Characterization of Nanoclay Dispersion in Epoxy Matrix by Combined Image Analysis and Wavelength Dispersive Spectrometry

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1. Introduction

Nanoclay has been gaining acceptance as a nano-meter scale reinforcement for polymers during the last two decades [1]. It has proven to be successful for reinforcing thermoplastics [2], however, its utilization in thermosetting resins has been problematic. To improve dispersion of nanoclay into thermosetting resins, a number of companies developed surface modifications, which replace the sodium ions with larger organic molecules. In the current study, we are investigating the dispersion characteristics of three commercially available nanoclays from Southern Clay Products Inc., by combining microscopic image analysis and wavelength dispersive spectrometry.

2. Experimental Studies

Three nanocomposite samples, each containing 5wt.% of nanoclay –either Cloisite® 15A, 25A or 30B- are fabricated to analyze their dispersion characteristics. Initially, the desired type of nanoclay is mixed with EPON 815C epoxy resin in a sonicator at 50°C for 60 minutes. Following the mixing period, the resin/nanoclay compound and EPI-CURE 3282 curing agent are placed in a custom made molding setup, and molded into a center-gated disk. The final cured parts are 152.4mm in diameter and approximately 3mm-thick. A radial section is cut out from the cured parts and used to analyze the dispersion of nanoclay along the radius of the disks. Nanoclay clusters larger than 1.5μm are analyzed by performing image analysis on the scanning electron micrographs captured along the radius of the disks, whereas smaller clusters are characterized by wavelength dispersive spectrometry (WDS). A sample SEM image indicating nanoclay clusters after image processing is depicted in Figure 1. The micro-structure of nanoclay clusters is studied by transmission electron microscope (TEM) images.

3. Results and Discussion

A total of 42 SEM images are analyzed to determine the radial volume content distribution of nanoclay clusters which are larger than 1.5μm, and the results are depicted in Figure 2. The distribution of nanoclay contents in the radial direction did not indicate any particular trend. The average nanoclay content is calculated as 4.63%, 3.39%, and 3.45% for nanoclay types 15A, 25A, and 30B, respectively. For a more thorough analysis, nanoclay clusters are divided into four different size groups, and percent contribution of each group to the overall number of clusters with respect to the inlet of the disk is depicted in Figure 3. It can be concluded from Figure 3 that clusters towards the outer edges of the disks are more likely to be smaller than the ones closer to the inlet. This may result from the breakdown of nanoclay cluster during mold filling due to the flow kinematics. Figure 4 depicts contribution of large and small clusters to the overall nanoclay content. It can be observed that the most effective dispersion state is achieved when 30B is used as the nanoclay in the epoxy matrix. For this particular type of nanoclay, small clusters contributed 42.3% to the overall nanoclay content, whereas contribution of large clusters is limited to 8.4%, the least of all three nanoclay types. Wavelength dispersive spectrometry is used to identify nanoclay clusters that are not covered by image analysis (clusters smaller than 1.5μm) and the results are depicted by a box and whisker plot in Figure 5. The nanoclay contents obtained from WDS is complementing the results obtained from image analysis. A detailed structure of a clay cluster is investigated by TEM. As indicated in Figure 6, extensive void formations and non-homogenous curing is observed. Non-homogenous curing may be caused by the surface modification of the resin [3].

4. References

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2. Y. Kojima, A. Usuki, M. Kawasumi, A. Okada, Y. Fukushima, T. Kurauchi and O. Kamigaito, *Mechanical Properties of Nylon 6-Clay Hybrid*, J. Mater. Res., **8(5)**, pp. 1185-1189 (1993)
3. J. Park and S. C. Jana, *Adverse Effects of Thermal Dissociation of Alkyl Ammonium Ions on Nanoclay Exfoliation in Epoxy Clay Systems*, Polymer, **45(22)**, pp. 7673-7679 (2004)

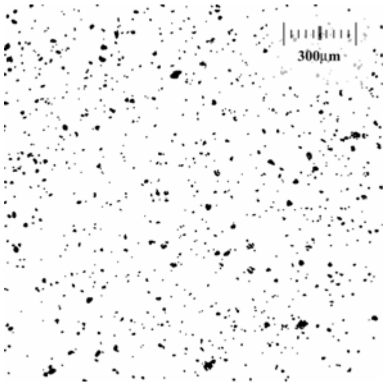

Figure 1: A sample SEM image after image processing.

