	0.6	0.8	1	1.2	1.4	1.6
Stearic acid	2	2	2	2	2	2	2	2	2
TMQ	1	1	1	1	1	1	1	1	1
6PPD	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Carbon black N660	50	50	50	50	50	50	50	50	50
Paraffinic oil	10	10	10	10	10	10	10	10	10
CBS	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.75
Sulfur	3	3	3	3	3	3	3	3	3
CTP-100	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2

3 Results and Discussion

The results of tensile test are listed in table (4) and table (5). Since table (4) has results of A group with conventional zinc oxide, and table (5) has results of (B) group with nano-zinc oxide.

3-1 Tensile strength

According to the curve of (A) group of the figure (1) and the table(4), the tensile strength increased progressively with increasing the conventional zinc oxide level from (0 phr) up to a maximum value at (5 phr).This observation is attributed to consolidation of network structure of the rubber chains with increasing ZnO level, this results is due to increase in crosslink density. But tensile strength decreases with increasing the zinc oxide level after(5 phr). This result is due to the relation between zinc oxide and crosslink density. The crosslink density is proportional to the concentration of zinc oxide. When the crosslink density increases, the elastomer becomes more elastic and the tensile strength increases and passes through a maximum as crosslink density (at 5phr) . When the crosslink density increases (after 5phr),the motion of rubber chains becomes restricted, and the tight network is incapable of dissipating much energy. This result causes the brittle fracture at low elongation, therefore tensile strength decreases with increasing zinc oxide level after (5phr) [17]. According to the curve of (B) group of the figure (2)and the table (5) , nano-zinc oxide increases tensile strength , and the maximum value of tensile strength at (1.2 phr).When compared the curve of (B) group of the figure (2), and the curve of (A) group of the figure (1),the tensile strength with (1.2 phr) of nano-zinc oxide is (16.59 Mpa), and is greater than tensile strength with (5phr) of conventional zinc oxide (16.097Mpa). This result is due to high surface area and dispersion of nano-zinc oxide in comparing with conventional zinc oxide.

3-2 Modulus at 300%

According to the curve of (A) group of the figure (3) and the table (4). The modulus at 300% increased progressively with increase in conventional zinc oxide level from (0 phr) up to a maximum value at (5 phr). This observation is attributed to consolidation of network structure of the rubber chains with increasing ZnO level, this result is due to increase the crosslink density. But the modulus at 300% decreases with increasing zinc oxide level after (5 phr).This result is due the relation between zinc oxide and crosslink density and the negative effect of increasing the crosslink density on the elasticity of the compound after (5phr) of the concentration of conventional zinc oxide which is explained in 3-1.According to the curve of (B) group of the figure (4) and the table (5). The modulus at 300% increased progressively with increase in nano-zinc oxide level from (0 phr) up to a maximum value at (1.2 phr). This observation is attributed to consolidation of network structure of the rubber chains with increasing ZnO level, this result is due to increase the crosslink density. But the modulus at 300% decreases with increasing the concentration of zinc oxide level after (1.2 phr). This result is due the relation between zinc oxide and crosslink density and the negative effect of increasing the crosslink density on the elasticity of the compound after (1.2phr) of the concentration of nano-zinc oxide. When compared the curve of (B) group of the figure (4),and the curve of (A) group of the figure (3),the modulus with (1.2 phr) of nano-zinc oxide is (11.356 Mpa), and is less than the modulus with (5 phr) of conventional zinc oxide (11.5 Mpa).

