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Electrical Conduction and mechanical properties of irradiated clay nanoparticles /SBR rubber composites

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SURFACE Modified nanoparticles clay was synthesized and treated by electron beam and clay fillers including acrylate monomer were studied. The current flowing through the treated clay/ SBR composites and untreated samples as a function of applied voltage was measured whilst maintaining the sample at 300 K. Direct current measurements were established to identify the conduction type of charge transport passing through the modified and unmodified SBR/clay composite samples. Where, it is deduced that Poole-Frenkel conduction mechanism was found to be dominant for all clay-SBR samples. The mechanical Tensile strength and elongation at break were estimated from stress strain curves measured and it was concluded that the tensile strength of SBR matrix increases significantly with clay loading up to 15 phr. The relation between V_{ro}/V_{rf} & C/ (1-C) was studied and the results showed enhanced polymer-filler interaction in the modified samples. Elongation at break increases slightly with clay loading up to 10 phr and then abrupt decrease is detected at 15 phr clay loading for all samples.

Keywords: Nano composites, Electron beam, SBR, DC properties, Mechanical properties.

Introduction

Clay has been used extensively in different rubbers as filler for many years and it is of great commercial interest it makes the producs cheap. Due to its low surface activity, clay has very poor reinforcing ability as compared to carbon black or precipitated silica. Modification of the filler surface to improve adhesion has become increasingly importantKondo, Miyazaki et al. (2008). A new method of clay surface modification by coating the clay fillers with an acrylate monomer, trimethylolpropanetriacrylate (TMPTA) followed by electron beam irradiation of the coated fillers has been reported Ray and Bhowmick (2002). It has been found that compared to pristine clay, the surface treated clay fillers show better physical properties while incorporated in nitrile rubber. Precipitated silica fillers have been modified by the above technique Ray and Bhowmick (2002) and it is found to exhibit significant property improvement while added in the ethylene -octene copolymer (Ray and Bhowmick 2001). A major test of the mechanical behavior of polymers, especiallythose plastics below their glass transition temperature, involves themeasurement of tensile strength. While it can be argued that tensilestrength is not thebest quantity to characterize engineering behavior, itis simple, inexpensive, and very widely reported (Karger-Kocsis and Zhang 2005). The conduction electrons are originated by impurities or from products of polymer degradation. in the recent years, there have been few attempts to study, for example, charge carrier mobility as an independent variable. Electrical conductivity is important in many rubber and plastic compounds including anti-static applications, wire and cable sheathing (Madani and Aly 2010), (Koh, Park et al. 1996) and shielding against electromagnetic interference (EMI) (Madani 2010). Electrical conduction in polymers has been studied extensively during the past two decades to understand the nature of charge transport in these materials. Various mechanisms, such as Schottky emission, Poole–Frenkelemission, space-charge limited conduction , and hopping conduction, have been suggested for the charge transport. Considerable interest has been shown on the effect of doping on the transport properties of

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polymers (Hernán, Morales et al. 1998) (Ishida and Miller 1984, Pukanszky 1995, Petrović, Javni et al. 2000). Depending on their chemical nature and the way in which they react with the host matrix, the doping substances alter the transport properties to different degrees. Consequently, the so-called fine particle filled polymers sometimes contain a number of loosened clusters of particles and exhibit properties even worse than conventional particle/ polymer systems (Herron and Thorn 1998, Von Werne and Patten 1999). In the present work clay, surface modification by coating the clay fillers with an acrylate monomer, trimethylolpropanetriacrylate (TMPTA) followed by electron beam irradiation of the coated fillers have been synthesized. Also, mechanical variation of conduction mechanisms and thermal decomposition behavior as well - as thermo mechanical were studied curves to investigate the effect of Nano clayparticles treated and untreated by trimethylloopropane triacrylate monomer (TMPTA) loading on SBR matrix.

