



Layered double hydroxides: Efficient fillers for waterborne nanocomposite films



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ABSTRACT

Using Layered double hydroxides (LDH) nanoparticles and film-forming latexes, waterborne nanocomposite films were produced by simply a heterocoagulation and solvent casting process. Self-standing and transparent films with LDH contents from 2.5 to 15 vol% were prepared. The structure, microstructure and mechanical behavior were thoroughly investigated by powder X-ray diffraction (PXRD), infrared spectroscopy (IR), transmission electron microscopy (TEM), scanning electron microscopy (FIB–SEM) and dynamic mechanical analysis (DMA). Favorable electrostatic interactions between pristine LDH and the latex ensured a good dispersion of the bidimensional LDH platelets in the films. Above a certain content of LDH, the formation of a well-defined cellular LDH network following the starting latex morphology was observed. Such a percolating microstructure induces a large mechanical reinforcement significant of a mechanical percolation behavior.

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1. Introduction

Layered double hydroxides (LDH) are anionic clay minerals displaying a brucite-like structure in which some of the divalent metal cations are substituted by trivalent metal cations (Rives, 2001; Duan and Evans, 2006; Forano et al., 2013). The resulting net positive charge in each layer of the LDH structure is balanced by anions in the interlayer spaces. LDH can be easily prepared in the laboratory with a large variety of chemical composition by modifying the nature of the metal cations or the intercalated anions. Different synthetic approaches including coprecipitation using basic agents or retardant base, electrogeneration, the polyol route and the sol–gel process... have been developed to tune the composition, size, shape and nanostructuration of the LDH layers (Wang and O'Hare, 2012; Forano et al., 2013).

The high and tunable charge density, high aspect ratio, high anion exchange capacity, and enormous compositional and dimensional diversity of LDH which is accessible by simply varying the nature and ratio of the feed ions and synthetic conditions, make them exciting candidates for a variety of applications. LDH have received great attention

for use in many fields (Costantino et al., 2013) ranging from adsorption of pollutants for water treatment (Ma et al., 2015), carbon dioxide capture (Fu et al., 2014), photo/bio catalysis (Li et al., 2014; Xiang et al., 2014), drug release (Rives et al., 2014), gene therapy (Choi et al., 2014), biosensor development (Mousty and Prevot, 2013), ... Since it was discovered in the early 1990s that the dispersion of clay mineral layers could enhance polymer matrix properties (Yano et al., 1993), great attention has been paid to the use of various layered materials as fillers (Giannelis, 1996; Okamoto, 2006; Mittal, 2009). Since LDH layers display a high aspect ratio in the nanometer size range, their potential as efficient nanofillers for the fabrication of clay mineral polymer nanocomposites with enhanced mechanical, thermal and flammability properties has been recognized (Leroux and Besse, 2001; Leroux et al., 2012; Matusinovic and Wilkie, 2012; Basua et al., 2014) and is the subject of the present work. Compared to layered silicates such as montmorillonite, laponite, and kaolinite, which are the most heavily studied filler materials, LDH offer distinct advantages including higher anion exchange capacity, higher hydroxyl and water contents and more easily tunable structural homogeneity. LDH has accordingly been incorporated into various thermoplastics, thermosets and elastomers (Leroux and Besse, 2001; Leroux et al., 2012; Matusinovic and Wilkie, 2012; Basua et al., 2014) to serve as a stabilizers, halogen scavengers or nanocarriers for functional agents (e.g., anti corrosion agents, anti-oxidants, dyes and photostabilizers...) (Feng et al., 2006; Perioli et al., 2006; Taviot-Gueho et al., 2007; Coelho et al., 2012; Stimpfling et al., 2013).

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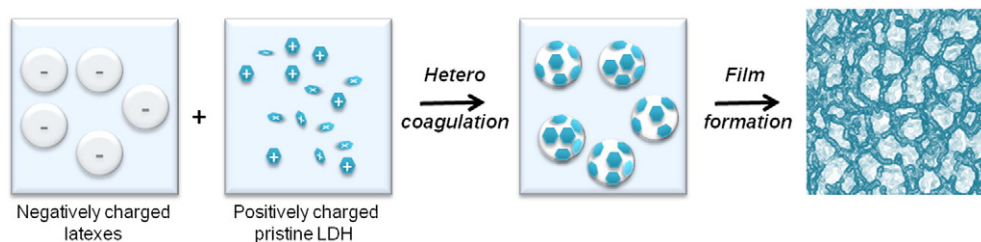


Fig. 1. Schematic illustration of the LDH/P(MMA co BA) nanocomposite film formation.

