

Surface modification of magnesium hydroxide using vinyltriethoxysilane by dry process



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ABSTRACT

In order to improve the compatibility between magnesium hydroxide (MH) and polymer matrix, the surface of MH was modified using vinyltriethoxysilane (VTES) by dry process and the interfacial interaction between MH and VTES was also studied. Zeta potential measurements implied that the MH particles had better dispersion and less aggregation after modification. Sedimentation tests showed that the surface of MH was transformed from hydrophilic to hydrophobic, and the dispersibility and the compatibility of MH particles significantly improved in the organic phase. Scanning electronic microscopy (SEM), Transmission electron microscopy (TEM) and X-ray powder diffraction (XRD) analyses showed that a thin layer had formed on the surface of the modified MH, but did not alter the material's crystalline phase. Fourier transform infrared (FT-IR) spectra, X-ray photoelectron spectra (XPS) and Thermogravimetric analysis (TGA) showed that the VTES molecules bound strongly to the surface of MH after modification. Chemical bonds (Si—O—Mg) formed by the reaction between Si-OC₂H₅ and hydroxyl group of MH, also there have physical adsorption effect in the interface simultaneously. A modification mechanism of VTES on the MH surface by dry process was proposed, which different from the modification mechanism by wet process.

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1. Introduction

In recent years, with the development of polymer material industry and the improvement of people's environmental consciousness, halogen-free alternatives flame retardants have garnered a considerable amount of attention from researchers and developers [1–4]. Magnesium hydroxide (MH), one such environmentally friendly flame-retardant, is already widely applied in halogen-free polymeric materials by virtue of its high decomposition temperature (about 340 °C, much higher than that of aluminium hydroxide at about 140 °C), smoke suppressibility, non-toxicity, and neutralization with acid gas from polymer combustion [5–7]. Now, MH with different morphological structures can be synthesized by several methods including the direct precipitation [8–10], magnesium oxide hydration [11], electrodeposition [12], hydrothermal [13], ultrasonics assisted synthesis [14], and so on. Whereas, MH has strong hydrophilicity and it is inclined to

agglomerate due to its strong polar. Therefore, as filler, MH has a poor compatibility with polymer and unevenly disperses in the matrix thus affecting the mechanical properties of the composite [15]. MH also has relatively low flame retardant efficiency. The filling content of MH must be higher than 60 wt.% to provide adequate flame retardancy to a polymer composite. Such high loading often causes unacceptable decline in mechanical properties and rheological properties of the resultant composites [16]. Surface modification may be a simple and effective way of mitigating these disadvantages.

Commonly used surface modification methods for MH can be loosely divided into three classes: on-line modification, dry process and wet process methods. The on-line modification could short the process line and obtain ultrafine MH, due to modification and preparation of MH can be acted on simultaneously [17,18]. In the case of dry process and wet process methods, there are versatile surface modifiers, such as fatty acids, titanate coupling agent, silane coupling agents, and so on [19–22], can be chosen for prepared MH. Silane coupling agent has gained more research attention due to its unique structure, which contains two different functional groups: one that is attracted to the resin, and the other that reacts with hydroxyl groups on the surface of the filler [23–25]. It has been reported that MH can also be successfully modified with silane coupling agents. Luo et al. [26], for example, modified

Abbreviations: MH, magnesium hydroxide; VTES, vinyltriethoxysilane; VTES-modified MH, magnesium hydroxide modified with vinyltriethoxysilane.

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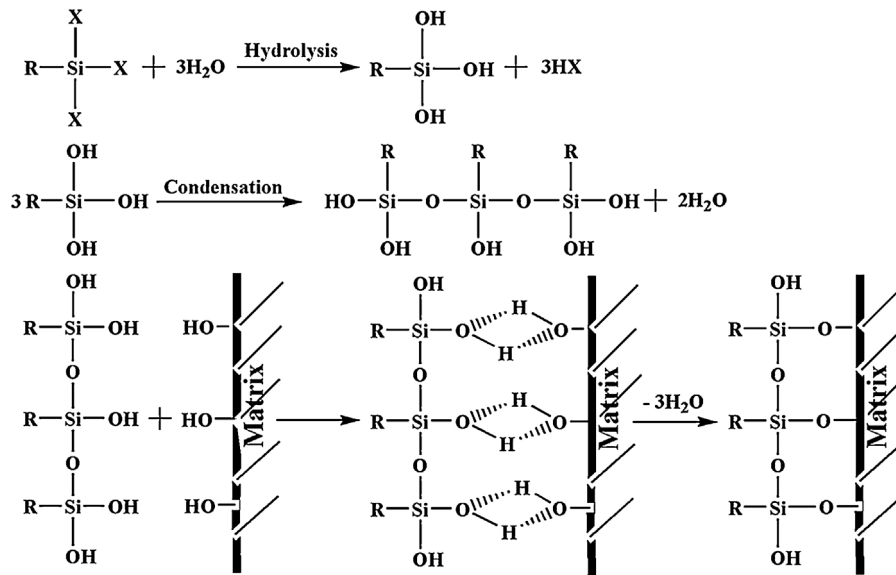


Fig. 1. Action mechanism of silane coupling agent on inorganic filler surface by wet process.

