



## Evaluation of Thermal and Mechanical Properties of Crumb/ Natural Rubber Nanocomposites



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ALTHOUGH crumb rubber or waste tires are not classified as hazardous waste, they can cause environmental pollution if disposed incorrectly or irresponsibly. Waste tires can be put to many beneficial uses from one side and to protect the environment via limiting its accumulation when reaching their end of life from the other side. Shredded tires can be added to rubbers to be used in making school playgrounds, equine bedding and football pitches. This may result in the reduction of the mechanical properties of the composites which can be kept by the use of higher levels of other fillers in addition to crumb and hence keeping and even more enhancing these mechanical properties. The crumb rubber was added to natural rubber in the ratio of 10:100 by weight with the partial removal of the carbon black, from 60 to 20 phr, by montmorillonite (organo-modified nanoclay). The modified nanoclay was added in different loadings; 0, 2.5, 5, 7.5, and 10 phr to the rubber mix formulations. Results of morphological analysis revealed the good miscibility and homogeneity between crumb and Natural rubber in one hand and all other ingredients in the other hand. The mechanical, thermal measurements the role of OMMT in enhancing the mechanical and thermal properties of the crumb/NR nanocomposites. The addition of crumb rubber reduces the hazardous working environments for personnel, lower the production cost by making a good use of waste tires and presents a green solution for getting rid of an environmental problem.

**Keywords:** Crumb rubber, Nanoclay, SEM, Mechanical properties, TGA, Thermal conductivity, SEM

### Introduction

Used tires are a major problem in Egypt. Used tires and end-of-life tires are tires that have completed their functional life and cannot be used on automobiles. It has been estimated that around one billion tires are withdrawn from use in the world every year. Since tires are made of vulcanized rubbers which do not decompose easily, disposable scrap tires is a serious environmental problem. Until recently, they were either buried with other industrial waste in landfill sites or stockpiled in huge dumps that could easily contain millions of tires. Scrap tires are valuable and viable resource; the challenge today is to find best way to utilize them in a wide range of applications. Development of technologies for recycling waste tires, which are acceptable from the environmental standpoint, and

cost-effective, has proven to be a difficult challenge due to complexities inherent in the reuse of tires. Establishing optimal processes for the reuse/recycling of scrap tires thus remains a worldwide challenge as we enter the new century [1].

Currently, there are several means of recovery: reuse, rethreading, recycling, landfill engineering and energy recovery. The scientific community's efforts in finding ways to reduce tire waste have led to intense research on rubber that includes the possibility of applying it in concrete [2-4], filler in natural rubber vulcanizates [5], and blends with polymers [6,7]. Sometimes, in order to achieve the suitable characteristics for its application, rubber waste must be pre-treated with chemicals as  $H_2SO_4$ ,  $HNO_3$ ,  $H_2SO_4/HNO_3$  [8] or grafted as in case of natural rubber [9].

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The inherent problems associated with the recycling of rubber products have resulted in the development of various methods to reuse elastomers. Attempts to recycle rubber for redesigning in its primary function include reclaiming, oxidative decoupling of rubber scrap [10] the use of microwave [11] and combination of biological and microwave treatment [12]. One of the promising alternatives to utilize waste tire rubber is the preparation of thermoplastics elastomers. Scrap rubber, namely waste tire dust, WTD, can be blended with thermoplastics such as ethylene vinyl acetate (EVA) to produce thermoplastic elastomers (TPEs) with a range of properties. Besides having physical properties of both, thermoplastics and elastomer and processability similar to that of thermoplastics; TPEs provides better material utilization, as scrap and rejects can be recycled. Replacement of TPEs virgin components with recycled polymers is very important from economic and ecological standpoints. Unfortunately, direct introduction of WTD into recipes of different polyolefin/rubber TPEs results in a significant decrease in their tensile properties due to the poor interfacial adhesion between the blend components [13-18]

As an approach to overcome this problem, researchers incorporated a number of different modification techniques such as recycle rubber particle size reduction, varying compatibilizing techniques [19,20] and performing oxidation treatments on the surface of waste rubber [21]. Ratnam *et al* [22] reported the effect of gamma irradiation of WTD on the mechanical properties and compatibility of EVA/WTD blends. The tensile strength, elongation at break percent, E%, and hardness of the EVA/WTD blend found an increase with gamma irradiation dose. This observation has been attributed to the improved compatibility of the blend upon pre irradiation of WTD. From the discussion of the results, it reveals that the compatibilization of EVA/WTD through pre-irradiation of WTD involves radiation-induced chain scissions which lead to break down of crosslinked network in WTD. The enhancement in the compatibility of EVA/WTD blend upon pre-irradiation of WTD was confirmed from the dynamic mechanical analysis and morphological examination of EVA/WTD blends. This progressive increase in E% with simultaneous decreases in impact strength of EVA/WTD blend with irradiation dose further revealed the devulcanization of WTD upon gamma irradiation.

