



Research paper

Characterization and comparison of thermal & mechanical properties of vermiculite polyvinylbutyral nanocomposites synthesized by solution casting method

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ABSTRACT

The aim of the study is to determine the effect of the vermiculite (Vk) and its organoclays, which were prepared by using cationic and anionic surfactants, on polyvinylbutyral (PVB) nanocomposites. The PVB nanocomposites were synthesized by using solution casting method, concerning the changes on the mechanical, and thermal properties of PVB. Modification of vermiculites with surfactant extends the interlayer space of the vermiculite and increases the interactions of vermiculite and PVB. The structural, morphological characterizations of PVB nanocomposites were examined by XRD (X-ray diffraction), and TEM (Transmission electron microscopy), and mechanical, thermal properties of the nanocomposites were determined by DMA (Dynamical mechanical analysis), and DSC (Differential scanning calorimetry) methods. The results showed that addition of vermiculite or modified vermiculite increased the elastic modulus of PVB. Modification of vermiculite with anionic surfactant resulted in a better exfoliation and improved the mechanical and thermal properties compared with modification of vermiculite with cationic surfactant in PVB nanocomposites.

1. Introduction

Clay mineral/polymer nanocomposites have received great attention in recent years because they offer considerable improvement in a wide range of properties for polymers with low filler loading. Mostly, clay minerals, such as montmorillonite, hectorite, synthetic mica, and vermiculite are used to prepare clay mineral/polymer nanocomposites. These types of clay minerals have around 1 nm size of a layer thickness and can easily be exfoliated in a polymer matrix with help of some modifications. The modifications are indeed crucial treatments to increase the compatibility of the polymer matrix and the clay mineral particles. After the modifications, the surfaces of clay mineral particles are turned into hydrophobic or organophilic surfaces. The interlayer space of clay minerals is expanded with cation exchange reactions, so the clay mineral particles can easily be dispersed and exfoliated in polymers (Theng, 1982; LeBaron et al., 1999; Alexandre and Dubois, 2000; Pinnavaia and Beall, 2000; Lagaly, 2001; Maiti et al., 2002; Yariv and Cross, 2002; Chang et al., 2003; Ray and Okamoto, 2003; Utracki, 2004; Kim et al., 2005; Usuki et al., 2005; Bergaya et al., 2006; Liu et al., 2006; Amarelis et al., 2007; İsci et al., 2007; El Mouzdahir et al., 2009; İsci et al., 2009; Seydibeyoglu et al., 2010; Qian et al., 2011; Ray, 2013; Gul et al., 2016).

Clay minerals are generally modified with cationic surfactants. The surfaces of clay minerals are mostly negatively charged so, positively charged heads of cationic surfactants interact electrostatically with the clay mineral, changing the surfaces to hydrophobic or organophilic ones. The interlayer space of these clay minerals expands as a result of cationic exchange reactions. Hence, cationic surfactants are the mostly used molecules to prepare organoclays for clay mineral/polymer nanocomposites applications (Lagaly et al., 1984; Lagaly, 1986, 1999; Bergaya and Lagaly, 2001; Lagaly, 2001). Anionic surfactants are almost never used to prepare organoclay. Because, the negatively charged head of the surfactant is attracted by the positively charged edges of the clay minerals and, does not contribute to the expansion of interlayer space. When the interlayer space of clay mineral expands, the 001 reflection is displaced to the lower angles. However, vermiculite show different intercalation behavior when subjected to a treatment of surfactants (İsci, 2017). Vermiculite generates a new XRD reflection as a result of the interaction with surfactant in addition to the angle shifts of the 001 reflection. The new reflection appears because of the rearrangement of the layers of vermiculite, called superstructure (Johns and Sen Gupta, 1967; Serratosa et al., 1970; Meisinger, 1985; Lee and Solin, 1991; Martins, 1992; Yariv and Cross, 2002; Slade and Gates, 2004). The rearrangement of the layers also can be achieved with

