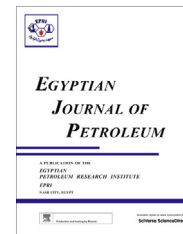




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FULL LENGTH ARTICLE

# Electrophysical characteristics of polyurethane/ organo-bentonite nanocomposites



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**Abstract** Modification of the Egyptian Bentonite (EB) was carried out using organo-modifier namely; octadecylamine ODA. Before the modification, the cation exchange capacity (CEC) of the EB was measured, also it was purified from different impurities using HCl and distilled water. The Organo-bentonite OB was characterized using IR, XRD, and TEM. PU/ODA-B nanocomposites were prepared by in situ polymerization then characterized by XRD and TEM. An amount of ODA-B ranging from 0.25% up to 5% by weight was added to the polyol component of the resin before mixing with toluene diisocyanate TDI. TEM showed that the nanocomposites achieved good dispersion in the polyurethane matrix. The mechanical, swelling and electrical properties of the nanocomposites were measured. The results indicate that the tensile strength of all the nanocomposites enhanced with the addition of OB compared with the pure PU. The crosslink density of the nanocomposites increases with increasing the content of OB. The Pool-Frenkel conduction mechanism predominates for all the nanocomposite samples and the blank one.

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## 1. Introduction

Polymers filled with nano-layered silicate clay particles have attracted researchers in the recent years due to their superior mechanical (tensile, impact, etc.), thermal (decomposition, mass loss, etc.) and physical (barrier, optical, etc.) properties. Such improved properties were noted at small filler content (< 5 wt%) in the polymer matrix with very little change in

density [1–4]. Clay/polymer nanocomposite materials have attracted great interest because nanoclays can reinforce almost all types of polymer matrices with similar properties than traditional composites but with less weight and better processability [5,6]. There is a large body of literature that discusses the mechanical property behavior of filled polymer systems [7–12]. These reports reveal that the modulus is the easiest mechanical property to estimate, because it is a bulk property that depends primarily on the geometry, modulus, particle size distribution and concentration of the filler. The tensile strength of a filled polymer is more difficult to predict because it

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depends strongly on local polymer-filler interactions as well as the above factors.

Recent investigations on conducting polymer composites have led to some important revelations relating to attainable electrical conductivity range and trends of change in the conductivity parameter with variations in: (i) the loading level [13], (ii) nature of the matrix polymer [14], (iii) degree of filler dispersion [13,15] and (iv) temperature [16,17].

Polyurethanes (PUs) are unique polymer materials with a wide range of physical and chemical properties. With well-designed combinations of monomeric materials, PUs can be tailored to meet diversified demands of various applications such as coatings, adhesives, fibers, thermoplastic elastomers, and foams. However, PUs also have some disadvantages, such as low thermal stability and low mechanical strength, etc. To overcome these disadvantages, a great deal of effort has been devoted to the development of nanostructured polyurethane (PU)/montmorillonite (MMT) composites in recent years [18–21]. The effects of clay type, clay content, and PU molecular structure on clay dispersion in thermoplastic PU nanocomposites have been studied. Good dispersion of clays in the PU matrix has been achieved through the modification of MMT with active surfactants containing more than two hydroxyl groups [20]. The presence of hydroxyl groups enhanced intra-gallery polymerization, which in turn led to better clay dispersion. However, the morphology of nanocomposites prepared by this approach was still a combination of exfoliation/intercalation and the method only worked at low clay content, i.e. less than 3 wt%, even for organoclay modified by surfactants with three hydroxyl groups. The exfoliated clay dispersion was only observed at low reaction rates in solution polymerization [20].

The aim of this work is to prepare nanobentonite for polymer nanocomposite hybrid materials based on Egyptian Bentonite (EB) raw material. The influence of modified nanobentonites on the mechanical and electrical properties of PU nanocomposite was also studied.

## 2. Experimental

### 2.1. Materials

Egyptian Bentonite (EB), is supplied from south of El-Hamamm district, grinding through ball-mill and saving at 0.6 micron. It was dried at 80 °C for 24 h under vacuum conditions. (Reported basal plane spacing,  $d_{001} = 1:26$  nm).