3-3 Elongation at break

According to the figures [5,6],and the tables (4,5), the elongation at break decreases with increasing zinc oxide level. This observation is attributed to increase in crosslink density with increasing of zinc oxide level. The increasing of crosslink density reduces the molecular chain mobility**.**

3-4 Crosslink density

According to figures [7,8], the crosslink density increases with increasing zinc oxide level. The increasing of crosslink density is due to the increasing of zinc ion (Zn^{+2}) ,which is shorten the crosslink and generate new crosslink precursors during the vulcanization process so as to increase the extent of crosslinking [1,6,16]. When compared the curve of (B) group of the figure (8) and curve of (A) group of the figure (7),the crosslink density with nano-zinc oxide level (0-1.6 phr) is less than the crosslink density with conventional zinc oxide level (0-8 phr). Although the crosslink density with nano-zinc oxide is less than the crosslink density with conventional zinc oxide ,but when compared the curve of (B) group and (A) , tensile strength with nano-zinc oxide is greater than tensile strength with conventional zinc oxide according to the figures (1) and (2), the modulus with nano-zinc oxide is approximately equals the modulus with conventional zinc oxide according to the figures (3) and (4)

Table (3) Mixing Schedule

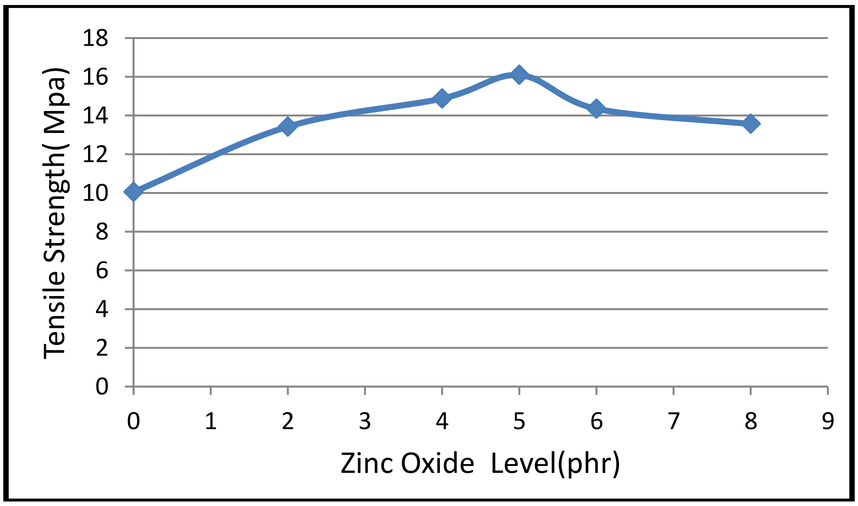
NO	Operations	Time (minutes)
1	Mastication of NR and SBR rubbers	4
2	Addition of Zinc Oxide	2
3	Stearic acid and antidegradants (TMQ) and 6PPD	2
4	Addition of Carbon black + Oil	4
5	Addition of Carbon black	3
7	Sulphur, CBS, CTP-100	3
8	Sweep and dumb	2
9	Total	20

Co	Tensile strength (Mpa)	Tensile strength Rate (Mpa)	Elongation (%)	Elongation Rate (%)	Modulus at 300 (Mpa)	Modulus at 300 Rate (Mpa)
A1	9.655 9.808 10.678	10.047	442 438 475	452	6.437 6.749 7.319	6.835
A2	12.539 14.136 13.621	13.432	433 410 395	413	9.990 9.670 10.050	9.903
A3	15.1 14.65 14.905	14.885	407 412 388	402	10.880 10.650 10.340	10.62
A4	16.43 16.01 15.85	16.097	338 375 360	358	11.760 11.530 11.210	11.5
A5	14.503 14.314 14.253	14.357	367 343 351	354	10.040 9.690 9.910	9.88
A6	13.82 13.5 13.65	13.567	322 324 344	330	7.100 8.570 7.880	7.85