In our present work, untreated ground crumb rubber was used as an additive to natural rubber formulations in presence of different concentrations of organo-modified nanoclay and the effect of nanoclay loading on the thermal and mechanical properties of crumb/NR was investigated.

## **Experimental**

### *Materials*

Natural rubber used in this study is SMR-20, supplied by Tecnopolimeri srl., Russia. This grade has good processing characteristics and physical properties. Its low viscosity and easier mixing characteristic, compared with RSS grades, can considerably reduce the mastication and mixing period. Nanoclay was of the organo-montmorillonit (OMMT) type whose particle size  $\leq 10$  micron and specific gravity of 200-500 kg/m<sup>3</sup>. Crumb rubber was obtained from a local manufactory and its particle size was 100  $\mu$ . All other ingredients were purchased from Transporting & Engineering Co., Egypt.

### *Preparation of crumb/NR nanocomposites*

The compositions used in this study were obtained by using a two-roll mill according to the formulations presented in Table (1). The preparation techniques have been described in details according to ASTM D 3182-16 procedures. The compositions were vulcanized at 152 °C. A master batch was prepared by adding 10 % Crumb rubber to 90 % NR. The nanoclay was then mixed in variable concentrations with crumb/NR rubber compounds. The carbon black and the remaining compounding ingredients, except the curative package, were then added and mixed under the above mentioned procedures. Finally, the curatives were subsequently added to the crumb/NR nanocomposites.

### *Rheology Measurements*

The cure characteristics of the rubber mixes were determined at 152 °C according to the technical procedures of the ASTM D 2084-17. The cure characteristics were measured using the oscillating disc rheometer, MRD 2000, Alpha Technology, UK. The test specimen was inserted into the cavity of the cure meter and cured automatically in a die cavity. The bioconical disc is imbedded in the sample and is oscillated through a small cavity at angle 3°. The force required to oscillate or rotate the disk to maximum amplitude was continuously recorded as a function of time, with the force being proportional to the shear modulus (stiffness) of the test specimen.

### Mechanical Testing

To study the stress-strain behavior of rubber materials, a Zwick Tensile Testing Machine is used. For this purpose, samples were cut from molded sheets into dumb bell shape with the dimensions of 150×150×2 mm. Tensile strength, Elongation Modulus and Elongation at break percent are measured according to ASTM D 412-16.

### Hardness Testing

Hardness is a property of considerable importance, usually included in rubber specifications along with the tensile properties. It is a simple way of obtaining a measure of elastic modulus of a rubber by determining its resistance to a rigid indenter to which a force is applied. Hardness testing measurements were carried out according to ASTM D 2240-15. Hardness determinations were made using a Zwick Hardness Tester 3150, Germany. The results were expressed as International Rubber Hardness Degree (IRHD) from 0 to 100 scale called Shore A. Shore-A can be translated in terms of force applied to the indenter with respect to the following equation:

$$F = 56 + 7.66 \quad (1)$$

Where,  $H_A$  is the hardness in shore-A units and  $F$  is the force applied in grams. Test specimens used were cylindrical in shape whose thickness and diameter were  $6.00 \pm 0.2$  mm and  $14.00 \pm 0.2$  mm respectively. Average values and statistical data were taken over 10 samples for each mix.

### Scanning Electron Microscopy SEM

This analysis was performed to study the surface of NR composite and that of crumb/NR nanocomposite layers. The SEM micrographs of surface were examined at various magnifications using an Inspect S Machine (FEI Company, Holland) equipped with an energy dispersive

X-ray analyzer (EDAX). The samples used to conduct SEM observation are bonded on the sample holders with conducting glue carbon. The morphologies of the final products in samples are observed at a microscopic level using SEM.

