

Synthesis and Luminescence Properties of Preparation of Organic/Inorganic Polymer Hybrid from Novolac Derivatives

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Recently, much attention has focused on the preparation of nano-ordered hybrid material consisting of both organic and inorganic moieties using the sol-gel technique. Especially, the preparation of a polymer hybrid by utilizing physical interactions (hydrogen bond) is convenient method. For example, Chujo & Saegusa reported the preparation of a transparent film from alkoxy silane with poly(2-methyl-e-oxazoline) or polyvinylpyrrolidone.¹ These materials showed significant properties such as a high thermal stability, film-forming property, and good mechanical property due to the nano-ordered combination. They are also expected to reveal several significant functions from each starting material.

Chujo & Tamaki previously reported a preparation of the polystyrene-silica gel polymer hybrid from an arylalkoxysilane utilizing the aromatic π - π interaction.² This method is a useful process for the formation of nano-ordered hybrid materials from a hydrophobic aromatic polymer. However, little is known about these examples. It is very important to extend this scope toward thermosetting resins.

In this presentation, we report the preparation of organic/inorganic polymer hybrids from a novolacs & new class of novolac derivatives utilizing the aromatic π - π interaction. (Figure 1) The nano-ordered silica-novolac composite is expected to exhibit good thermal stability, mechanical properties, and processability of coating.

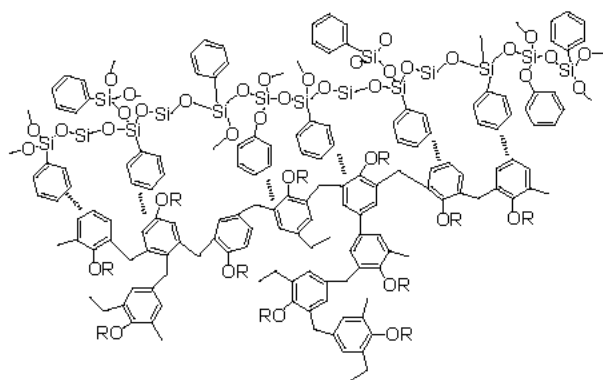
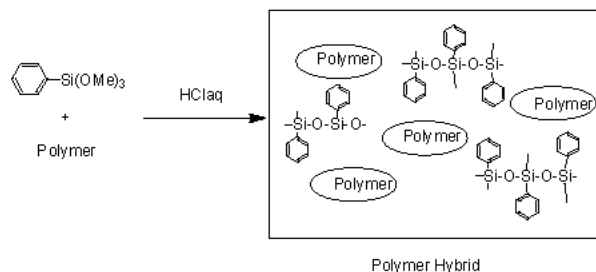


Figure 1. Preparation of Organic/Inorganic Polymer Hybrid by Means of Aromatic Interaction

Results and discussion

The organic/inorganic polymer hybrids were prepared by the hydrochloric acid (HCl)-catalyzed sol-gel reaction of phenyltrimethoxysilane (PhTMOS) in the presence of novolac derivatives. (Scheme 1) The typical procedure is as follows. PhTMOS (500 mg) and anisole novolac (500 mg) were dissolved into THF (15 mL), and aqueous HCl (1 mL, 0.1 mol/L) was added as a catalyst for the sol-gel reaction in room temperature. The mixture was stirred for 30 min in a glass bottle. The resulting mixture was then placed in a clear polypropylene (PP) container covered with a paper towel, and left in the drying apparatus. The temperature was raised to 80 °C at the rate of 10 °C/10 min (reaction rate: "Fast condition") and then kept at the same temperature for four days to give a transparent PhTMOS

film containing anisole novolac was obtained. The homogeneity of the obtained polymer hybrids was supported by SEM analysis.



Scheme 1. Preparation of Polymer Hybrid

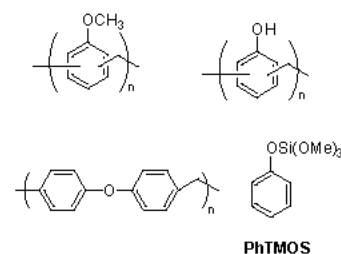


Figure 2. Precursors of Polymer Hybrids

On the other hand, for the "Slow condition" (heating rate of 10 °C/1 h), a phase separation occurred. The transparent polymer hybrid could not be obtained regardless of polymer content

Similar reactions in the presence of phenol novolac afforded the corresponding polymer hybrids. However, no polymer hybrid was obtained from the diphenyl ether novolac because of its poor solubility at a high concentration.

From these results, the most significant factor for the successful synthesis of the polymer hybrid was the "heating rate."

In conclusion, nano-ordered functional novolac-silica gel polymer hybrids were prepared by the sol-gel reaction of phenyltrimethoxysilane (PhTMOS). This polymer hybrid has a significant potential as a heat-resistant material. A new material science of a novolac film or composite will be developed by this methodology.

Acknowledgement

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References

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- (2) R. Tamaki, K. Samura, and Y. Chujo, *Chem. Commun.*, 1131 (1998).