Experimental Work

Materials

All the materials under investigation are commercially available products. The styrene butadiene rubber (Grade : 1502) containing 23.5% styrene was supplied by Synthetic and Chemicals Ltd, Barielley, India. The clay powder (bentonite, BE125, mean size 100 nm, density= 2.5 gcm^{-3}) was obtained from Spectrum Chemicals & Laboratory Products, USA. The acrylate monomer, trimethylolpropanetriacrylate (TMPTA) (flash point >100^0C, b.p.>100^0C, specific gravity 1110 kg/m^3) was obtained from UCB chemicals, Belgium. TMPTA was used as grafting monomer without further purification.

Sample Preparation

Before being mixed with the monomers, the Nanoparticles were preheated at 120°C for 5 hr to eliminate the absorption water on surface of the particles. Then 100 g of the powder fillers were mixed with 100 ml 3wt% solution of TMPTA in acetone in a glass beaker under constant stirring with a glass rod and then the solvent was removed by evaporation technique, followed by grinding of dry fillers to obtain the surface coated fine powders. The treated filler, acrylate modified, was irradiated in an electron beam accelerator (Model ICT) in the presence of air at NCRRT, Cairo, Egypt. Treated nanoparticles were irradiated at a dose of 100 kGy. The dose determined by the FWT 60-00 dosimeter that was calibrated using the CERIC/CEROUS dosimeter. The uncertainty in the delivered dose was estimated to be 1.15% (Yang, Yang et al. 1996).

Energy dispersive X-ray spectroscopy (EDX) was performed on untreated, irradiated and treated fillers in a JEOL-JSM 5800 scanning microscope operating at an accelerating voltage of 30 kV and equipped with Linux X-ray analyzer. However after TMPTA modification, a noticeable decrease in the oxygen/elements ratios from 0.91 to 0.87 is observed as compared to the control sample, indicating the presence of acrylate TMPTA in it (Ray and Bhowmick 2002). In order to do a comparative study, both unmodified, and modified clay fillers were incorporated in SBR by different concentrations. The formulations of different mixes are given in Tables 1 and 2 for non-modified and modified clay respectively. SBR rubber was mixed in a Bra bender Plastic order PLE-319 at 80 rpm rotor speed for 2 min at Temperature 80°C and then the compound ingredients were then added constituents in ratios as shown in Tables (1) and (2) respectively. The total time of mixing was 10 min. After compounding, the stocks were left for 24 hr to mature. It was then cured into sheets of 2mm thick using hot press at 5 MPa pressure in an electrically heated press (type carver M-154) and heating temperature 170± 20C for 20 min. To insure reproducibility, the samples were conditioned at 70°C for 20 days (Nasr and Madani 2005).

TABLE 1. Composition of SBR-untreated clay Nano composites.

Ingredient, phr[(a)]	Sample desination					
	blank	**Su5**	**Su10**	**Su15**	**Su20**	**Su30**
SBR	100	100	100	100	100	100
Zinc oxide	5	5	5	5	5	5
Stearic acid	2	2	2	2	2	2
Clay	0	5	10	15	20	30
DCP	1	1	1	1	1	1
Sulphur	2	2	2	2	2	2

TABLE 2. Composition of SBR- treated clay Nano composites.

Ingredient, phr[(a)]	Sample desination				
	St5	**St10**	**St15**	**St20**	**St30**
SBR	100	100	100	100	50
Zinc oxide	5	5	5	5	5
Stearic acid	2	2	2	2	2
Modified clay	5	10	15	20	30
DCP	1	1	1	1	1
Sulphur	2	2	2	2	2

Characterization

Direct Current Measurements:

The electrical measurements were carried out using a DC voltage/ current generator, along with a precision digital electrometer (Keithley 6514) to determine the current generated on application of a known voltage to filled rubber blend vulcanizes. The sample holder consists of two parallel clamps of brass electrodes, which were isolated from each other using Teflon.

Mechanical Measurements:

Dumbbell shapedspecimens for tensile are cut from the moldedslabs. Tensile strength was done according to ASTM D-412-06 the values of tensile strength, modulus,percentage elongation at break are recordeddirectly from the digital display at the end of each test.Tensile strength and elongation at break were estimated from stress strain curves measured by using a tension meter (carried out with the use of H10KS Hounsfield Co. UK); tension speed was 50mm/ min. tensile tests were carried out on dumbbell shaped specimens. Three samples per formulation were tested. By using the dimensions of samples the stress and strain were calculated.