### Thermal Gravimetric Analysis TGA

Thermal Gravimetric Analysis (TGA) of all crumb/NR nanocomposites studies were carried out using Shimadzu-50 Thermogravimetric Analyzer in presence of air at a rate of 10 °C/min, using temperature range of 25 to 650 °C. Degradation temperature of the composites was studied through this analysis.

### Thermal Conductivity

Thermal conductivity measures the ability of a material to conduct heat, and is defined as the quantity of heat ( $Q$ ) transmitted through a unit thickness ( $L$ ) in a direction normal to a surface of unit area ( $A$ ) due to a unit temperature gradient ( $\Delta T$ ) under steady state conditions and when the heat transfer is dependent only on the temperature gradient. If a flat sample is placed between two flat isothermal plates maintained at two different temperatures, and a uniform one-dimensional temperature field has been stabilized, the temperature field in the sample should be uniform within all the sample's volume (size of the plates is supposed to be much larger than thickness of the sample). The temperature gradient can be determined by measurements of the temperature difference between the two hot and cold plates ( $\Delta T = T_{\text{hot}} - T_{\text{cold}}$ ) and thickness of the sample  $\Delta x$ , because in this case the average temperature gradient  $dT/dx$  is equal to  $(-\Delta T/\Delta x)$ . This test was carried out on samples of 30×30×5 cm using Laser Comp instrument and according to ASTM C-518. This test can be described as follows; the sample is placed between two plates,

TABLE 1. Rubber formulations and ingredients

Mix Symbol	B <sub>r</sub>	B1	B2	B3	B4	B5
		Ingredients				
NR	100	90	90	90	90	90
Crumb rubber	0	10	10	10	10	10
Nanoclay	0	0	2.5	5	7.5	10
ZnO	5	5	5	5	5	5
Stearic Acid	2	2	2	2	2	2
6 PPD	1	1	1	1	1	1
MBT	0.5	0.5	0.5	0.5	0.5	0.5
TMTD	1.5	1.5	1.5	1.5	1.5	1.5
Carbon Black	60	20	20	20	20	20
Sulfur	2.5	2.5	2.5	2.5	2.5	2.5

6PPD: N-(1,3-Dimethylbutyl)-N'-phenyl-p-phenylenediamine, MBT: 2-mercaptothiazole, TMTD: Tetramethylthiuram Disulfide

the first one is a flat electrical heating plate and the other one is flat electrical cooling plate. All the information about the sample is recorded and the test started. It may be 4 to 5 hours till reaching the conditions of steady state. Through this time the software store the temperature of the hot plate and the cooled plate and the heat flux through the samples. Finally the thermal conductivity was calculated using the following Equation;

$$K = \Phi \Delta x / \Delta T \quad (2)$$

Where;  $\Phi$  is heat flux in  $W/m^2$  flowing through the sample,  $K$  is its thermal conductivity ( $W m^{-1} K^{-1}$ ),  $\Delta x$  is the sample thickness in m,  $\Delta T$  is the temperature difference between the hot and cold surfaces of specimen expressed in  $^{\circ}C$ .

## Results and Discussion

### Rheometric Characterization

Table (2) shows the rheometric parameters of crumb/NR nanoclay vulcanizates with different loadings. The rheometric characteristics such as minimum torque ( $M_L$ ), maximum torque ( $M_H$ ),  $\Delta M$ , delta torque (difference between maximum torque and minimum torque), optimum cure time ( $t_{90}$ ) and cure rate index (CRI) were recorded.  $M_L$  is a measure of stiffness of the unvulcanized rubber nanocomposites taken at the lowest point of the cure curve. It can be noticed from Table (2) that the minimum torque increased with increasing the nanoclay loadings reaching a maximum value that exceeds that of the reference rubber composites. This is because of the viscosity increase due to crosslink formation before curing.  $M_H$  is a measure of stiffness or shear modulus of the fully vulcanized rubber nanocomposites at the vulcanization temperature. A remarkable increase in  $M_H$  can be seen as a result of nanoclay increments to crumb/NR nanocomposites.