MH by silanization reaction with γ -aminopropyltriethoxysilane (γ -APS) in aqueous solution at different pH values and found that γ -APS hydrolysis and condensation are activated in alkaline solution, facilitating multilayer adsorption of γ -APS molecules on the surface of MH. Yuan et al. [27] modified MH with Trimethoxy[3-(phenylamino)propyl]silane by wet process and found that both the dispersivity and lipophilicity of modified MH improved remarkably. Schofield et al. [28] modified MH with ammonium stearate and γ -aminopropyltriethoxysilane (γ -APS) and found that APS treatment enhanced both tensile strength and elongation compared to untreated filler.

Most reports on MH surface modification using silane coupling agents adopted wet processes and related modification mechanisms. Dry process methods, conversely, though simple and easily industrialized [29], have been rarely researched. In this study, we modified the surface of MH using VTES (a silane coupling agent) by dry process, then measured the performance of the modified MH powder and the interfacial interaction between MH and VTES. The modification mechanism at work in the dry process we propose here differs substantially from that of the wet process, as discussed in detail below.

2. Experimental

2.1. Materials

MH powder (H-5) with average diameter of 1.25 μm , specific surface area (BET) of 130 m^2/g and purity of 99.8% was provided

by Albemarle Corporation. Silane coupling agents (VTES) was purchased as commercial grade from Jingzhou Jiangnan Fine Chemical Co., Ltd. Paraffin liquid was chemically pure and used as provided without further modification.

2.2. Surface modification of MH

Modification of MH powder was proceeded according to the following steps. The MH powder was first dried at 110 $^\circ\text{C}$ for 12 h to eliminate any absorbed water on the surface of the powder. Then dried MH powder was then added into a high-speed mixer (Type Y100L2-4, Jiangsu, China), at 100 $^\circ\text{C}$ temperature and 1000 rpm/min stirring rate. Once reaching the set-up temperature, VTES was slowly added into the high-speed mixer by disposable syringe and mixed continuously for 10 min.

2.3. Characterization

The morphologies of original and modified MH were observed with a JSM-6701F field emission scanning electron microscope (FESEM) from JEOL. The phase and crystallographic structures of the samples were examined by X-ray diffraction (XRD) on an X'Pert PRO (PANalytical B.V.) powder diffractometer working with $\text{CuK}\alpha$ radiation ($\lambda = 1.54178 \text{ \AA}$) at a scan rate of 0.05 $^\circ/\text{s}$. Thermogravimetric analysis (TGA) was carried out on a STA449F3 thermal analyzer (Netsch, Germany) from 30 $^\circ\text{C}$ to 1000 $^\circ\text{C}$ at a heating rate of 10 $^\circ\text{C}/\text{min}$ in N_2 atmosphere. The infrared spectra of original and modified MH were measured with an FT-IR spectrometer

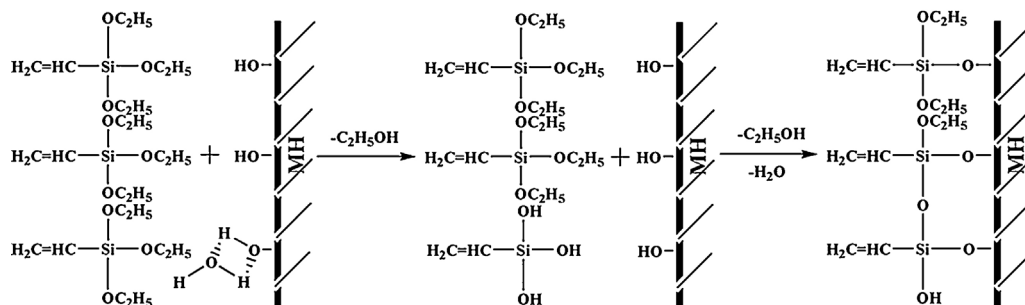


Fig. 2. Action mechanism of VTES on MH surface by dry process.

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