Results and Discusion

DC conduction mechanism.

The current (I) pass through the bulk modified run modified clay /SBR composites samples as a function of applied voltage (V) is measured while we maintaining the sample at 300 K and plotted as shown in Fig. 1 and 2. We recognize that at low voltages, the current increased gradually with applied voltage and at higher voltages; the rate is slower. This behavior can be explained in terms of Schottky's field-assisted thermoionic emission charge transport (Pillai, Narula et al. (1981), Sharma, Adinarayana et al. 1991, Tyagulskii, Tyagulskii et al. 2013), and the relation for current will be:-

$$I = AsT^2 \frac{\exp\left[\phi / K - C(V/\varepsilon d)^{1/2}\right]}{T} \qquad (1)$$

Where A, is the Richardson constant, s, the electrode area, ϕ, the metal work function , d, the thickness of the dielectric Ɛ , K, the permittivity ,K, the Boltzmann constant, and T, is the temperature in Kelvin. If V expressed in volts and d in cm, the value of C is 4.058 For the Poole-Frenkel effects, the value of C, replaced by 2C.

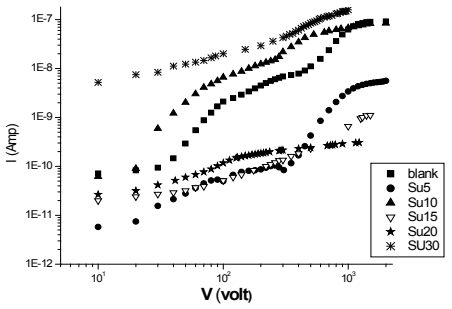

Fig. 1. I – V Characteristic curves for untreated Clay samples at room temperature (300K).

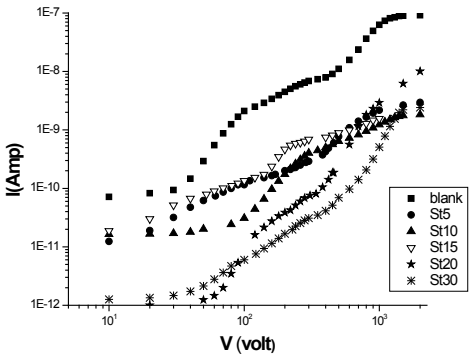

Fig. 2. I – V characteristic curves for treated Clay samples at room temperature (300K).

If the temperature of the sample is maintained constant (300K), then a plot of (log J) versus ($E^{1/2}$) give the required information with respect to the mechanism of charge –transport. The (log J) versus ($E^{1/2}$) plots for modified and unmodified clay/SBR Nano composites are introduced in Fig. (3) & (4).

The plots show a linear behavior with appreciable deviation from linearity at lower fields, which can be attributed to accumulation of space charge at the electrodes. The slope of these plots at higher fields yields important information regarding the nature of the conduction process,and the current-voltage temperature dependence follow the relation

$$I \propto \exp\left(\frac{e\beta E^{1/2}}{KT}\right) \qquad (2)$$

Where,E is the applied field and β, a constant characteristics of the conduction mechanism.

The linear behavior of log J versus $E^{1/2}$ plots in the present study points to an electronic-type conduction mechanism. Here, the charge carriers are released by thermal activation over a potential barrier. The physical nature of such a potential barrier can be interpreted in two ways, It can be the transition of electrons over the barrier between the cathode and the dielectric (Schottky emission). Alternatively, charge carriers can be released from traps into the dielectric(Poole-Frenkel effect) (El Tayyan and Khogali 2004).

In order to differentiate between these two, the values of β at different temperatures were calculated from the slopes of log j versus $E^{1/2}$ plots.

The theoretical value of β can be calculated separately for either the Schottky or the Poole-Frenkel mechanisms by use of the following respective equations

$$\beta_R = \left(\frac{e^3}{4\pi\varepsilon\varepsilon_0}\right)^{1/2}$$

$$\beta_{PF} = 2\beta_{RS}$$

Where, the dielectric constant (at 103 Hz),Ɛ for all samples are measured, $Ɛ_o$ =8.85 x 10^{-12}F/m, and *e* = 1.6 x 10^{-19}C, then, The experimental as well as the theoretical values of β for both the Schottky and Poole-Frenkel mechanisms are calculated and tabulated. It is clear from Table 3 that all samples obey Pool-Frenckel conduction mechanism except the blank one, which isSchottky conduction mechanism.