This increase in  $M_H$  which exceeds that of the reference crumb/NR reference mix, is due to more crosslinks formation; the higher the  $M_H$  value, the higher the crosslink density [23]. Delta torque is a direct indication of the effect of adding different nanoclay concentrations on the torque of the vulcanizate at the curing temperature. Table (2) describes this relationship clearly.  $\Delta M$  increases continuously with increasing the content of nanoclay. These differences represent the amounts of torque developed during curing and they are directly proportional to the increase in crosslinking in the rubber nanocomposites during curing as a result of adding nanoclay in different loadings. The delta torque is an indirect indication of the degree of crosslinking of the rubber vulcanizates [24]. The effect of adding nanoclay in different amounts on the optimum cure time of the crumb/NR nanocomposites is expressed from the cure time data of the whole series. The first addition of nanoclay (2.5g) reflects a sharp decrease in the  $t_{90}$  that is at a much lower level than that of the reference rubber composite. This behavior continues to decrease -with a lower rate- with further increase in nanoclay. These results can be explained on the basis of the good interactions and interfacial adhesion between the nanoclay and the rubber matrix. The speedy vulcanizing reaction in the nanocomposites was activated by the large surface area of the nanoclay and hence leads to lowering of the curing temperature [25].

Table (2) indicates also a high CRI values for crumb/NR nanocomposites and shows higher vulcanization rate as a result of nanoclay introduction in the crumb/NR mix. This may be attributed to the nanoclay acts as an effective crosslinking agent for SBR/EPDM nanocomposites and it is leading a substantial increase in the rubber vulcanization rate

TABLE 2. Rheological Parameters of crumb/NR nanocomposites

Mix	$B_R$	$B_1$	$B_2$	$B_3$	$B_4$	$B_5$
Min. Torque, $M_L$	1.26	1.01	1.05	1.13	1.21	1.29
Max. Torque, $M_H$	14.10	8.54	10.21	12.81	13.95	14.50
Torque Diff., $\Delta M$	12.84	7.53	9.16	11.68	12.74	13.21
Scorch time, $T_{S2}$	2.21	3.70	1.91	1.85	1.79	1.73
Cure Time, $T_{90}$	5.6	6.94	4.19	3.89	3.80	3.68
Cure Rate Index, CRI	28.58	30.86	43.86	49.02	49.75	51.28

[26]. In fact, the amino groups existing in the nanosilicate structure, which originate from the organophilization of the nanoclay, ease the curing reaction of neat rubber [27].

#### Mechanical Properties

The mechanical properties of crumb/NR nanocomposites, such as toughness, impact strength, elongation at break, etc., determine their potential applications by stress-strain behavior. The measurement of tensile strength, elongation modulus and elongation percent at break in addition to hardness are given. From figure (1), it can be observed that the tensile strength increases with the increase of nanoclay loading. The addition of 2.5, 5, 7.5 and 10 phr of nanoclay to the control crumb/NR compound clearly improves tensile strength from 3.13 in the control rubber to 10.44 Mpa in rubber nanocomposite containing 10 phr which exceeds that of the reference sample, representing 333 % increase in the tensile strength approximately.

The elongation modulus measured of all the crumb/NR nanocomposite vulcanizates are represented in Figure (2). E-Modulus of crumb/NR nanocomposites showed a slight decrease with increasing concentration of nanoclay to 2.5 phr followed by an increase with further OMMT loading addition to reach its maximum value for the crumb/NR nanocomposite containing 10 phr. This increase in E-Modulus may be for one reason; the increase in the crosslink density of the nanocomposites at higher packing level of nanofiller. However, this increase still does not exceed the value of the E-Modulus recorded by the control sample. These results can be explained on the basis of results obtained by morphology studies showing that the increase in nanoclay loadings revealed more dispersion on the clay and all other ingredients and giving rise to better miscibility of ingredients and homogeneity in the nanocomposite matrix. In low loading region, reinforcement is rising with organoclay incorporation.

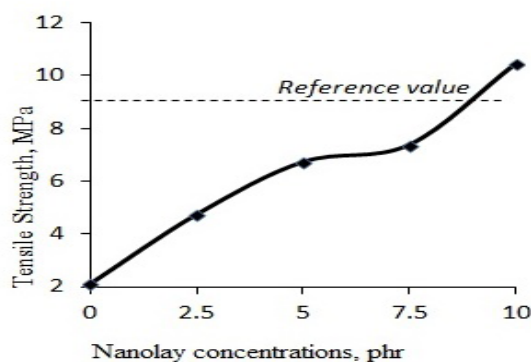


Figure (1): Effect of adding different concentrations of nanoclay on the tensile strength of crumb/natural nanocomposites.

