Preparation of Phenolic Resin/Silver Nanocomposites via in-situ Reduction

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Abstract: Resol type phenolic resin/silver nanocomposite was prepared by *in-situ* reduction method, in which the curing of phenolic resin and the formation of silver nano-particles took place simultaneously. The silver ions were reduced completely to silver nanoparticles, which were dispersed homogeneously in the resin matrix with narrow size distribution.

Keywords: Nanocomposite, silver, nanoparticle, phenolic resin, *in-situ* reduction.

Polymer/metal nanocomposites have attracted considerable interest because of their potential application in catalysts, electronics, electromagnetics and nonlinear optics¹⁻³. They have been prepared by blending polymer with nanometer metal particles⁴, reducing polymer-metal ion complexes with reducing agents⁵, and reducing metal ions in the polymer matrix by high-energy radial irradiation³. In this paper, we report a new approach to phenolic resin/silver nanocomposites, which have potential applications in catalysts preparation, electromagnetic shielding, heat conduction materials *etc*.

The preparation of phenolic resin/silver nanocomposites, in which the curing of phenolic resin and the formation of silver nano-particles took place simultaneously, was carried out in the following way: silver nitrate was dissolved to make an ammonia solution according to the weight ratio of silver nitrate to ammonia (25 wt%) to ethanol 1:1:1. It was then slowly added to the resol type phenolic resin under stirring. The mixture was stirred further for 10 minutes to form a transparent solution. The volatile was removed under vacuum, and the curing was effected by heating at 180°C for 3 hours.

In the phenolic resin/silver nanocomposites thus obtained, silver nano particles of narrow size distribution are dispersed homogeneously in the resin matrix. A TEM micrograph for the phenolic resin/silver nanocomposites containing 3wt-% silver is shown in **Figure 1**.

Figure 2 displays the XRD patterns of mixtures of phenolic resin and Ag^+ ion (3 wt% Ag^+) treated at 25°C for 24 hours, 50°C, 120°C, for 2 hours and 180°C for 3 hours respectively. The peaks at 2Theta values 38.1, 44.2 and 64.3 are due to the (111), (200) and (220) diffractions of crystalline silver respectively³. It is seen that the intensity of

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diffraction increased as the temperature of the treatment enhanced.

To monitor the transfer of silver ion into metallic silver, the resin solution was diluted to 0.1 g resin/mL, and coated on glass slides for fluorescence measurements. The 320 nm fluorescence emission was caused by the free Ag^+ ions in the resin matrix⁶. It was seen that the fluorescence intensity decreased gradually as Ag^+ ions were reduced (**Figure 3**). A heat treatment at 120°C for 2 hours resulted in *ca*. 65% decrease in the fluorescence intensity. After a heat treatment at 180°C for 3 hours, the fluorescence disappeared, indicating the complete reduction of silver ions.







Figure 2 XRD patterns of mixture of phenolic resin and AgNO₃ treated at (a) 25° C 24 h, (b) 50° C 2 h, (c) 120° C 2 h and (d) 180° C 3 h.





It is well established that silver atoms are formed when silver ions obtain electrons from the donor, then the silver atoms aggregate to form clusters. In the above process for the preparation of phenolic resin/silver nanocomposites, no exotic reducing agent was

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introduced into the system purposefully. Therefore, the electron donors must be some components of the phenolic resin system. Residual formaldehyde and methylol groups may be responsible for the reduction of silver ions.

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