Microwave preparation of uniform CeO_2/Organo-Clay nanoparticles and its composition with resin epoxy

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Abstract

Organoclay is an organic-inorganic composite consisting of the layers of two tetrahedral silica sheets and one octahedral alumina sheet pillared by organic reagents to improve the physical and chemical properties. We report the synthesis of CeO_2/organo-clay nanocomposite with a uniform distribution of particles in morphology by a programmed method of microwave heating reaction. Cerium nitrate hexahydrate as an initial metal source was added to the dispersed organoclay in aqueous solution and then transferred into a domestic microwave oven with a power of 900 W for 20 min. The synthesized nanocomposite was characterized by using Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersion of x-ray spectrometry (EDX) techniques. XRD pattern clearly revealed the formation of the CeO_2/ organoclay nanocomposite having pillared planes. Scanning electron microscopy (SEM) images of the resulting product indicated a uniform morphology of nano-sized particles with an average size of 33 nm. The composition of the resulting product into a matrix of resin epoxy in different weight ratios was studied.

Keywords: Microwave, Green, Resin epoxy, OrganoClay.

1. Introduction

Composites are known as the macroscopic compounds containing several components with the distinct interfaces. In these compounds, a reinforced material is completely introduced into a surrounding matrix to improve the physical and chemical properties [1-4]. High mechanical strength to weight ratio, High resistance to corrosion, good thermal insulation properties, High strength and Low volume to weight ratio are enumerated as a series of advantages of the nanocomposites in different classifications. Ceramic matrix composites (CMC), metal matrix composites (MMC), polymer matrix composites (PMC) that is the most common categories of composites such as polyaniline, resin epoxy and *etc.,* are a classification of composite materials [5-7]

Metal oxide/Organoclay hybrid materials are employed as the reinforced materials in many nanocomposite because of having the potentially high-aspect ratio and high-surface area. In fact, metal oxides in the interlayer spaces of the layered silicates of clay can lead to form the practical products with the great enhanced properties. These materials are environmentally friendly and readily available in large quantities and have been accepted as a wide spectrum of inorganic reinforced compounds for organic matrixes [8-10].

In this work, we synthesized CeO_2/Organoclay nanoparticles by microwave heating technique, composed it with resin epoxy matrix in acetone solvent, painted on the metal substrate with a different coating thicknesses and characterized.

2. Experimental

2.1. Materials

Organoclay and epoxy resin were produced by Iranian companies. Other chemicals and reagents were purchased from the Fluka and Merck companies and used without further purification.

2.2. Synthesis of nanocomposite

2.2.1. Synthesis of CeO_2/organo-clay

An appropriate aqueous solution of $Ce(NO_3)_3.6H_2O$ (30 wt%) was added to a suspension of organoclay and stirred for 2h. Then, the prepared mixture was transferred into a domestic microwave oven with a power of 900 W and left for 20 min. The obtained product was centrifuged, washed several times with distilled water to eliminate any unreacted ions and dried at 80°C overnight.

2.2.2. Synthesis of CeO_2 /organo-clay/ epoxy resin nanocomposite

Epoxy resin was mixed with a chosen quantity of the aforementioned CeO_2/organo-clay nanocomposite by acetone as solvent with magnetic stirrer at room temperature for 10 min. Then, a stoichiometric amount of a curing agent (30 wt%) was added and stirring was continued for 10 min. In order to study the structural and morphological properties, the specimen were coated on a metal substrate and left at room temperature for 24 h to evaporate the solvent and dry.

2.3. Characterization

The powder XRD measurements were performed using STOE diffractometer with monochromatized Cu Kα radiation (λ=1.5418 Å). FTIR spectra were recorded on a Shimadzu-8400S spectrometer in the range of 400–4000 cm^{-1} using KBr pellets. Scanning electron microscopy (SEM) images and energy-dispersive X-ray spectroscopy analysis (EDX) to discover the elements present in the products were taken on a Philips XL-30 FESEM with gold coating.