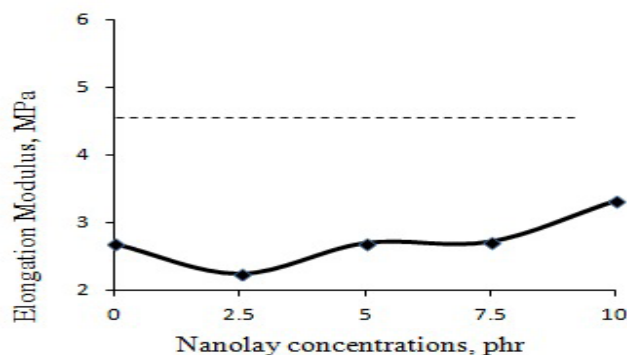
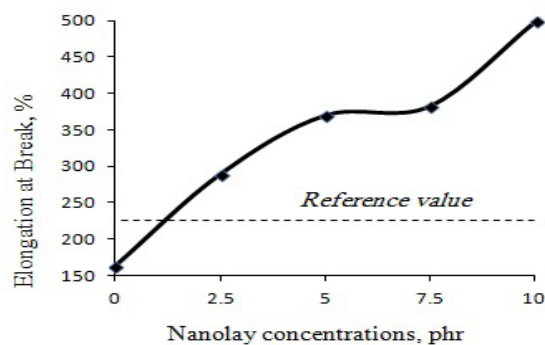


Figure (2): Effect of adding different concentrations of nanoclay on the Elongation Modulus of crumb/natural nanocomposites





**Figure (3): Effect of adding different concentrations of nanoclay on the Elongation at break % of crumb/natural nanocomposites**

#### Scanning Electronic Microscopy SEM

The SEM micrographs are shown in Figure (2). It is clear that the control sample ( $B_1$ ) showed a smooth surface indicating a complete miscibility of the NR and crumb rubber. The addition of 2.5 and 5 phr of OMMT lead to a surface roughness as shown in samples  $B_2$  and  $B_3$  respectively. However at higher concentration 7.5 and 10 phr, as shown in samples  $B_4$  and  $B_5$  respectively, the samples surface became smoother indicating a one phase rubber blend. It seems that at higher concentrations of OMMT the nanofiller form a dispersible network within the rubber blend matrix which enhances the rubbers miscibility with each other.

#### Thermogravimetric Analysis TGA

High temperature Thermal Analysis (50-650°C) curves for the sample are shown in Table (4). The temperature for the onset of degradation is the temperature at which 10% degradation occurred ( $T_{10}$ ), the temperature at which 50% degradation occurred ( $T_{50}$ ) and the temperature at which 90 % degradation occurred ( $T_{90}$ ) were calculated.

From the Table (3), it can be seen that, at low nanoclay concentrations, crumb/NR nanocomposite samples reveal lower onset degradation temperature levels than that of the control composite. The onset degradation temperature was low for mix containing no nanoclay ( $B_1$ ) and it showed a steady increase in its value with leveling up the nanoclay concentration. For  $T_{50}$  and  $T_{90}$  temperature values, they both show similar behavior. Thus, this effect was enhanced with further increase of nanoclay in the crumb/NR nanocomposites showing that the addition of nanoclay to crumb/NR enhances its

thermal stability. This increase can be attributed to the increase in the crosslink density. The crosslinking increases the rigidity of the rubber mix, which in turn increases the thermal stability [28,29]. This proves that increasing loading of nanoclay is responsible for the increase in thermal stability.

#### Thermal Conductivity

Table (5) shows the results of the thermal conductivity of crumb/NR nanocomposites containing 0.0 and 10.0 phr of organo-modified nanoclay  $B_1$  and  $B_5$ , respectively in addition that of polystyrene. Polystyrene is usually used and added to construction materials among other ingredients to increase their heat resistance. Its thermal conductivity ranges from 0.32 to 0.038 W/Mk. The thermal conductivity of these samples was measured according to ASTM C-518. The measured thermal conductivity of these samples varied between 0.137 and 0.144 W/Mk, for samples without nanoclay and with nanoclay, respectively. Compared to polystyrene insulation materials, crumb/NR rubber with or without nanofiller shows very promising thermal insulation properties. To be fair enough, the addition of nanoclay enhances the thermal insulation of crumb/NR nanocomposites to reach the value of the control composite.