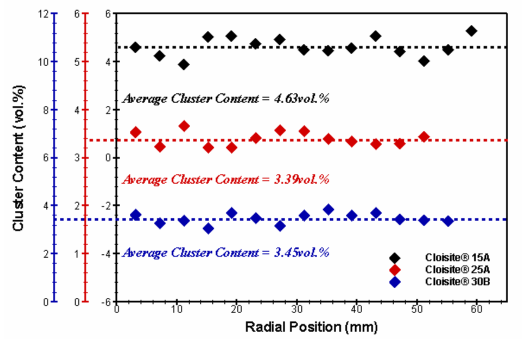

Figure 2: Radial distribution of nanoclay cluster larger than 1.5µm.

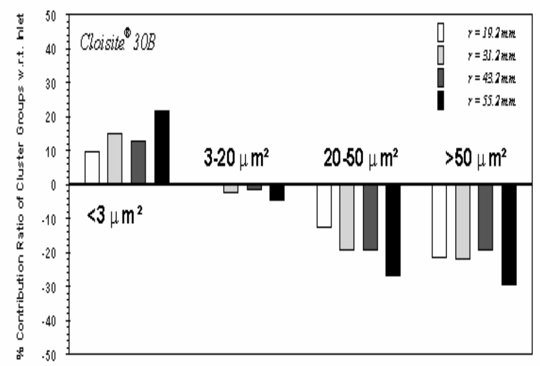

Figure 3: Percent contribution ratio of different cluster sizes w.r.t. the inlet.

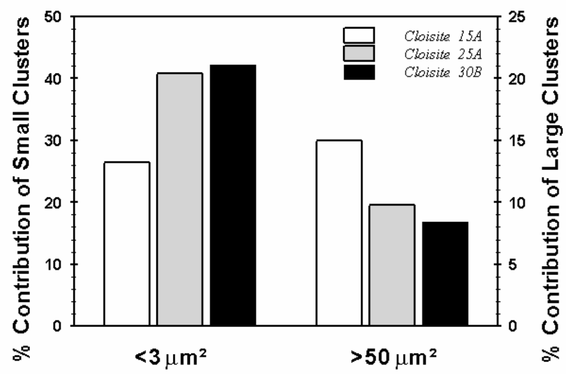

Figure 4: Overall percentage of small and large nanoclay clusters.

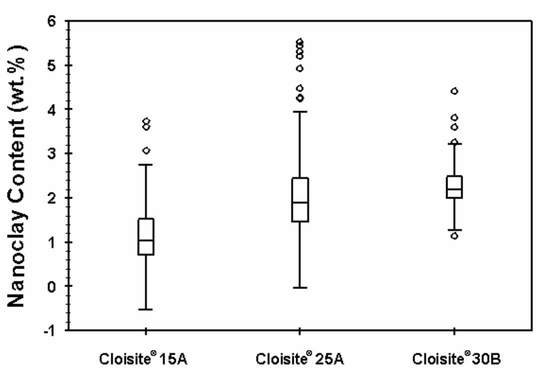

Figure 5: Box and whisker plot summarizing the WDS data.

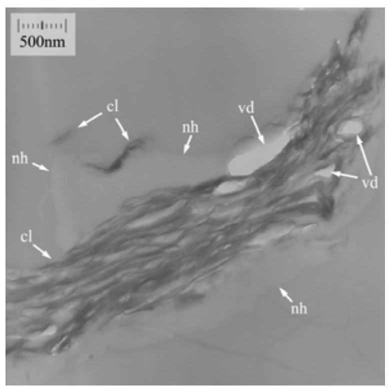

Figure 6: TEM image of a nanoclay cluster. cl: cluster, vd: void and nh: non-homogenous curing.