Octadecylamine ODA  $\text{CH}_3(\text{CH}_2)_{17}\text{NH}_2$  (surfactant) ( $M_w$  269.51 g/mole), hydrochloric acid and sodium chloride, were provided by Aldrich, USA. Toluene-2,4-diisocyanate TDI was obtained from Fluka, Germany. Polyethylene glycol (PEG)  $M_n = 600$  g/mol, OH functionality = 2.0 and Methyl Ethyl Ketone (MEK), were obtained from Fluka, Germany and Butanone, ADWIC Co respectively.

### 2.2. Preparation of organo-bentonite [22]

EB was purified to remove impurities, i.e. carbonates, iron hydroxide and organic matter, then activated to Na-B according to methods mentioned in a previous work [23]. The organo-bentonite was synthesized by ion exchange reaction between Na-bentonite and octadecylamine ODA. The solution

of the ammonium salt used was heated at 80 °C for a few minutes. The ODA was protonated by adding HCl. Aqueous suspension of 0.5 wt% Na-B was prepared and heated at 80 °C. The prepared alkylammonium salt solution was drop wise added to the Na-bentonite suspension and maintained at 80 °C for 12 h under vigorous stirring. Then the suspension was cooled to room temperature. The equation for calculating the intercalating agent needed for a cation-exchange reaction is [24]:

$$\frac{120}{100} \times \text{grams of clay} \times 1.5 = \frac{X}{M_w \text{ of intercalating agent}} \times 1 \times 1000 \quad (1)$$

where  $X$  represents the amount of intercalating agent used, 120/100 represents the cationic exchange capacity (CEC) of 120 meq/100 g of the bentonite, and 1.5 (> 1) is the alkyl amine/bentonite ratio and it indicates an excess amount of intercalating agent used. The molar alkyl amine/HCl ratio was 1:1. The cation exchange capacity, CEC, of the resulted bentonite was 120 meq/100 g.

Finally, the treated bentonite particles were collected by centrifugation and subsequently washed with deionized water several times to remove residual chloride or cations until no halides were detected in the filtrate by silver nitrate test. The dispersion and washing were accomplished using a 50/50 ethanol/water mixture. The filter cake was then placed in a vacuum oven at 80 °C for 24 h. The dried cake was ground and screened with a 325-mesh sieve to obtain the organophilic-inorganic material ODA-B.

### 2.3. Preparation of PU/organobentonite nanocomposites

A series of PU/ODA-B nanocomposites were prepared. The preparation process was as follows: various amounts (0.25, 0.50, 0.75, 1, 3 and 5 wt%) of ODA-B were swelled with Methyl Ethyl Ketone MEK and stirred for 4 h at 70 °C. The PEG/ODA-B mixture was blended for a particular PU/ODA-B nanocomposite with the calculated amount of toluene diisocyanates TDI (2 g), (NCO/OH ratio was 1:4). The mixture was stirred vigorously for 3 h at 70 °C. Then the PU/ODA-B nanocomposites were obtained when the viscous products were poured into molds and left for 2 days to ensure complete removal of MEK. The formed film was pulled from the mold and its thickness was measured using thickness gauge. The thicknesses of the samples were ranged from 1.5 to 2.5 mm. For tensile test the specimens were prepared in a rectangular bar with dimensions  $100 \times 3.5 \times 1.5\text{--}2.5$  mm.

### 2.4. Organo-bentonite characterization

Fourier-Transform Infrared Analysis FTIR was operated in the transmission mode, in the wave number range of 4000–400  $\text{cm}^{-1}$  by mixing with KBr powder on a Mattson 1000, series LC operating, Issue I (0791) spectrophotometer. The concentration of the samples in the KBr was held constant to 0.7% (w/w). The spectra were obtained using a resolution of 4  $\text{cm}^{-1}$  and were averaged over 100 scans. Standard software (Omnics ESP, version 5.1) was used for data acquisition and analysis. X-ray diffraction patterns of different forms have been investigated using a modern PANalytical diffractometer, Xpert PRO model. Nickel filtered copper radiation ( $\lambda = 1.542$  Å) was used. All

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