#### Conclusion

Morphological analysis (SEM) showed that composites containing crumb rubber can be added to NR and its porphology was enhanced with increasing the nanoclay loadings. Nanoclay forms a dispersible network within the rubber blend matrix which improves the rubber miscibility with each other. Rheogical results declared that the more nanoclay included to crumb/

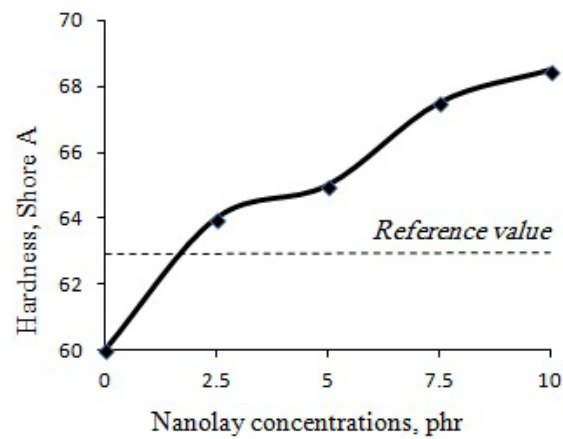


Figure (4): Effect of adding different concentrations of nanoclay on Hardness (Shore A) of crumb/natural nanocomposites