3. Results and discussion

The XRD patterns of the raw organoclay and processed product are illustrated in Fig. 1. In clay X-ray pattern is observed three strong peaks at angels of 3.34º, 6.98º (group of several peaks) and 19.81º (2θ). These reflection peaks revealed the obvious shifts to lower angles of 1.26º, 6.27º and 19.6º (2θ), respectively and also, the a decrease in intensity of peak at angel of 3.34º and increase at angels of 6.98º and 19.81º, which confirm the linking of metal oxide species to organoclay sheets and produce the CeO_2/organoclay nanocomposite [8,9]. The identified signals at angels of 28.66º, 33.22º, 47.69º, 56.59º, 77.47º and 79.47º can be attributed to cerium oxide phase (JCPDS - 075-0076) incorporated by organoclay.

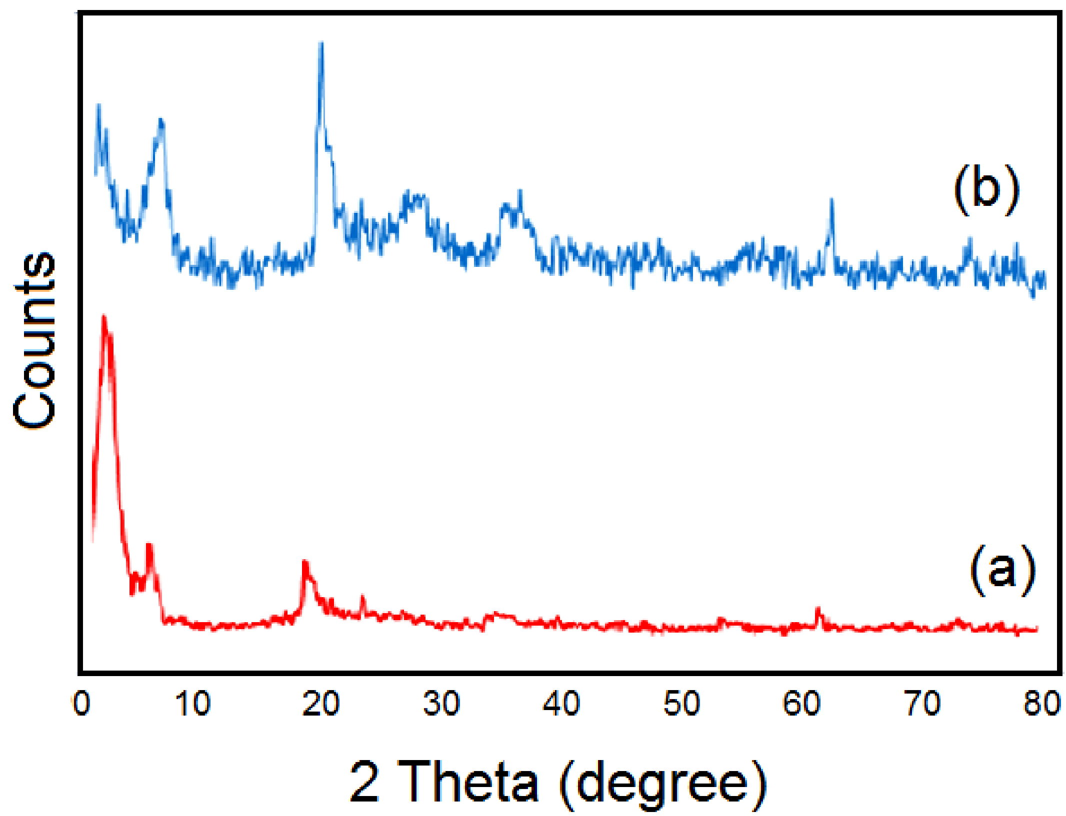

Fig. 1. XRD patterns of the organoclay (a) and cerium oxide/organoclay nanoparticles (b).

EDX analysis of the resulting product demonstrated the used stoichiometric amount of CeO_2 exist in nanocomposite. These data clearly revealed the hybridization of the CeO_2 between layers of clay, which can be a good evidence for the formation of cerium oxide/organoclay.

