

Department	Functional Materials Engineering	Supervisor	Tsutomu Takeichi
Name	Tomotaka Saito		

Abstract

Title	INTERCALATION KINETICS, AND NOVEL PREPARATION METHOD FOR POLYMER NANO-COMPOSITES
-------	--

(800 words)

In the last two decades, polymeric nano-composite is one of the fast-growing category of material science on a worldwide basis. Getting attention on importance of nano-technology today, some results of polymeric nano-composites have been industrialized in many countries.

Polymer nano-composite consists of polymer matrix and nano-sized filler which has at least one of the dimension is of the order of a nano-meter, and the filler is dispersed into polymer matrix in nano-scale. First report of the material was from Toyota Central Research & Development Laboratories' (TCRDL) group for polyamide 6/clay nano-composite via in-situ polymerization of ϵ -caprolactam, in which alkylammonium-modified montmorillonite (MMT) was thoroughly dispersed in advance. The resulting composite with loading only 4.2 wt% clay possessed a doubled modulus, a 50%-enhanced strength, and an increase in heat distortion temperature of 80 °C as compared to that of neat polyamide 6.

The main reason for these improved properties in nano-composites is interfacial interaction between matrix and OMLF as opposed to conventional composites. A few weight percent of OMLF are properly dispersed throughout the matrix thus creates a much higher surface area for polymer-filler interfacial interactions than do conventional micro-composites. In case of the nano-particle use as nano-filler, the key technology is dispersion technique, in other words, preparation method for nano-composite materials.

Continued progresses in nano-scale controlling have contributed to the rapid development of polymer/OMLF nano-composites. Three types of main approaches for the polymer nano-composite preparation are reported, i.e., in-situ polymerization, intercalation of polymer or pre-polymer from solution (solution cast), and melt compounding.

In case of preparation for non-polar or weak polar polymer-based nano-composites, compatibilizer was added through melt compounding process to apply shear stress to OMLF layers. In other words, non-polar polymer-based nano-composite preparation without compatibilizer is not possible through above three methods. To solve this issue, we should investigate a novel preparation method, from industrial point of view, which is able to apply to the practical industrial production system.

The main objective of this study is to bring out the factors that concerned with nano-composite formation and to feed back these factors for the preparation of the polymer nano-composite. For this purpose, we investigated fundamental study of the phase transition of the cationic surfactant located at the interlayer space in montmorillonite, and examined the intercalation behavior of the small molecules by using various types of nano-fillers, which have

strong impact on the explanation for the intercalation kinetics. We chose an optimal OMLF and conducted the Poly(p-phenylenesulfide) (PPS)-based nano-composites preparation with and/or without shear processing. Furthermore, we have developed more innovative compounding process, i.e., solid-state processing, to delaminate the stacked-and-layered filler in the polymer matrix. And we adopted non-polar polymer as matrix to confirm the processing ability, and to investigate the effect of polarity of the matrix polymer on solid-state processing. The present thesis consists of the following 6 chapters.

In Chapter 1, the previous reported papers are totally reviewed about the polymer nano-composite studies and preparation methods for the materials, from which the unsolved problems are picked up and the motivation of the present research will be clarified.

In Chapter 2, we investigated nonisothermal order-disorder transition behavior of alkylammonium ions in nano-confined space i.e., interlayer space of MMT, compared with crystallized dioctadecyl dimethyl ammonium bromide (DC₁₈DM-Br) through temperature modulated differential scanning calorimeter, wide-angle X-ray diffraction (WAXD), and Fourier transform infrared spectroscopy (FTIR).

In Chapter 3, we investigated the intercalation behavior of small molecules, as a model compound for PPS, into various OMLFs interlayer evaluated from WAXD analysis. In the first part of Chapter 3, the effect of miscibility between small guest molecules and intercalants on the basis of DSC analysis was investigated. After that, the intercalation behavior of the molecules into the various types of OMLFs was discussed. In the remaining part, we chose an optimal OMLF and conducted the PPS-based nano-composites preparation with and without shear processing.

In the first part of Chapter 4, we discussed intercalation behavior with various polymers and OMLFs prepared by melt compound. The results show same trends as small molecules investigated in Chapter 3. In latter part, novel preparation methods, solid-state processing, to improve nano-filler dispersion and to delaminate the stacked-and-layered filler in PPS matrix was conducted. From the results, effectiveness of solid-state processing is clearly showed.

In Chapter 5, we also challenged to prepare non-polar polymer based nano-composite. It is considered that preparation for non-polar polymer-based nano-composite is difficult because polarity of matrix polymer is very important for nano-filler dispersion. Fortunately, we successfully prepared polypropylene/OMLF nano-composite with solid-state processing. In this study, two thresholds during solid-state processing were found for OMLF dispersion.

Finally, we summarized the principal results and conclusions derived from this study in Chapter 6.