

## **SYNTHESIS AND XRD CHARACTERIZATION OF NATURAL RUBBER/CLAY NANOCOMPOSITE**

F. M. Rais<sup>1</sup>, Z.A.Talib<sup>1</sup>, W. M. Z. W. Yunus<sup>2</sup>, W. D. W. Yusoff<sup>1</sup>, A. Kassim<sup>2</sup>

<sup>1</sup>*Physics Department,*  
<sup>2</sup>*Chemistry Department,*  
*Faculty of Science and Environmental Studies*  
*University Putra Malaysia*  
*43400 UPM, Serdang*

*Email: fadhlina\_rais@yahoo.com*

### **ABSTRACT**

Natural rubber/clay nanocomposites were synthesized via solvent method with the presence of cetyltrimethylammonium bromide (CTAB) as the compatibilising agent. Compatibilising agent is very important to compatibilize the chemistry of the clay and the hydrophobic of the rubber matrix. The composites were divided into various percentages of organoclay and the nanostructure of the composite was investigated by Shimadzu X-ray diffractometry (XRD) with radiation at 60kV and 80mA. The XRD patterns of NR/clay nanocomposites showed that the characteristic diffraction peak of the organoclay disappeared for nanocomposite with one percent to five percent of organoclay in the composite. This is attributed to the homogenous dispersion of the organoclay in the natural rubber matrix and forming an exfoliated nanocomposite. Between seven to fifteen percent of organoclay, the graph slowly showed a strong peak at  $2\theta = 2.6$  with the d-spacing = 33.953. This happened because of the insertion of the natural rubber in between the clay layer to form an intercalated nanocomposite. Thus, by varying the concentration of modified clay, different type of NR/clay nanocomposite can be obtained.

### **INTRODUCTION**

Nanocomposite material is an introduction for a combination of two or more material which one of the materials is in nanometer scales. The synthesis of natural rubber/clay is one of the nanocomposite material where it is a formation of an interaction between polymer (natural rubber) and inorganic (clay) phase. Because of the differences in the properties, dispersion of these two materials is very difficult. A compatilising agent which is usually alkylammonium ion is the most popular agent used and it can easily exchange with the organic ions situated in the galleries between the silicate layers. It is typically a molecule with hydrophilic and organic function. The replacement of the inorganic exchange cation in the galleries of the clay by alkylammonium surfactant can compatibilize the surface chemistry of the clay and the hydrophobic of the polymer matrix and also expands the clay galleries. As a result, the longer the surfactant chain length and the higher the charge density of the clay, the further apart the clay layer will be forced [1]. The dispersion of organoclay-modified layered silicate in the polymer matrix leads two classes of nanocomposites. In the first class, the insertion of the natural rubber in between the layer denoted as intercalated nanocomposite. In second-

class silicate layers of about 1nm thick are exfoliated and dispersed in the polymer matrix, denoted as exfoliated nanocomposites [2]. Polymer-clay nanocomposite shows remarkably property improvement such as increased tensile properties, decreased gas permeability, increased thermal stability and flame retardant compared to conventionally-scaled composites. Furthermore, in both scientific and industrial area, the improvement in mechanical, thermal, chemical, optical, electronic and magnetic application had been also achieved significantly. Therefore, it is important to understand the structure-property of polymer-clay nanocomposite for designing new materials with desirable and predicted properties [3].

## **MATERIALS AND METHODS**

The materials used for the preparation of the sample were purified montmorillonite (Kunipia-F), cethylmethylammonium bromide and natural rubber (SMR CV 60). Toulene were used to dissolve the rubber.

The preparation of modified clay was performed by the following method. First, mixture of 20g of montmorillonite (Kunipia-F) and 600ml of hot deionised water were mixed thoroughly in a 1000ml beaker and this solution was heated in an oil bath at 80<sup>0</sup>C for 1 hour. In a 500ml beaker, 18.223g of compatibilising agent [cethylmethylammonium bromide (CTAB/c19)] and 200 ml hot water were dissolved at temperature of 80<sup>0</sup>C. After one hour, this solution was poured into the montmorillonite-water solution with vigorous stirring by using a mechanical stirrer for one hour at the same temperature. The white precipitates were collected and filtered, washed with hot deionised water for two days until the water solution become transparent. The precipitate was then dried in an oven at 50<sup>0</sup>C for about two days. The dried modified-montmorillonite solution was then grinded until it became powder (~ 100 $\mu$ m).

