

Toward high performance epoxy/halloysite nanocomposites: New insights based on rheological, curing, and impact properties



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ABSTRACT

The present work is aimed to study the effects of various processing parameters such as mixing methods (ultrasonication and mechanical mixing), curing system and types of halloysite on the rheological, curing and impact behavior of epoxy nanocomposites reinforced with halloysite nanotubes (HNTs). It was shown that proper design of process and material is of crucial importance to get the full benefit of HNTs as reinforcing nanofiller. The viscosity of the uncured epoxy increased after the incorporation of HNTs, while the impact properties of the cured epoxy/HNTs nanocomposites were found to be highly dependent on the characteristics of base epoxy matrix, as determined by changing the type of curing agent. Compared to the commonly used mechanical mixing method, ultrasonication effectively improved the dispersion of HNTs in epoxy matrix, and considerably enhanced the viscosity and impact strength of the epoxy/HNTs nanocomposites, especially at high HNTs loading. Fracture mechanism studies revealed that shear yielding happened in the epoxy and the incorporation of HNTs deteriorated the impact properties of final epoxy nanocomposites. Types of HNTs had a significant effect on the impact properties of epoxy/HNTs nanocomposites in a way that those nanocomposites reinforced with longer and more uniform HNTs showed significantly higher impact strength.

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1. Introduction

Epoxy based nanocomposites have attracted extensive research in the past decades on improving the inherent brittleness of the epoxy resin. Various kinds of nanofillers such as carbon nanotubes (CNTs) [1], layered silicate clays [2], nanosilica [3] and graphene [4] have been used to reinforce the epoxy matrix, and considerable improvement in the mechanical, thermal, and electrical properties was reported. However, drawbacks like high cost, difficulty in mixing and complicated processing methods restricted the large scale production of epoxy nanocomposites. The common method for preparation of epoxy nanocomposites involves mixing of nanofillers with liquid epoxy resins followed by addition of hardener, molding and curing of the mixture to obtain a crosslinked product. Achieving a good dispersion of nanofillers in the epoxy resin has been the key challenge for synthesis of epoxy nanocomposites with optimum mechanical and electrical properties. Various techniques such as mechanical mixing [5,6], ultrasonication [7,8], high-shear mixing [9,10], roll milling [11,12], ball milling [6,13] and solvent assisted mixing [14,15] have been used to achieve a well dispersed

epoxy/nanofiller nanocomposite. However, finding an efficient, scalable, and economical mixing method to achieve the best dispersion and optimum properties for a specific type of nanofiller is still a matter of research. Another challenge in preparation of epoxy nanocomposites is that incorporation of nanofillers changes the rheological and curing properties of epoxy [16]. The rheological and curing characteristics play a crucial role in the processing and mechanical features of such nanocomposites. Therefore, understanding these issues is necessary for the optimization of the processing and properties of the epoxy nanocomposites.

Incorporation of nanofillers into epoxy generally results in the viscosity built-up and shear-thinning behavior of epoxy nanocomposites, due to the frictional interactions [17–21]. Dispersion of nanofillers affects the rheological properties of epoxy nanocomposite, too. Well-dispersed nanocomposites exhibit higher viscosity, which is attributed to higher interactions between nanofiller and epoxy and increased frictional forces [18,20,22,23]. So, the rheological studies can be used as a simple method for characterization and controlling the dispersion of nanofillers in the epoxy matrix [18,19,22,24]. Rheological behavior of uncured resin is also important from the processing point of view for manufacturing of thermoset materials in applications such as coatings and fiber reinforced composites [25–27].

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The curing behavior of epoxy is also affected by incorporation of nanofillers. Incorporation of CNTs catalyzed the curing reaction of epoxies and lowered the initiation temperature of curing [28,29], while the overall curing process was slowed down and the degree of curing reduced accordingly [29]. Modification of CNTs could change the curing behavior of epoxy/CNTs systems [30–33]. Besides the interaction of CNTs with epoxy and curing agent, the dispersion of CNTs could also affect the curing behavior of the prepared nanocomposites [28–33]. Clay nanofillers were also reported to affect the curing of epoxy resins [34–38]. In layered silicate clays, not only incorporation of nanofiller and its dispersion affected the curing behavior of epoxy resin, but also the curing properties of the epoxy influenced the dispersion state (exfoliation and intercalation) of the nanoclays [39–42]. The extent of exfoliation of the clay in epoxy systems was controlled by the balance between the diffusion rate of the curing agent into the clay galleries and the extra gallery reaction rate of the epoxy system [39–42].

