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Effects of Epoxy-Polyester Hybrid and Nanoclay on Morphology, Rheological and Mechanical Properties of Styrene-Butadiene Rubber

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Properties of SBR compounds filled with two kinds of filler, Epoxy-Polyester Hybrid resin(EPH) (10, 20, 30, 40 phr) and Nanoclay (Closite 15 A) (1, 3, 5, 7 phr) were studied. Microcomposite samples and nanocomposite samples were prepared by Haake internal mixer. Curing agents and additives i.e. dicumylperoxide (DCP), Carbon Black (CB) and oil were added to the compound on the two-roll mill. The modified silicate is analysed by X-ray which suggested intercalation of elastomer chains into silicate layers. Mechanical properties of the samples suggested that both fillers increase modulus, but sample containing 30 phr resin show higher value of modulus than sample containing 7 phr nanocaly. Rheological measurement showed that both fillers lead to an increase in viscosity and dynamic modulus of samples which is as a result of good interaction established between polymer/filler. Moreover, due to nanometer scale of nanoclay particles, reinforcing effect of Nanoclay was more noticeable and in micrometer scale of Epoxy-Polyester Hybrid particles, Epoxy resin cured by Polyester is used to improved wet ability of SBR compounds. SEM photomicrographs of cryogenically fractured samples confirmed the mentioned results.

Keywords: Rheological Properties, Epoxy-Polyester Hybrid, Nanoclay, XRD, SEM Photomicrograph, Mechanical Properties

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1. INTRODUCTION

Polymer-clay nanocomposites are of great interest for both scientific challenges and industrial applications [1–4]. In the open literatures, the polymer-clay nanocomposites are generally classified into three groups according to their structures, i.e., nanocomposites with intercalated, exfoliated, or both of intercalated and exfoliated structures. To achieve a nano-scale dispersion of silicates in a polymer matrix, the silicates are generally pretreated with alkylammonium ions to produce an organoclay. This treatment is effective in opening up the gallery of silicate, and the organo-clays can then be easily dispersed in the polymer matrix and form nanocomposites with an intercalated or exfoliated structure. In the former case, the spacing between the silicate layers is increased by the incorporation of extended polymer chains into the layers. The tradition methods to prepare polymer nanocomposites are to intercalate monomers or polymers into swellable-layer silicate hosts, and especially for rubbers, there are mainly three kinds of methods for preparing the rubber matrix nanocomposites: latex, solution, and melt compounding. Considering industrialization, melt intercalation is one of the feasible operations, in which polymer is directly intercalated into modified silicate layers (organoclay) in the molten state to prepare nanocomposites. The driving force of the intercalation is determined by physical and/or chemical interactions between polymers and modified silicate. Regarding to the low cost of the Epoxy-Polyester Hybrid resin(EPH) and improvement observed in rubber compounds, high advantages of using of EPH powders in polymer industry has been evidenced [5]. This study considers the effect of two kinds of fillers on the properties of SBR.

2. EXPERIMENTAL

2.1 Material

SBR, Nanoclay (Closite 15A), EPH and cure ingredients were supplied from Bandar Imam petrochemical co, Southern clay company (U.S.A), Local company (Iran) and Bayer co respectively.

2.2 Samples Preparation

Samples were prepare by Haake internal mixer at temperature of $105 \,^{\circ}$ C and the rotor speed was 60 rpm for 7 min. Curing agents were mixed with compound on two-roll mill (Polymix 200 L) at 25 °C and rotor speed of 70 rpm (see Table 1).

2.3 Characterization

X-ray diffraction patterns of the samples were recorded on a STANDI MP model X-ray Diffractometer "STOE" X'Pert (40 kV, 30 mA) by using Cu-Ka radiation ($\lambda = 1.540598$ Å) with a scanning rate 2°/min at room temperature. The basal spacing of silicates was estimated from the position of the plane peak in the WAXD intensity profile using the Bragg's law, d = $\lambda/(2\sin\theta max)$. The rheological measurements were performed using a RPA 2000 oscillatory rheometer (Alpha Technology Company) at 80 °C and frequency of 0.1-1000 (rad/sec). To study the morphology, the cryogenically tensile fractured of samples were coated with gold and viewed with a TESCAN scanning electron microscope.

3. RESULTS AND DISCUSSION

XRD is a powerful technique to follow the intercalation of elastomer chains into the silicate layers of clay and dispersion of organoclay in the polymer matrixn.

Sample Code	SBR(phr)	Cloisite15A(phr)	EPH (phr)	Carbon Black(phr)	Oil(phr)	DCP(phr)
S	100	-	-	30	5	0.5
N1	100	1	-	30	5	0.5
N3	100	3	-	30	5	0.5
N5	100	5	-	30	5	0.5
N7	100	7	-	30	5	0.5
P10	100	-	10	30	5	0.5
P20	100	-	20	30	5	0.5
P30	100	-	30	30	5	0.5
P40	100	-	40	30	5	0.5

Table 1-Composition of Prepared Samples

Fig. 1 shows the XRD patterns Prepared samples. The interlayer spacing of the original Cloisite 15A $(2\theta = 2.97)$, (d = 31.5Å). Shift of the organoclay diffraction peak to lower 2θ values indicated that elastomer chains intercalated between consequent silicate layers. This clearly indicates that inter lamellar spacing of the clay are enlarged after melt compounding. As expected intensity increases with an increase in nanoclay. Fig. 2 shows the comparison of complex viscosity and dynamic moduli, G' of sample containing nanoclay and EPH. As seen, both fillers have a increasing effect in rheological properties. In fact addition of nanoclay and EPH, lead to an increase in complex viscosity which can be attributed to the good interaction established in the interface of polymer/filler. It is noticeable that EPH causes a greater effect on the viscosity which means more reinforcing than nanoclay. Moreover the viscosity has a descending trend by frequency increase which is because of pseudoplastic nature of compounds. On the other hand, G' of samples have a ascending trend by filler loading. In comparison with pristine SBR, the filled samples show higher value of dynamic modui. It is because of this fact that both fillers form a network structure which increases interaction between polymer/filler and as a consequence, G' increases. It is obvious that EPH filled samples show higher dynamic moduli[3, 6]. Insights on the morphology of prepared sample are illustrated in Fig. 3 where SEM images in the same magnification of cryogenically fractured of samples with and without EPH and Nanoclay are reported The harsh fracture surface of sample containing 5% nanoclay and sample containing 30 phr EPH compared to pristine SBR is representative of good interaction between matrix and filler particles, implying the effective reinforcement of fillers.



Fig. 1 – XRD results of prepared samples



Fig. 2 – Rheological properties of samples filled with a) EPH and b) Nanoclay $% \left({{{\mathbf{F}}_{\mathrm{s}}}^{\mathrm{T}}} \right)$



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Fig. 3 - SEM Photomicrographs of samples

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4. CONCLUSION

Rubber comounds based on SBR/nanoclay and SBR/EPH were prepared via vulcanization process and characterized by several techniques. In these materials rubber chains could penetrate into silicate layers resulting an increase in d-spacing of layers which was confirmed by XRD results. On the other hand enhanced viscosity and dynamic modulus of samples suggested a good interaction between polymer matrix and filler particles that also was viewed by SEM photomicrographs.

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