TABLE 3. Theoretical and experimental values of β for Schottky and Poole- Frenkel mechanism.

β_{PF}	β_{RS}	β_{Exp}	**Sample**
2.92 E-05	1.46E-5	1.38E-05	**Blank**
Untreated samples			
2.84E-05	1.42 E-05	3.75E-05	**Su5**
3 E-05	1.50 E-05	2.78E-05	**Su10**
2.9 E-05	1.45 E-05	3.14E-05	**Su15**
2.8E-05	1.40 E-05	2.5E-05	**Su20**
2.64E-05	1.32 E-05	3.02E-05	**Su30**
Treated samples			
2.7 E-05	1.35 E-05	2.58E-05	**St5**
2.96 E-05	1.48 E-05	3.9E-05	**St10**
2.94 E-05	1.47 E-05	2.65E-05	**St15**
3.1E-05	1.55 E-05	4.2 E-05	**St20**
2.66 E-05	1.33 E-05	2.97E-05	**St30**

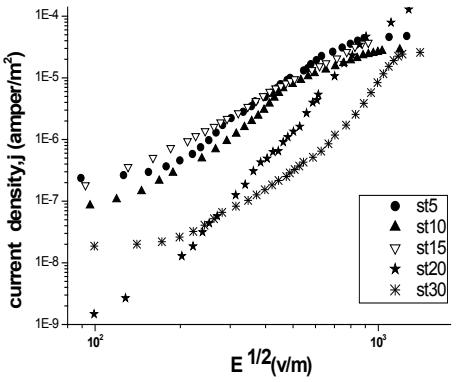

Fig. 3. Log (J) versus $E^{1/2}$ for (SMEB) samples

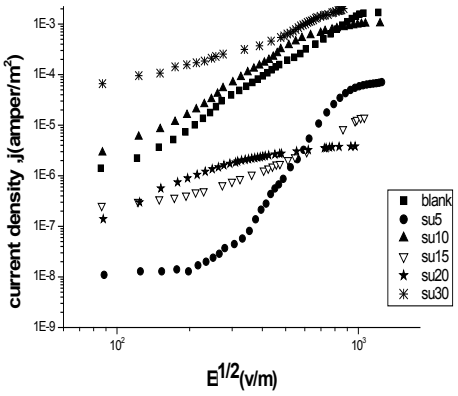

Fig. 4. Iog (J) versus E1/2 for unmodified samples.

Mechanical properties

Polymer Nano composites represent a new alternative to conventionally filled polymers due to their filler size and dispersion. Nano composites exhibit markedly improved properties compared to the pure polymers or their traditional composites(El-Nashar, Mansour et al. 2006). These includes increased modulus and strength (Lagashetty and Venkataraman 2005). The improvement of the mechanical properties of the material have been related to the very small size of the reinforcing clay platelets on one hand, and their large aspect ratio on the other (Van Es 2001).

The relation of the stress-strain is shown in Fig. 5 and 6 for untreated and treated clay, respectively, where with introducing the organic modifier is to increase the interfacial interaction with the polymer chains. So, all the compositions showed a tensile strength higher than the pristine rubber. And by increasing the filler loading, the tensile strength of the prepared composites increases too.