Among nanofillers, halloysite nanotubes (HNTs) are clay minerals that occur naturally in the form of nanotubes and they resemble

the structure of multi-wall CNTs. Incorporation of HNTs into epoxy resins remarkably increased the impact toughness of epoxies [5,43]. It was found that besides isolated HNTs, clusters of HNTs would be formed in epoxy/HNTs nanocomposites which can deteriorate the mechanical properties of the nanocomposite. Deng et al. [6] used ball mill mixing to improve the dispersion of HNTs in epoxies, which led to further enhancements of strength, modulus and glass transition temperature (T_g) [6,44]. The ball mill homogenization used for mixing of HNTs and epoxy was rather complicated (masterbatch route) and time consuming (25 h for preparation of ball milled epoxy/HNTs master batch) [6]. Hence, finding better mixing methods to attain the best process-property balance is essential.

HNTs considerably changed the curing and rheological behavior of rubbers such as ethylene propylene diene monomer (EPDM) [45], styrene butadiene rubber (SBR) [46], and natural rubber (NR) [47]. However, the effects of HNTs on curing and rheological properties of epoxy nanocomposites have not been fully studied yet. As the curing behavior and rheological properties can affect

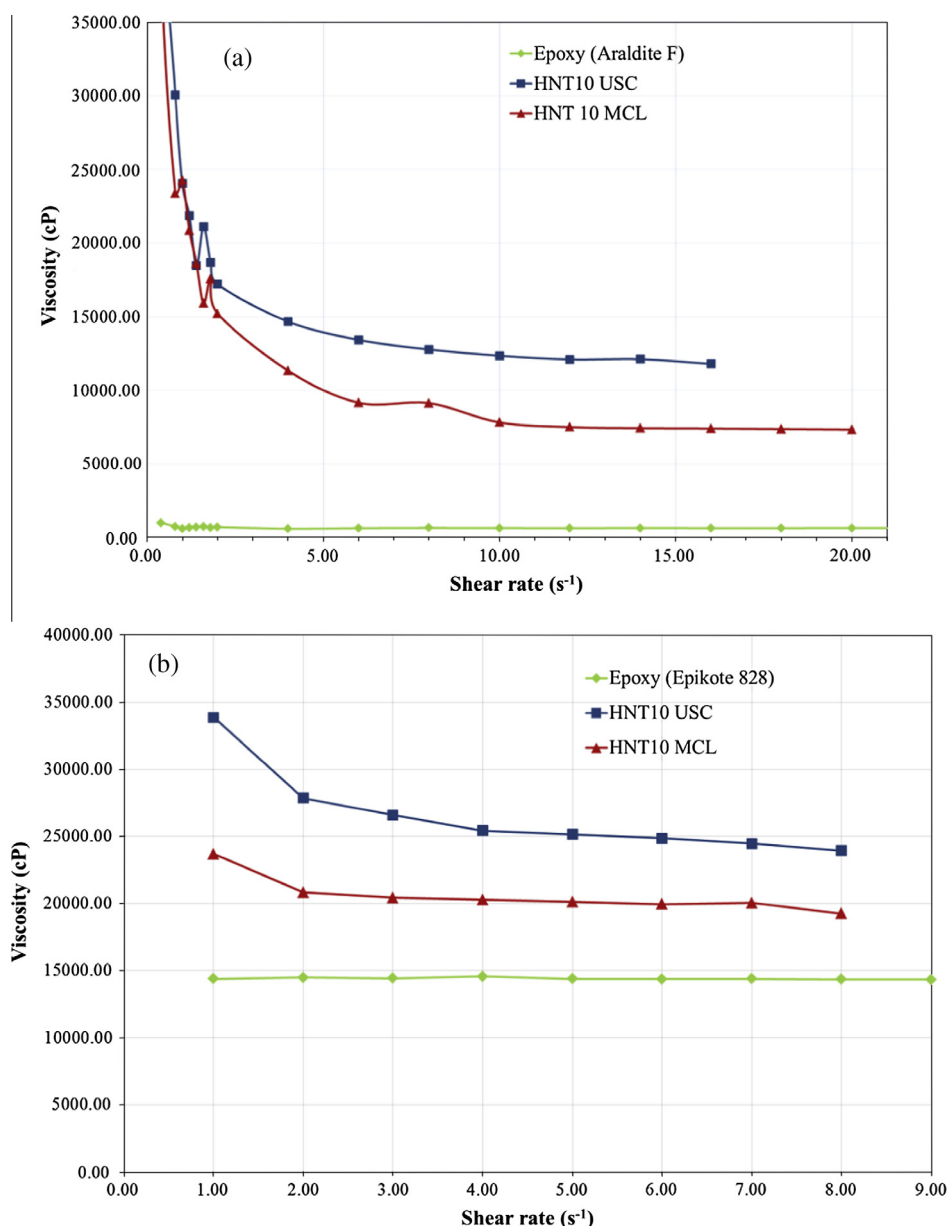


Fig. 1. Typical flow behavior of epoxy/HNTs nanocomposites: (a) low viscosity (Araldite F) and (b) medium viscosity (Epikote 828), USC: Ultrasonically mixed, MCL: Mechanically mixed.

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