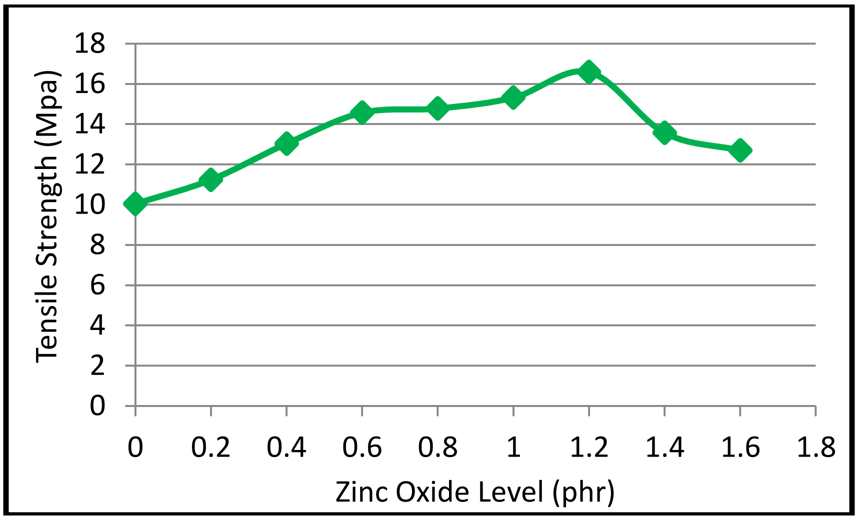
Table (4) Results of Tensile Properties with Conventional Zinc Oxide.

Table (5) Results of Tensile Properties with Nano- Zinc Oxide.

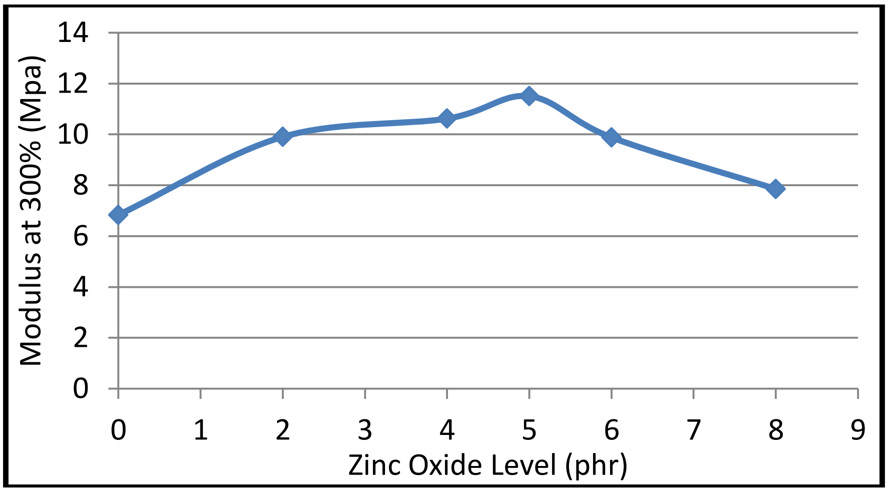
Co	Tensile strength (Mpa)	Tensile strength Rate (Mpa)	Elongation (%)	Elongation Rate (%)	Modulus at 300 (Mpa)	Modulus at 300 Rate (Mpa)
B1	9.655 9.808 10.678	10.047	442 438 475	452	6.437 6.749 7.319	6.835
B2	12.947 11.547 9.211	11.235	435 428 418	427	7.399 7.794 8.019	7.737
B3	11.997 14.074 13.037	13.036	417 427 420	421	6.960 8.626 8.933	8.173
B4	14.457 14.697 14.550	14.568	413 403 378	398	9.380 8.667 8.656	8.901
B5	15.044 14.907 14.400	14.784	390 411 388	396	8.518 10.018 8.693	9.076
B6	15.823 14.720 15.431	15.325	389 380 407	392	10.456 9.786 10.376	10.206
B7	16.648 16.259 16.863	16.59	371 357 375	368	12.035 11.546 10.486	11.356
B8	14.156 12.109 14.446	13.57	382 364 351	366	7.667 7.440 7.869	7.659
B9	12.989 14.730 10.366	12.695	361 354 370	362	6.831 8.138 7.845	7.605



