전자파 차폐용 나일론 6-폴리피롤 복합직물의 특성

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Characterization of Nylon 6-Based Polypyrrole Composite Fabrics for EMI Shielding

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1. Introduction

Among the many electrically conducting materials, polypyrrole (PPy) is one of the most promising, intrinsically conducting polymers (ICP) due to its high conductivity, oxygen resistant and good environmental stability¹. To enhance the mechanical properties, the researchers have studied the polymer-textile composites. These composites can provide the both excellent physical properties and electrical conductivity. Therefore these conducting textile composites have been attractive in the fields of static charge dissipation, EMI shielding and military camouflage materials^{2·6}.

In this research, electrically conductive polypyrrole-nylon 6 composite fabrics (PPy-N) were sequentially prepared by chemical oxidative polymerization (COP) and electrochemical polymerization (ECP) of pyrrole on the nylon 6 woven fabrics. We investigated the effects of COP condition and ECP on the properties of the resulting composites such as electrical conductivity, environmental stability and electromagnetic interference shielding effectiveness (EMI SE).

2. Experimental

2.1 Materials and reagents

Nylon 6 woven fabrics were washed with distilled water and dried prior to use.

Pyrrole was used after vacuum purification. Ferric chloride (FeCl₃), benzenesulfonic acid (BSA), anthraquinone-2-sulfonic acid, sodium salt (AQSA-Na) of the special reagent grade were used without further purification.

2.2 Preparation of polypyrrole-nylon 6 composite fabrics

Electrically conductive polypyrrole-nylon 6 composite fabrics (PPy-N) were prepared using electrochemical polymerization (ECP) as well as chemical oxidative polymerization (COP). In COP, nylon 6 fabrics were first immersed pyrrole dissolved in the aqueous BSA solution at room temperature for 2 hours. As an initiator for successive polymerization, the aqueous FeCl₃ solution containing BSA was added to the solution with the fabrics. In the mixed solution, molar ratio of pyrrole, BSA, FeCl₃ was 1: 0.33: 2.33 respectively. Polymerization was carried out at 5°C for 1 hour. ECP was carried out in an aqueous electrolyte solution by applying a constant current to the composite fabrics prepared by COP and a stainless steel plate as the working and the counter electrodes, respectively. To prepare the electrolyte solution, 1ml pyrrole was dissolved in 40ml of 0.05 M solution of AQSA-Na.

2.3 Characterization

PPy content in the composite fabrics was measured by the weighing method on the basis of the weight change of the fabrics before and after polymerization of PPy. The electrical conductivity of composites was measured using the standard four-probe method. The frequency dependence of the electromagnetic interference shielding effectiveness (EMI SE) of composites was measured with vector network analyzer (HP 8719 ES) and scattering-parameter test set from 50 MHz to 13.5 GHz.

3. Results and Discussion

The effect of liquor ratio on conductivity and PPy content in the composite fabrics prepared by COP is shown in figure 1. Both the conductivity and the PPy content of composite fabrics increased as liquor ratio decreased from 20 to 8. However lower liquor ratio resulted in lower conductivity despite of higher PPy content in the composite fabrics. The result can be explained that the conduction layer can not be formed uniformly on the matrix due to insufficient liquor ratio.

The change of conductivity with variation of pyrrole concentration in COP and after ECP is presented in figure 2. The conductivity of PPy-N prepared by COP increased with pyrrole concentration and then leveled off with a value of 0.87 S/cm at 18%. The conductivity of composites dramatically increased with ECP and

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reached a value of 9.8 S/cm. This increase was resulted from the increase of conduction layer as exhibited in figure 3. However the conductivity of the resulting composite fabrics did not showed significant difference although the composites before ECP showed different conductivity. From the result, it can be explained that ECP played dominant role to form the conduction layer comparing with COP.

The conductivity stability of composite fabrics at elevated temperature in air is shown in figure 4. Both in PPy-N prepared by COP and those prepared by COP and ECP, the conductivity was retained almost constant at 120°C for 600 min. However the conductivity degradation at higher temperature was revealed the different tendency. When the experiment temperature was 150°C and 180°C, the conductivity of PPy-N prepared by COP was abruptly decreased because of dissociation of dopant from the PPy chain. But in case of PPy-N prepared by COP and ECP, the total loss of conductivity was less than 1 order of magnitude.

4. Conclusions

Polypyrrole was polymerized chemically and electrochemically in sequence on a nylon 6 woven fabrics, giving rise to polypyrrole-nylon 6 composite fabrics (PPy-N) with highly electrical conductivity. The electrical conductivity and EMI SE of the PPy-N are approximately 10 S/cm and 30 dB over a wide frequency range up to 13.5 GHz respectively. The conductivity stability of PPy-N prepared by COP and ECP is better than for PPy-N prepared by only COP.

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Acknowledgement

This study was supported by Dual Use Technology Program of the Ministry of Commerce, Industry and Energy, Korea (2000)

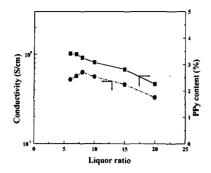


Figure 1. The effect of liquor ratio on conductivity and PPy content in the composite fabrics prepared by COP.

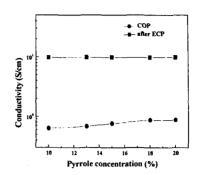
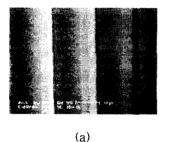
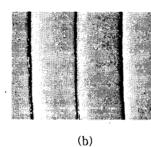


Figure 2. The change of conductivity with variation of pyrrole concentration in COP and after ECP.





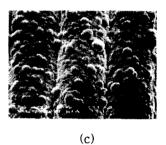
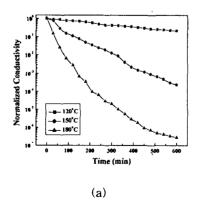


Figure 3. Planar view of SEM micrographs ($\times 2,000$) (a) pristine nylon 6 (b) PPy-N prepared by COP

(c) PPy-N prepared by COP and ECP.



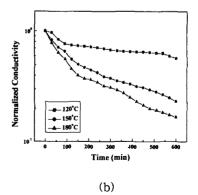


Figure 4. The conductivity stability of composite fabrics at elevated temperature in air.

(a) PPy-N prepared by COP (b) PPy-N prepared by COP and ECP.