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PREPARATION AND ELECTROCHEMICAL PROPERTIES OF ELECTRICALLY CONDUCTIVE POLYETHERIMIDE - POLYPYRROLE COMPOSITES

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Abstract In this work, electrically conductive polyetherimide(PEI)-polypyrrole(PPy) composites have been prepared and their electrochemical properties were investigated. The composites were prepared on indium-tin-oxide(ITO) coated glass electrode. The composite films showed lower conductivity but better thermal stability than PPy homopolymer film. The properties of the composite films were strongly dependent on the preparation method of the PEI matrix.

INTRODUCTION

Composite materials based on electrically conducting polymers have attracted much scientific and technological interest in recent years[1,2]. The processing of conducting polymers has been taken through the development of composites of the conducting plastics. The added components are chosen for their mechanical and chemical properties such as cohesion, adhesion, air stability, and flexibility.[3] Interpenetrating networks of polypyrrole (PPy) filaments in swollen insulating plastic matrices have been also produced in electrochemical cells. The electrodes such as conducting glass are precoated with an insulating polymer such as poly(vinyl alohol) or poly(vinyl chloride) and exposed to a solution of pyrrole monomer. The resulting blends exhibit good mechanical properties while maintaining respectible levels of electronic conductivity[4,5].

In this work, electrically conductive polyetherimide(PEI) - PPy composites have been prepared and their electrochemical properties were investigated. PEI was chosen because of its high glass transition temperature and exellent hydrolytic stability as well as high mechancal strength.[6,7]

EXPERIMENTAL

The composites were prepared on indium-tin-oxide(ITO) coated glass electrode (size 1 x 1 cm^2). Preparation of the electrically conductive composites were prepared in two

steps; first, 1 or 2 wt.% of PEI(General Electric, Ultem) solution in N,N-dimethylacetamide(DMA) were prepared with stirring at 80-90℃ for 30 min. Then, the electrode was coated by dip-coating or spin-casting method to prepare PEI matrix for composite films. In the case of dip-coating of PEI matrix, the solution was dried vertically or horizontally. For the spin-cast matrix using a spin-coater(Headway Reserach Inc.), the speed of spinning was varied as 1,000, 1,500, 2,000, and 2,500 rpm. Then, pyrrole was electropolymerized on the PEI coated ITO glass electrode in 0.036 M sodium dodecylsufate(SDS)/H_2O electrolyte solution system containing 0.36 M pyrrole monomer. In this case, the current density was 10 mA and PPy film thickness was equivalent to 20 C/cm^2.

Thermal stabilities of the composite films were examined by Thermogravimetric Analyzer(TGA) (DuPont 2100) at a heating rate of 10 ℃/min under N_2 atmosphere. The electric resistance of the electrode side of the composite films were measured by the standard four-probe technique. The morphology of the electrode side of the composite films was observed by using Scanning Electron Microscope(SEM; JEOL JSM 5200).

RESULTS AND DISCUSSION

Table 1 and 2 show the resistance values of composite films prepared in SDS/H2O electrolyte solution system by electropolymerization method. Regardless of the preparation method(dip-coating or spin-casting) of the PEI matrix, the composite films showed higher electric resistance values(i.e. lower conductivity) than PPy homopolymer. Composite films prepared from 1 wt.% solution of PEI in DMA showed lower electric resistance than those prepared from 2 wt% solution of PEI in DMA. It means that the electric resistance value was strongly influenced by the concentration of PEI. Also, the electric resistance value was increased with dipping (i.e. coating) times. The spin-cast matrix with 2 wt% concentration of PEI exhibited lower electric resistance values as the speed of the spinning increased. However, in the case of the spin-cast matrix for lower concentration of PEI(i.e. 1 wt.%), the effect of the spinning speed was negligible.

In the case of the dip-coated matrix, the electric resistance value was also influenced by the drying method of the PEI matrix film. In the same concentration of the PEI/DMA solution, the vertically dried film showed higher electric resistance value than that of horizontally dried one, meaning that the state of PEI on the ITO coated glass electrode strongly affected the electrochemical properties of the composite films. The results of morphological investigation clearly proved the above results.