When there is a considerable strength rise of clay-g-TMPTA/ SBR composites at a clay content = 10 phr, and by increasing the treated filler we found that thestrength is decrease. Here the optimum value is attained at 10 phr of filler loading. By the use of treated filler, one can achieve best mechanical properties by the incorporation of small amount of filler. Moreover, the treated clay basal SBR samples are more improve with respect to untreated one. It well known that the interface adhesion markedly influences the mechanical behavior of particulate filled polymer composites. The degree of reinforcement depends on the extent of polymer-filler interactions. The extent of polymer-filler interactions is estimated from swelling experiments using a plot of V_{r0}/V_{rf} versus the C/ (1-C) plot according to the Kraus equation (Kraus 1963, Kraus 1965).

$$\frac{V_{r0}}{V_f} = 1 - \frac{m}{1-c} \qquad (3)$$

Where V_{rf} is the volume fraction of rubber in the filled vulcanized and is given by(Chattaraj, Kalidaha et al. 1996):

$$V_r = \frac{(D - F)\rho_r^{-1}}{(D - F)\rho_r^{-1} + A_0\rho_s^{-1}} \qquad (4)$$

Where T is the weight of the test specimen, F is the weight fraction of the insoluble components in the specimen, D is the deswollen weight of the test specimen, A_o is the weight of the absorbed solvent (corrected for swelling increment), p_r is the density of SBR, and p_r is the density of the solvent. V_{ro} is the volume fraction of rubber in the gum vulcanized, C is the volume fraction of filler in the vulcanization, and m is the polymer- filler interaction parameter obtained from the slope of the V_{ro}/V_{rf} versus C/ (1-C) plot. The slop should be positive for reinforcing filler having good polymer-filler interaction and negative for nonreinforcing filler with very weak polymer-filler interaction.

It was observed from Fig. 7 that polymer-filler interaction for treated samples is higher than for untreated samples.

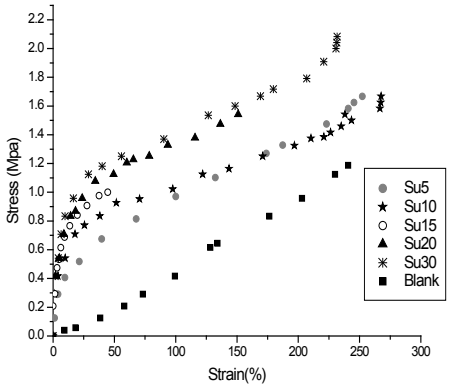

Fig. 5. Stress–strain curves for untreated samples at 300K.

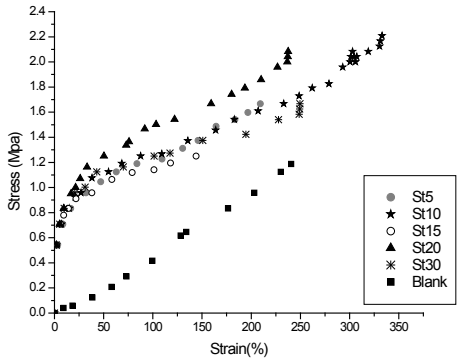

Fig. 6. Stress –strain curves for treated samples at 300K.

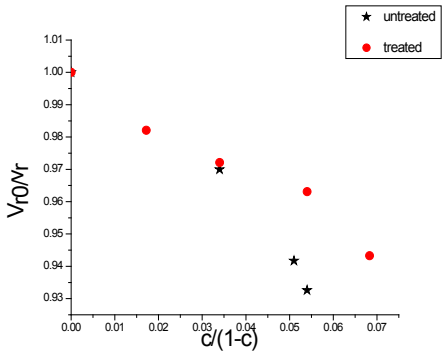

Fig. 7. shows the relation of V_{ro}/V_{rf} versus c/ (1-c) for both treated and untreated samples.

The elongation at break for treated and untreated clay filled composites increases with an increase the loading of filler up to 10 phr and then followed by a decrease up to 15%. This reduction is more pronounced for untreated clay loaded samples and is due to stiffening of the matrix by the clay particles or filler. In fact, with further increases in filler loading the molecular mobility decreases due to formation of physical bonds between filler particles and polymer chains that stiffen the matrix up to 15 phr. This results in a drop in elongation at break with filler loading Table 4 beyond 10 phr clay. However, a higher elongation at break is observed for SBR loaded with 10 phr of treated clay. This is mainly because of the greater chain mobility of these samples than that of untreated clay loaded SBR samples.

Increasing the clay loading beyond 15 phr, leads to an increase of the elongation at break for both treated and untreated clay particles.

TABLE 4. Elongation at break versus clay loading for untreated and treated samples.