For preparing the NR/clay, 15g of natural rubber (SMR CV 60) were dissolved in 500ml toluene in a 1000ml beaker. Various amount of modified clay, from one percent to fifteen percent, was dispersed in 50ml toluene. The modified clay-solution was then added to the rubber-toluene solution. The solution was stirred vigorously for at least two hours. When the solution had mixed homogenously, it was removed from the beaker and was put in a plate for casting. The sample was pressed in a hot press at 100<sup>0</sup>C for 30 min. The sample was then ready for analyzing.

The nanostructure of the composite was investigated by using Shimadzu X-ray diffractometry with radiation at 60kV and 80mA. The XRD pattern of modified clay, purified clay, natural rubber and NR-clay nanocomposite were obtained and compared.

## RESULTS AND DISCUSSION

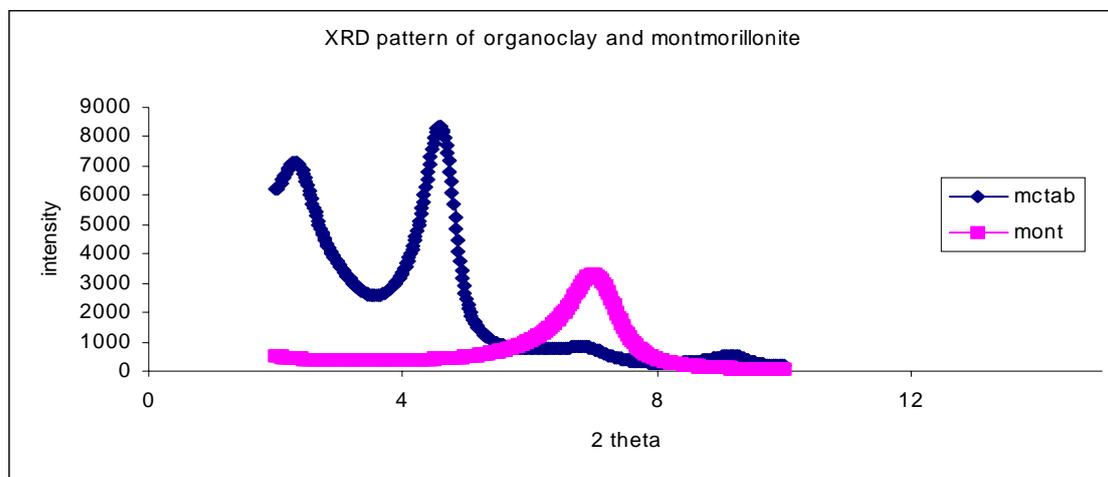


Figure 1: The XRD pattern of modified clay (mctab) and clay (montmorillonite).

Organoclay, mctab is made from montmorillonite modified with cetyltrimethylammonium bromide. XRD of montmorillonite and  $C_{19}H_{42}BrN$ - montmorillonite is shown in figure 1. The interplanar spacing of the mctab is increased from  $12.75\text{\AA}$  to  $19.38\text{\AA}$  after the sodium ions in the gallery were exchanged with  $C_{19}H_{42}BrN$ . This is due to reduction of the hydrophilicity of montmorillonite by allowing dispersion in organic solvent. As montmorillonite has an excess negative charge, it can combine with organic cation to yield organophilic montmorillonite intercalated with organic cation.