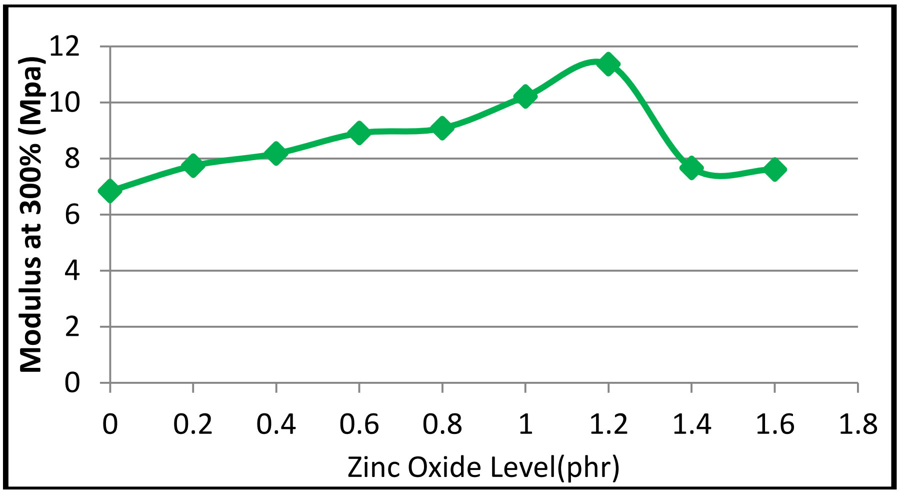
Fig(1) Dependence of Tensile Strength on Conventional Zinc Oxide level for (A) groups of compounds.



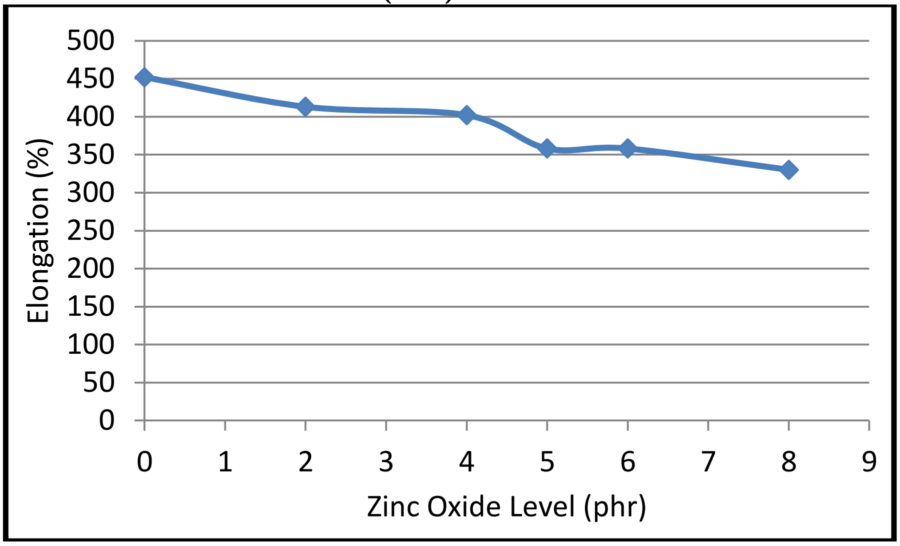
Fig(2) Dependence of Tensile Strength on Nano- Zinc Oxide level for (B) groups of compounds**.**