Clay content (phr)	Elongation at break (%)	
	Untreated clay	Treated clay
0	259.2	
5	252.2	209.25
10	267.6	336.8
15	44.5	143.9
20	151.05	237.6
30	231.7	249.6

Crosslink density determination.

The crosslink densities of the treated and untreated clay loaded SBR samples were determined from the elastic rubbery moduli of the samples according to the rubber elasticity theory modified by Nielsen (Landel and Nielsen 1993).

$$\upsilon = \frac{E}{3RT} \qquad (5)$$

Where υ represents the crosslink density, R is the gas constant, T is the temperature in Kelvin (300k), and E is the elastic moduli obtained from the stress-strain curves (as shown in Fig. 4 for both groups).

The strong impact of the presence of the clay on the initial modulus of the materials,as evaluated by the slope at the origin of the nominal stress-strain curves,can be observed in Fig. (8). The initial modulus increases from 0.5Mpa to 8.8, 8 Mpa with increasing the filler volume fraction from 0 to 20 phr for both of treated and untreated clay respectively. However, the increment of the modulus with the clay contents appears strongly non-linear, suggesting two different regimes, at low and high montmorillonite content, respectively.

The calculated values for the crosslink densities are tabulated in Table 5. It was found that υ increases with both treated and untreated clay contents and treated samples have higher increasing rate of υ values than untreated one.

The kinetic theory predicts a directly proportionality relationship between the stress at break σ_b and υ, the crosslink density, on the other hand, the strain at break, ε_b, should increases as the crosslink density decreases (Fedors and Landel 1975).

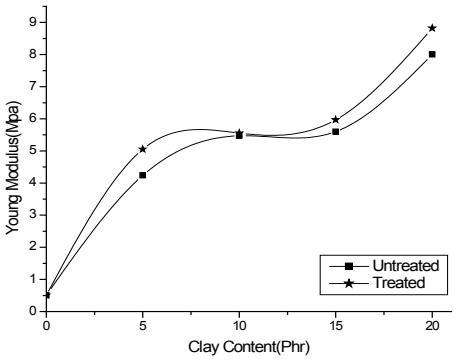

Fig. 8. Young's modulus versus clay loading for untreated and treated samples (at 300K).

TABLE 5. Crosslink density for untreated and treated samples (at 300K).

Clay content (phr)	Crosslink density (mole of chain /cm^3)	
	Untreated clay	Treated clay
0	67.9	
5	566.4	675.3
10	731.6	742.8
15	749.1	797.9
20	1070.2	1179.1

To elucidate the tensile behavior of the test samples, the HT model is tested by using non-Gaussian chain statistics. The HT model in conjunction with non-Gaussian chain statistics for rubber elasticity provides for a limited extension of the chain and leads to anequation predicted elsewhere (Haward 1999). In this equation L^{-1} represent the inverse Langevin function.

Where $L(x) = (\coth x - \frac{1}{x})$

Where L^{-1} is the inverse Langevin function. However, according to Cohen (Cohen 1991), the inverse Langevin function may be accurately approximated by a pade equation andchecked by Cohen(Cohen 1991, Haward 2000), to bring for us an equation simpler for nominal stress *f* and more easily to be calculated

$$f = \frac{Y_0}{\lambda} + \frac{C_r}{3}\left\{\frac{\lambda\left[3-\left(\frac{\lambda^2}{n}\right)\right]}{\left[1-\left(\frac{\lambda^2}{n}\right)\right]} - \frac{\left[3-\left(\frac{1}{\lambda n}\right)\right]}{\lambda^2\left[1-\left(\frac{1}{\lambda n}\right)\right]}\right\} \quad (6)$$

Where λ is the extension ratio and n is the number of flexible units between crosslinks,and in this article Y_0, is the yield stress and may be separately measured or treated as a disposable constant,generally, the two values are much the same.Furthermore, it can readily be seen that when n is large the equation approximates to Gaussian form where the second term becomes Cr (λ-λ^{-2}). It should be noted that when uniform extension takes place, as assumed here, nominal stress can be converted into true stress using the constant volume assumption so that true stress is = f λ . In order to support the validity of the HT model, the stress-strain curves for samples loaded with different concentration of modified and unmodified clay has been fitted by equation (6) which reflects the good fitting between both theoretical and experimental values, and we found that the fitting parameter, n, increases with unmodified clay contents meanwhile; it has a maximum value with modified clay at 10 phras indicated in Table 6.