FT-IR spectra of the organoclay and prepared composite are indicated in Fig. 2. The organic functional groups of organoclay are observed in Fig. 2a. A decrease of these absorption bands is observed in FT-IR spectrum of nanocomposite (Fig. 2b), which may be related to substitute the cerium oxides nanoparticles instead of a part of organic groups exist in layered silicates of clay. The absorption bands below 500 cm^{-1} can be related to the vibration frequency of metal-oxygen.

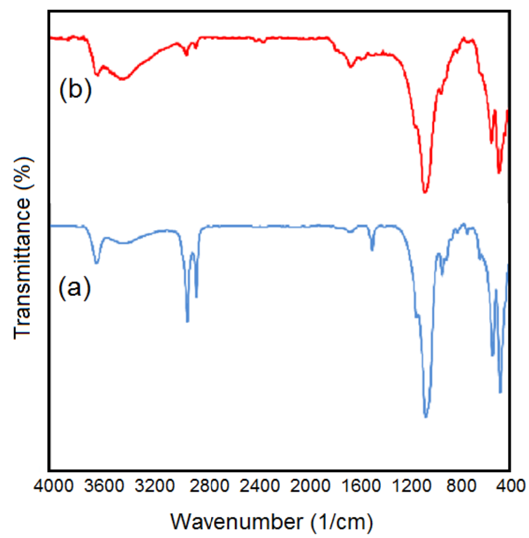

Fig. 2. FT-IR spectra of organoclay (a) and CeO_2/clay nanocomposite (b).

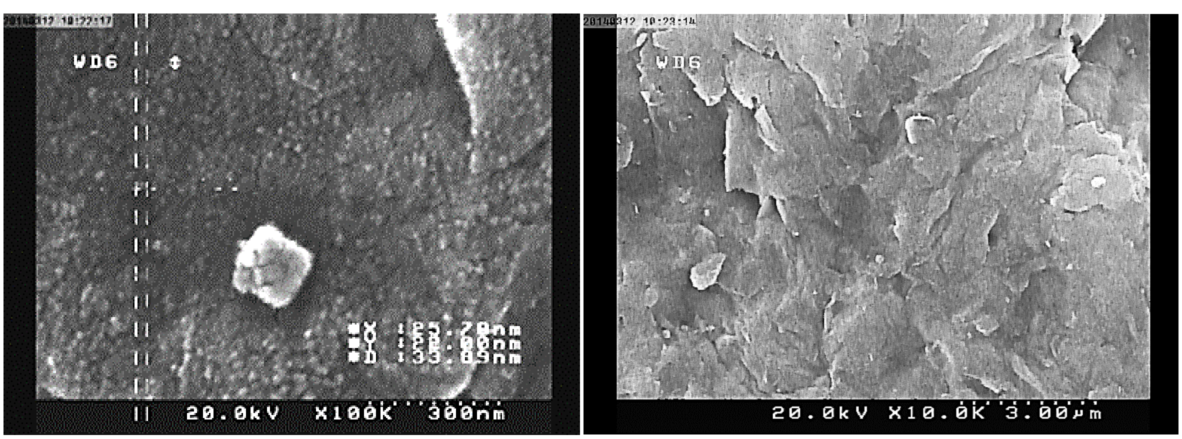

Fig. 3. SEM images of CeO_2/organoclay nanoparticles(left) and CeO_2/ organoclay/resin epoxy nanocomposites(right).

The morphological study of products was carried out by SEM images (Fig. 3). The SEM images presented the uniform distribution of metal oxide nano-sized particles in matrix of composite with an average particle size of 33 nm. In addition, SEM image of CeO_2/clay/resin epoxy showed a uniform distribution surface of inorganic nanoparticles in organic matrix of epoxy.

4. Conclusions

In a summary, CeO_2/organoclay nanocomposite was successfully synthesized by a facile microwave heating technique and then, the resulting product was inserted into the resin epoxy matrix to produce inorganic-organic hybrid compound. The resulting composite in nanoscale can be introduced as a high forcefully composite in comparison with pure CeO_2/organoclay or resin epoxy polymer.

Acknowledgments

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