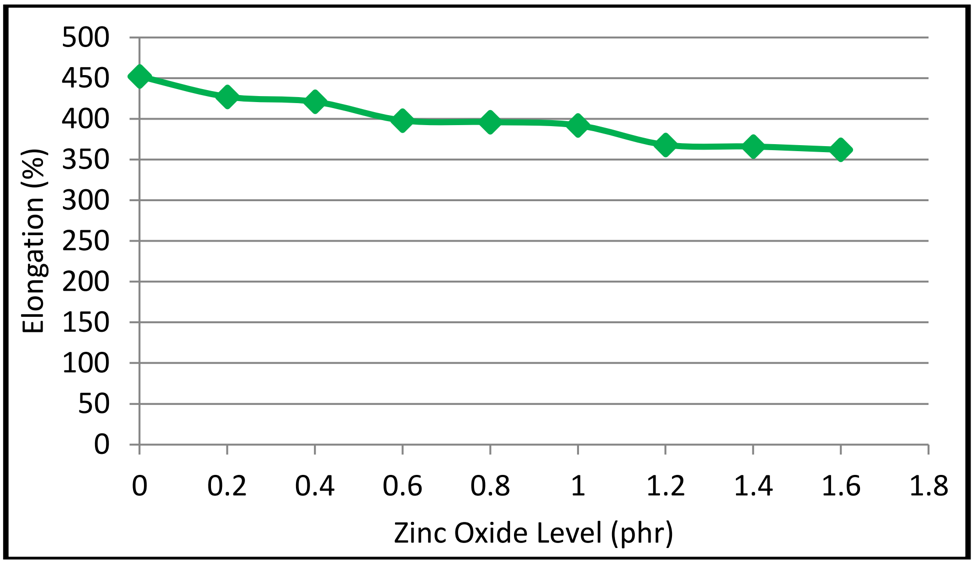
Fig(3) Dependence of Modulus at 300% on Conventional Zinc Oxide level for (A) group of compounds.



Fig(4) Dependence of Modulus at 300% on Nano- Zinc Oxide level for (B) group of compounds.



Fig(5) Dependence of Elongation at break on Conventional Zinc Oxide level for (A) group of compounds.



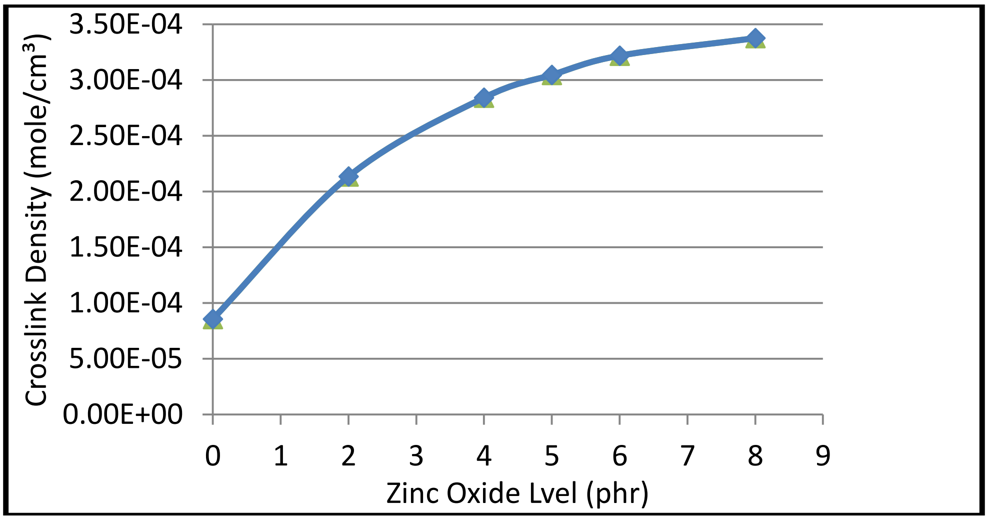
Fig(6) Dependence of Elongation at break on Nano- Zinc Oxide level for (B) group of compounds.

Table(6) Results of Swelling test for (A) group with Conventional Zinc Oxide.