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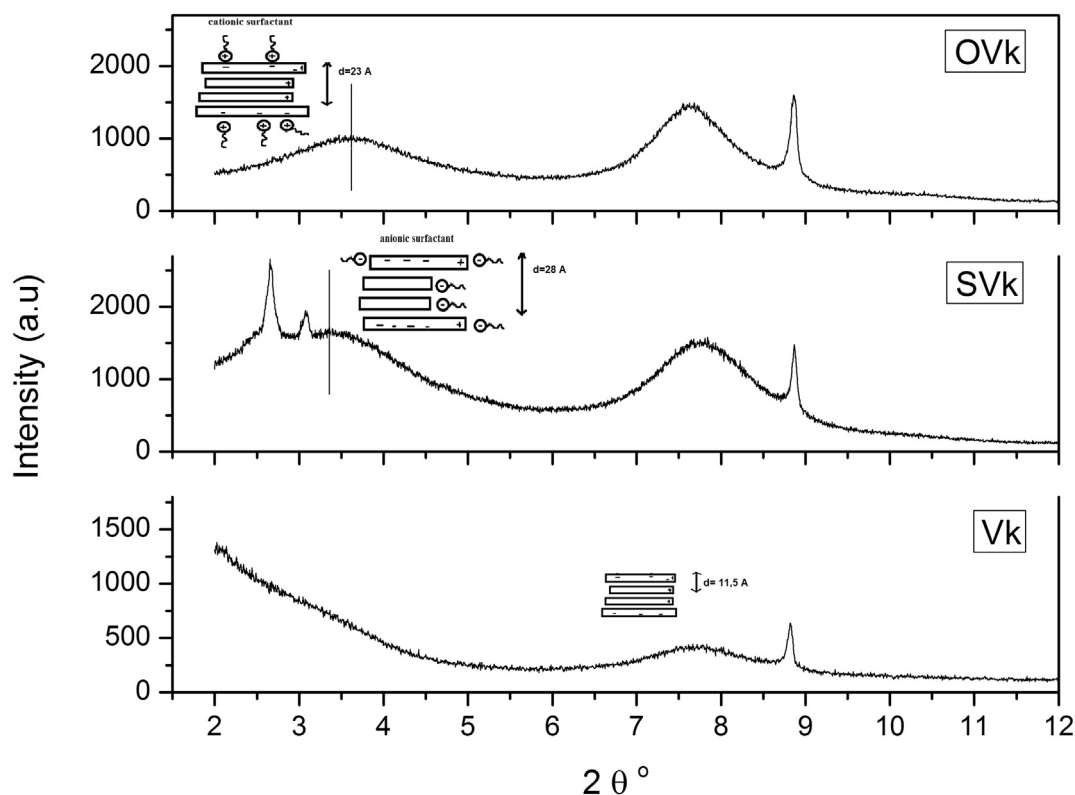


Fig. 1. XRD patterns of Vk, SVk and OVk.

anionic surfactants. Since the edges of vermiculite are positively charged, the interaction of negatively charged heads of anionic surfactant molecules result in superstructure of vermiculite more than cationic surfactant does (Johns and Sen Gupta, 1967; İsci, 2017).

PVB is a widely used polymer especially at the applications which requires strong binding and adhesion, flexibility, optical clarity. Addition of vermiculite does not change the appearance of the polymer but increases the mechanical strength and thermal stability. The modification of vermiculite increases the exfoliation capability of the clay mineral in PVB, like all the other clay mineral-polymer composites. However, modification of vermiculite with anionic surfactant creates a superstructure, with increase of interlayer space which exceeds that of cationic surfactants. The degree of exfoliation is higher for vermiculite modified with anionic surfactant, which results in higher mechanical strength and thermal stability.

In this study, polyvinylbutyral, PVB nanocomposites were prepared by solution casting with vermiculite and modified vermiculites. The vermiculite was modified with sodium dodecyl sulfate (SDS), or Octadecyltrimethylammonium bromide (ODTAbR) to prepare and compare the effects of the vermiculite and its organoclays in PVB nanocomposites. The prepared PVB nanocomposites with the vermiculite or the organovermiculites were characterized by XRD and TEM methods, and the effect of the superstructure of vermiculite on exfoliation was discussed. Besides, the effect of the exfoliation was discussed on the mechanical and thermal properties of PVB nanocomposites.

2. Experimental section

2.1. Materials

The vermiculite sample (Vk) obtained from Gold Butte District, Clark County, Nevada, USA. The vermiculite was subjected to a heat treatment at temperatures over 700 °C the mineral expands, multiplying its volume between 15 and 20 times, and the expanded form of

the vermiculite was used in this study.

Sodium dodecyl sulfate (SDS) and octadecyl trimethyl ammonium bromide (ODTAbR) was used to prepare organovermiculites as anionic and cationic surfactants respectively. SDS ($C_{12}H_{25}NaO_4S$) was purchased from Aldrich Chemical Co with molar mass $M_W = 288.38$ g/mol and ODTAbR ($C_{18}H_{35}N^+(CH_3)_3Br$) was purchased from Fluka with molar mass $M_W = 392.52$ g/mol.

Polyvinylbutyral was purchased from Aldrich. The average molar mass of PVB is between 90,000–120,000 g/mol.

2.2. Preparation of organovermiculites

Vermiculite samples were dispersed in water and the pH was adjusted to 4, to increase the adsorption capability of the clay mineral (Jozefaciuk and Bowanko, 2002; Bergaya et al., 2006). Then, 10^{-2} mol/l of each surfactant in water, were added to the clay mineral dispersion and ultrasonicated for half an hour. The dispersions were shaken for 2 days and then centrifuged and dried at oven at 50 °C. The dried organovermiculite samples were grinded and labeled as SVk for SDS addition, and OVk for ODTAbR addition. After the modifications, the surface charges of the prepared organovermiculites were determined by the zeta potential measurements. The zeta potential of unmodified vermiculite, SVk and OVk were determined as -29 mV, -43 mV and $+26$ mV respectively.

2.3. Preparation of nanocomposites

The Vk, SVk and OVk samples were dispersed in acetone 4% w/w and then shaken for 2 days for better swelling of clay minerals. 5 g of PVB in 100 ml acetone and the appropriate amount of the clay mineral dispersion were added then blended for 100 min at about 40 °C, so the final products contain 0.1, 0.5, 1, and 2% clay mineral in PVB. The products were dried in room temperature. The clay mineral/PVB films were obtained after the drying.

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