So the number of flexible chain per unit crosslinks is higher for samples loaded with unmodified clay, reflects the good reinforcement gained by modified clay than unmodified one.

TABLE 6. Fitting parameter (n) for all samples.

Clay (phr)	Unmodified	mdified
5	5×10^6	5×10^6
10	5×10^7	6×10^6
15	4×10^7	9×10^5
20	6×10^7	4×10^6

Conclusion

Clay/SBR Nano composite were prepared using internal mixture of modified and unmodified clay filler. The charge carriers are released by thermal activation over a potential barrier. The physical nature of such a potential barrier can be interpreted in two ways, It can be the transition of electrons over the barrier between the cathode and the dielectric (Schottky emission) or charge carriers can be released from traps into the dielectric (Poole-Frenkel effect) and to differentiate between these two, the theoretical and experimental values of β were calculated separately for both the Schottkyand the Poole-Frenkel mechanisms. It was clear that all samples obey Pool-Frenckel conduction mechanism except the blank one, which followed Schottky conduction mechanism.

With increasing the filler loading, the tensile strength of the prepared composites increases up to 15 phr. The optimum value of thetensile strength for the prepared clay is attained at 10 phr of filler loading. Moreover, the mechanical properties of the treated clay basal SBR samples showed more improvement compared to unmodified one. Elongation at break increases slightly with clay loading up to 10 phr and then abrupt decrease is detected at 15 phr clay loading (for modified and unmodified samples). In addition, the fitting parameter, n, increases with unmodified clay contents meanwhile; it has a maximum value with modified clay at 10 phr.

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التوصيل الكهربي والمواصفات الميكانيكيه لمتراكبات مطاط ال SBR المحمل بحبيبات متناهية الصغر من الميكا المشعه

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تستخدم متراكبات البوليمرات المحملة بالمعادن، أشباه الموصلات، أسود الكربون، ، انواع الطمي النانومترية المختلفة كمواد لها تطبيقات صناعية متعددة أو يتم تحضيرها بغرض التطوير في خواص البوليمرات.

وتظهر متراكبات البوليمرات عند تحميلها بالطمي النانومتري تحسن ملحوظ في خواصها بالمقارنة بمثيلتها من المتراكبات الأخري. فعند تصميم وتجهيز مواد جديدة لها خواص مطلوبة و محددة يجب ان تتوافر في المادة الخواص التي نحتاج لها والقيام بدراستها.

و من خلال منحنى الاجهاد-الانفعال للعينات وجد ان القوه التوتريه للعينات المحمله بالطمى النانومترى ازدادت بنسب كبيره بالمقارنه مع العينه الغير محمله بالطمى كما انها تزداد بزياده نسب الطمى بهذه العينات

ووجد أن الاستطاله عند الكسر للعينات المحمله بالطمى النانومترى (المعالج والغير معالج) تزداد بزياده نسب الطمى حتى ١٠٪ ثم تنقص عند نسبه ١٥٪.

كما وجد ان الكثافه الوصليه تزداد للعينات المحملة بالطمي كما انها اكبر فى حاله العينات المعالجة .

*وتمت الاستعانه بنموذج HT لتوضيح السلوك التوترى للعينات وحدث تطابق جيد للبيانات المعمليه والنظريه.

تم قياس التيار المار بعينات المطاط مع الطمى النانومترى كداله فى فرق الجهد عند درجه حراره ٣٠٠ كلفن .ووجد أن هناك سلوكا خطيا لمنحنى التيار-الجهد للعينات مع وجود انحراف عن السلوك الخطى عند الجهود المنخفضه وذلك بسبب وجود الشحنات الفراغيه عند اقطاب الدائره المستخدمه فى القياس.

جميع العينات تظهر سلوكا متفقا مع موصليه Poole-Frenckle ما عدا العينه الغير محمله بالطمى فانها تظهر سلوكا متفقا مع موصليه Schottky .