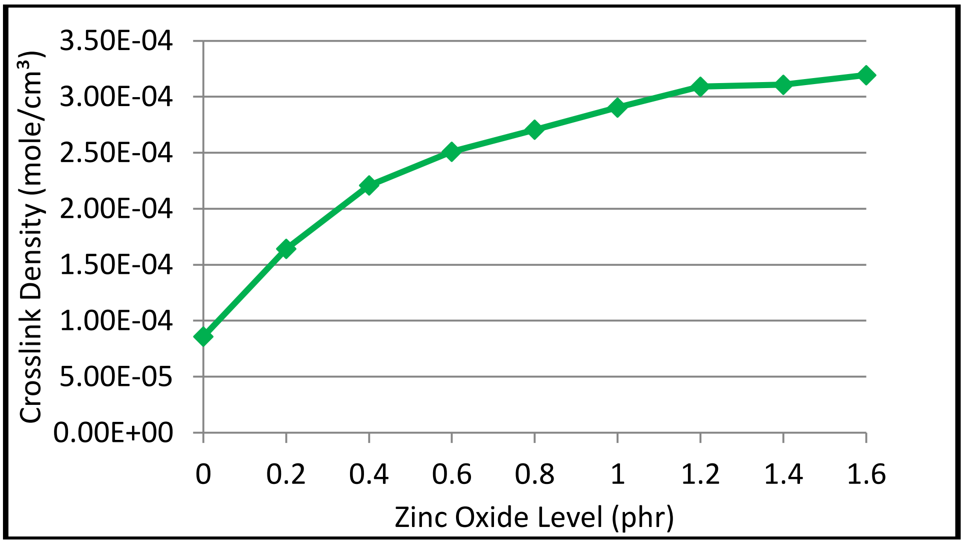
Co	Density (gm/cm^3)	W_s (gm)	W_d (gm)	Volume fraction	Crosslink density ($mole/cm^3$)
A1	1.085	3.7131	1.3439	0.2546	$8.578X10^{-5}$
A2	1.098	2.9803	0.9423	0.3719	$21.352\ X10^{-5}$
A3	1.095	3.725	1.3144	0.4129	$28.426\ X10^{-5}$
A4	1.109	3.8081	1.643	0.4231	$30.472\ X10^{-5}$
A5	1.117	2.9688	0.9696	0.4312	$32.195\ X10^{-5}$
A6	1.129	3.366	1.3009	0.4382	$33.762\ X10^{-5}$

Table (7) Results of Swelling test for (B) group with Nano- Zinc Oxide.

Co	Density (gm/cm^3)	W_s (gm)	W_d (gm)	Volume fraction	Crosslink density ($mole/cm^3$)
B1	1.085	3.7131	1.3439	0.2546	8.578 $X10^{-5}$
B2	1.094	3.0764	1.0523	0.3358	16.41 $X10^{-5}$
B3	1.092	3.8007	1.1662	0.3767	22.083 $X10^{-5}$
B4	1.096	3.5422	1.1662	0.3949	25.103 $X10^{-5}$
B5	1.089	3.2873	1.0646	0.4058	27.06 $X10^{-5}$
B6	1.012	3.8602	1.1628	0.416	29.037 $X10^{-5}$
B7	1.093	3.4746	1.0097	0.4252	30.909 $X10^{-5}$
B8	1.097	2.9819	0.9664	0.4259	31.07 $X10^{-5}$
B9	1.101	2.9144	0.9491	0.43	31.938 $X10^{-5}$



Fig(7) Dependence of Crosslink density on Conventional Zinc Oxide level for (A) group of compounds.



Fig(8) Dependence of Crosslink density on Nano- Zinc Oxide level for (B) group of compounds.

4-Conclusions

1-Conventional zinc oxide gives the NR/SBR blend optimum tensile properties (tensile strength and modulus at300%) at (5phr) of zinc oxide level.
2-Nano-zinc oxide gives the NR/SBR blend optimum tensile properties (tensile strength and modulus at300%) at (1.2phr) of zinc oxide level.
3-Tensile strength with nano-zinc oxide at (1.2 phr) is greater than tensile strength with conventional zinc oxide at (5 phr).
4-Modulus at300% with nano-zinc oxide at (1.2 phr) is less than tensile strength with conventional zinc oxide at (5 phr).
5-Elongation at break decreases with increasing zinc oxide level.
6-Crosslink density increases with increasing zinc oxide level.

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