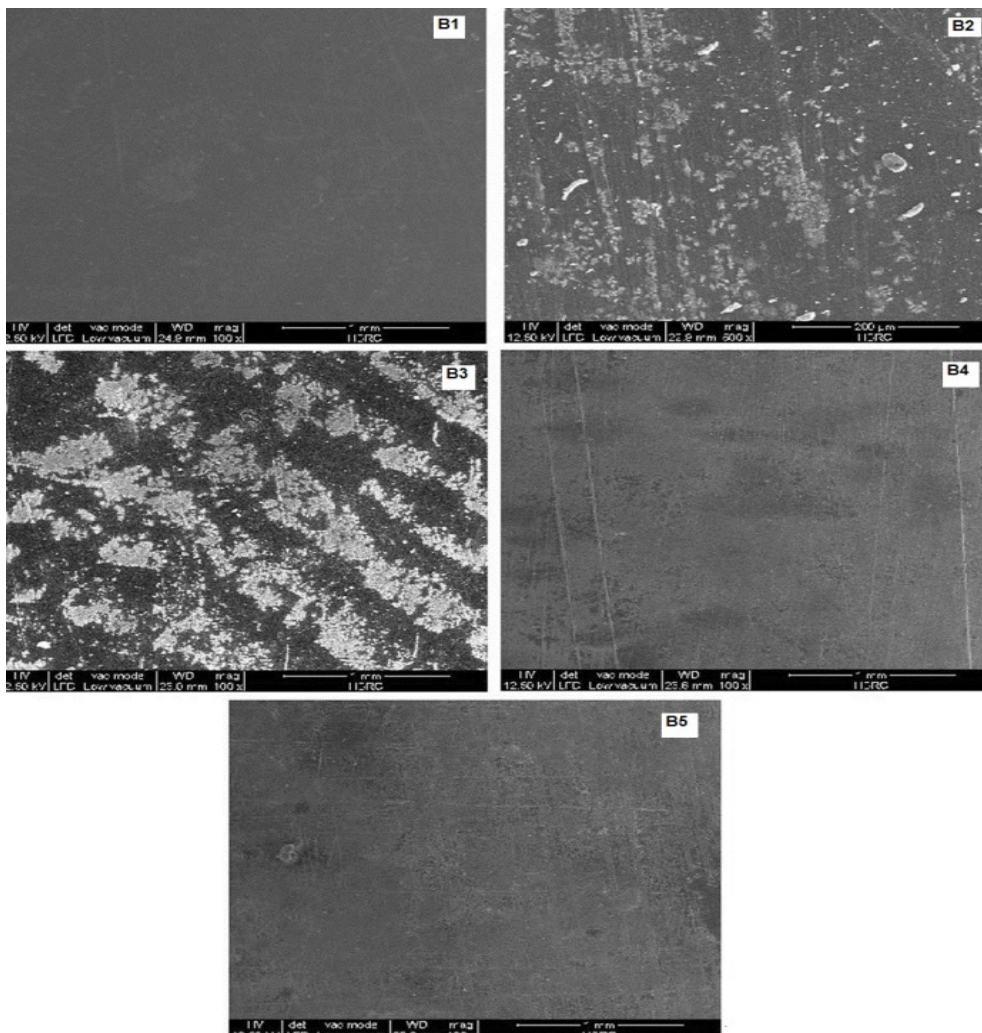


Fig. 5. SEM Micrographs of crumb/NR nanocomposites Having different organo-modified nanoclay loadings

**TABLE 3. Thermal properties of crumb/NR nanocompounds (°C)**

Mix	T10	T50	T90
Bo	357	395	428
B <sub>1</sub>	348	374	389
B <sub>2</sub>	353	390	420
B <sub>3</sub>	353	390	424
B <sub>4</sub>	354	392	425
B <sub>5</sub>	356	398	431

**TABLE 4 .Thermal conductivity measurements of B1 and B5, compared to polystyrene.**

Material	Thermal conductivity, W/m K	Thickness, mm
B <sub>1</sub>	0.137	10.3
B <sub>5</sub>	0.144	10.8
Polystyrene	0.035	50.0

NR composite, the better the maximum torque and cure time will be. Regarding the mechanical properties and hardness, the addition of 2.5, 5, 7.5 and 10 phr of nanoclay to the control crumb/NR compound clearly improves the mechanical properties, specially the tensile strength from 3.13 in the control rubber to 10.44 Mpa in rubber nanocomposite containing 10 phr, representing 300 % increase. TGA data indicates that the high stability of all crumb/NR nanocomposites. Thermal conductivity measurements reflect that crumb/NR nanocomposites are highly recommended for thermal insulation purposes and constructional applications. Thus, the incorporation of crumb rubber into the natural rubber mix was found to provide more safe processing conditions to personnel and to offer a way of making good use of waste tires in producing useful nanocomposites with mechanical and thermal properties that can be applied for shielding and roofing constructional purposes.

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## تقييم الخواص الميكانيكية والحرارية لمتراكبات فتات المطاط/المطاط الطبيعي النانوية

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بالرغم من أن فضلات إطارات السيارات أو كما يطلقون عليه فتات المطاط لا يصنف ضمن المواد الضارة بالبيئة، إلا أنه من الممكن أن تتسبب الطرق الغير الخاطئة أو الغير المدروسة بعناية إلى كوارث بيئية. ومن الجدير بالذكر أنه يمكن الاستفادة من فتات المطاط وإضافته إلى البوليمرات لتصنيع مواد مفيدة من جهة والحفاظ على البيئة من تراكم مثل هذه المواد من جهة أخرى. على سبيل المثال، تستخدم كسرات الإطارات إلى المطاط المستخدم في عمل أفنية المدارس، فراشة الخيول وكذلك ملاعب كرة القدم. وقد يتسبب فتات المطاط، عند إضافته إلى متراكبات المطاط، إلى تأثير الخواص الميكانيكية لهذه المتراكبات تأثيراً سلبياً ولكن هذا التأثير يمكن تلاشيته بزيادة نسبة المواد الأخرى المضافة للمطاط ومن ثم الحفاظ على هذه الخواص. في هذا البحث، قمنا بإضافة فتات المطاط إلى المطاط الطبيعي بنسبة ١:١٠ وخفض نسبة أسود الكربون والإستعاضة عن هذه النسبة بإضافة الطفلة النانوية بنسب مختلفة من الطفلة النانوية بنسب مختلفة: ٠، ٢، ٥، ١٠، ١٥، ٢٠، ٣٠، ٤٠، ٥٠ جزء لكل ١٠٠ جزء من المطاط. وقد أوضحت نتائج دراسة التركيب البنائي للمتراكبات النانوية المحضرة بهذه الطريقة الإمتزاج والتجانس الجيد بين فتات المطاط والمطاط الطبيعي من جهة وبين باقي المكونات المضافة إلى المتراكبات النانوية من جهة أخرى، كما أوضحت النتائج دور الطفلة النانوية في الحفاظ على معدلات الخواص الميكانيكية والعزل الحراري.