In most of these applications highly dispersed LDH platelets are desirable to maximize the interfacial area with the surrounding polymer. In order to achieve this, LDH (which are highly polar and therefore not naturally compatible with non-polar materials), are first modified to increase their compatibility with the polymer matrix sometimes exfoliated to convert the layered structure into individually dispersed single layers. Compatibility can be increased by electrostatically adsorbing or chemically grafting molecules onto the surface of the particles (Illaik et al., 2008; Zhu et al., 2008; Kovanda et al., 2010). Various strategies including *in-situ* polymerization, solution mixing and melt blending have then been used to produce LDH polymer nanocomposites.

In the polymer community, especially in the field of coatings, latex blending (Steward et al., 2000) is well-known as an efficient sustainable waterborne route to produce polymer nanocomposites (Keddie and Routh, 2010). A particular advantage of this technique is that the nano-scale dimensions of the polymer latex and the filler particles are retained in the resulting nanocomposites, favoring the formation of

nanostructured materials and providing access to percolating networks for superior mechanical, electrical, and barrier properties. To promote colloidal stability and physical blending, polymer and surfactant are usually used to modify the filler surface. Carbon nanotubes (Wang et al., 2008) and graphene (Noël et al., 2014) have been for instance incorporated into latex-based nanocomposites to produce electrically conductive materials. Alternatively, latex-based nanocomposite films can also be formed using hybrid particles. Negrete-Herrera et al. (2007) reported on the use of armored latex with laponite platelets (Bon and Colver, 2007; Bourgeat-Lami et al., 2010) to generate clay mineral-containing nanocomposite films with a cellular microstructure.

In the present work, we describe the synthesis of waterborne LDH-containing nanocomposite films by heterocoagulation of positively charged LDH layers and negatively charged latex particles and subsequent drying of the dispersion. Pristine non-organically modified LDH layers were used in this process without stabilizing agent in the medium. To investigate the formation of a cellular network into the film, different LDH contents were used in the process. Firstly the structure and

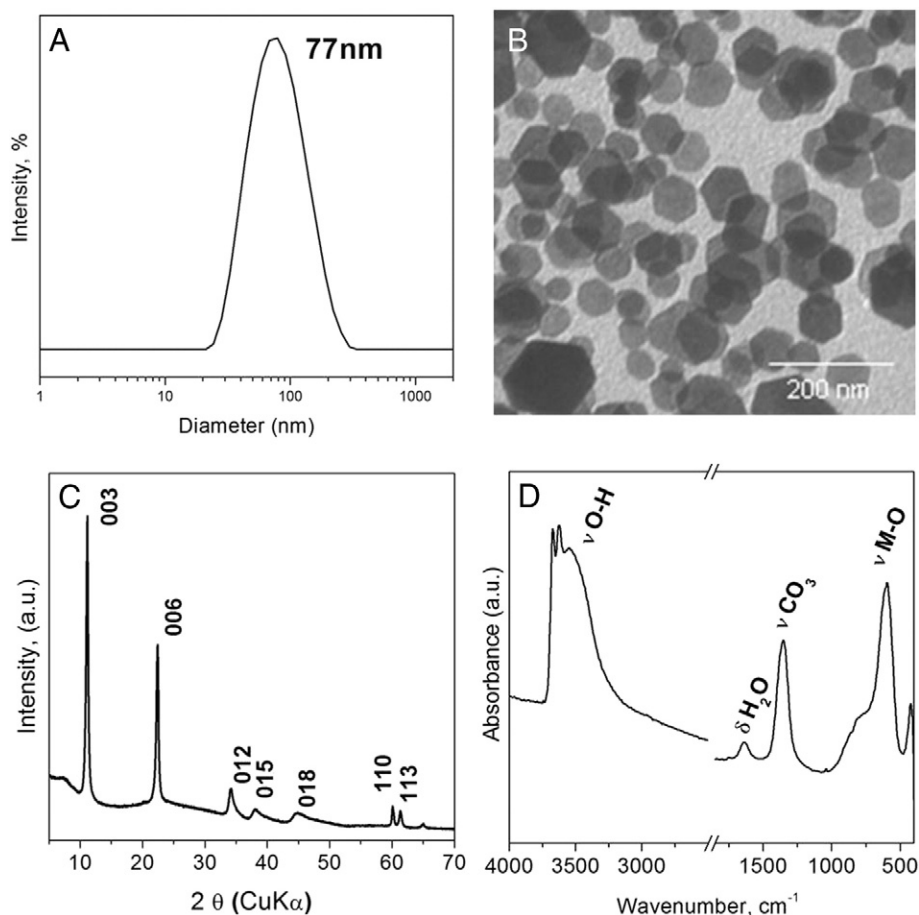


Fig. 2. A) DLS measurement, B) TEM image, C) PXRD pattern and D) FTIR spectrum of MgAl-CO₃ pristine LDH nanoparticles.

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