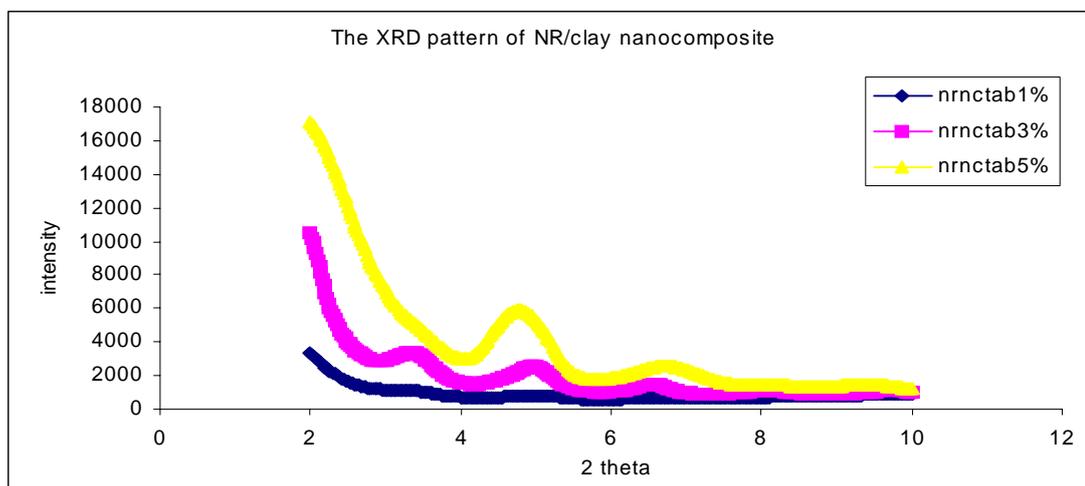


Figure 2: The XRD pattern of NR/ Clay with the presence of one percent to five weight percent of modified clay.

The characteristics peaks at  $2\theta = 4.5$  in the XRD pattern of the mctab disappeared with the organoclay content of 1wt% to 5wt% as shown in figure 2 suggesting that the interplanar spacing of these two nanocomposite are larger than  $\sim 40\text{\AA}$  [4]. This is also attributed to the homogenous dispersion of the organoclay in the natural rubber matrix to form exfoliated nanocomposite.

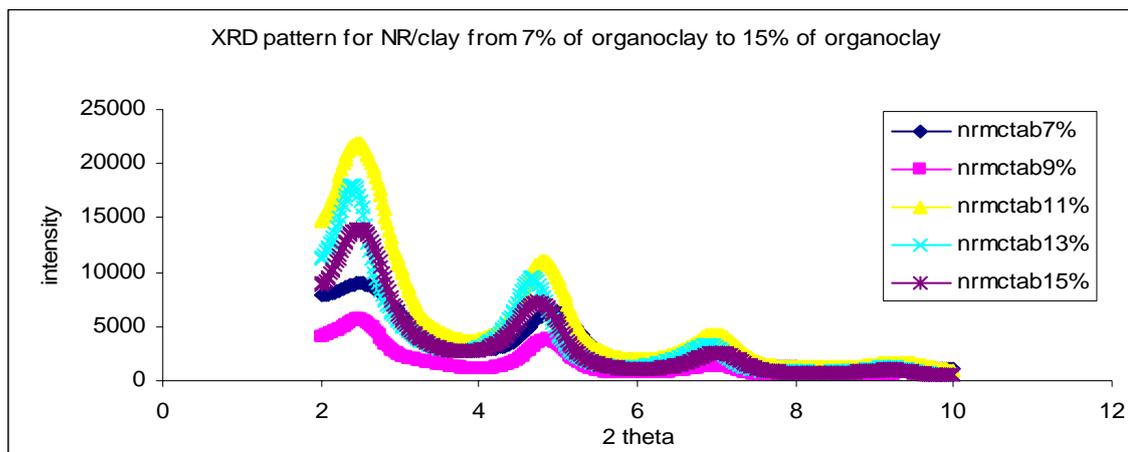


Figure 3: The XRD pattern of NR/ Clay with the presence of seven percent to fifteen weight percent of modified clay.

A different pattern of XRD results happened at organoclay content of 7wt% to 15wt%, suggesting that there is still some ordered structure in the nanocomposite. This happened because of the insertion of the natural rubber in between the clay layer to form an intercalated nanocomposite.

### CONCLUSION

Natural rubber/clay nanocomposite was successfully synthesized by solvent method. Cethyltrimethylammonium bromide was used as the compatilising agent to expand the clay galleries. XRD pattern showed two different characterizations. At 1wt% to 5wt%, the organoclay dispersed homogenously and formed exfoliated nanocomposite where as a the insertion of the natural rubber in between the clay layer of 7wt% to 15wt% of organoclay content in nanocomposite formed an intercalated nanocomposite.

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