Figure 1 shows the image of PEI coated on the ITO electrode with 2 wt.%

TABLE I The electric resistance values and the thermal decomposition temperatures of dip-coated composite films with different drying method (horizontally or vertically) and PEI concentration(wt.%).

Samples	Resistance (Ω)	Thermal Decomposition Temperature(℃)
PPy	25.49	160
horizontally dried, 1 wt%	30.03	163
vertically dried, 1 wt%	34.63	169
horizontally dried, 2 wt%	114.59	173
vertically dried, 2 wt%	157.35	177
horizontally dried, dipping *two times* 2 wt%	245.78	197
vertically dried, dipping *two times* 2 wt%	285.31	200

concentration. In the case of the dip-coated PEI matrix, vertically dried film(b) showed more homogeneous phase than the horizontally dried film(a), yielding higher electric resistance values because of thicker insulating matrix character.

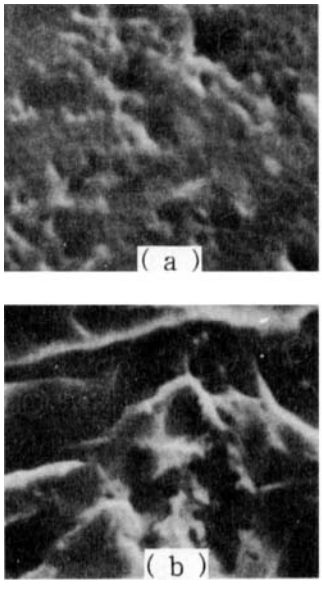
When the PEI concentration is lower(1 wt.%), not much difference was observed in morphology. Also in the case of the spin-cast PEI matrix, morphologies of samples were similar regardless of the spinning speed when the PEI concentration was 1 wt.%. However, at higher PEI concentration(2wt.%), the morphology was different depending on the spinning speed, as shown in Figure 2. The result implies that the preparation method is important to obtain composite films with homogeneous matrix for final composite materials.

In Table 1, the thermal stability increased with increasing contents of PEI. The vertically dried composite films showed higher thermal stability than the horizontally composite films. The thermal stability was, however, not strongly affected by the spinning speed for the spin-cast films except 1,000 rpm, as shown in Table 2.

It is noteworthy that all the composite films exhibited higher thermal stability when compared to that of the PPy homopolymer film.

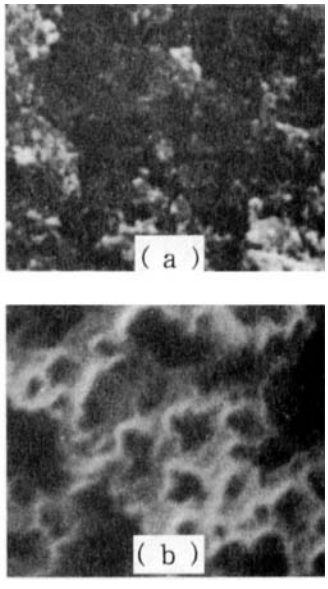
TABLE II The electric resistance values and the thermal decomposition temperatures of spin-cast composite films with different spinning speed. [PEI concentration; 2 wt.%]

Spinning speed for spin-cast films (rpm)	Resistance (Ω)	Thermal Decomposition Temperature(℃)
1,000	263.75	192
1,500	148.39	180
2,000	144.17	181
2,500	139.45	180



——— 5 μm (× 5,000)

FIGURE 1 SEM micrographs of PEI/PPy composites [dipping in 2 wt% solution] (a) horizontally dried and (b) vertically dried.



——— 5 μm (× 5,000)

FIGURE 2 SEM micrographs of PEI/PPy composites [spin-casting in 2 wt% solution] (a) 1000 rpm and (b) 2500 rpm.

CONCLUSIONS

The PEI-PPy composites films showed lower conductivity but better thermal stability than PPy homopolymer films. It was concluded that the preparation method of the PEI matrix is important to obtain composite films with homogeneous structure for final composite materials.

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