New Method for the Preparation of Thick Conducting Polymer Composites

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SYNOPSIS

A highly porous and absorbable crosslinked polystyrene, prepared by the concentrated emulsion polymerization method, was used as host polymer for the preparation of conducting, large objects, polymer composites. The composites, whose conductivity can be as high as 0.80 S/cm, were prepared by (i) imbibing the host polymer with a pyrrole (or oxidant) solution, (ii) partially drying the imbibed host polymer, and (iii) imbibing again with an oxidant (or pyrrole) solution for polymerization to take place. The electrical conductivity of the composite and the penetration of polypyrrole in the host polymer are influenced by the polymerization conditions (i.e., the concentrations of oxidant and pyrrole and the nature of the solvents used for the oxidant and pyrrole), the order in which the two imbibing solutions are introduced, and the drying time used after the first imbibation. The mechanical properties of the host polymer are improved with the incorporation of polypyrrole. Scanning electron micrographs of the composites indicate that the polypyrrole coats uniformly as a film the inside of the porous host polymer.

INTRODUCTION

Since the discovery of electrical conducting polymers,[1,2] they have drawn considerable attention as possible substitutes for metallic conductors or semiconductors in a wide variety of electrical and electronic devices.[3-5] Potential advantages of conductive or semiconductive polymers lie in their light weight and in the versatility with which their synthesis and fabrication can be accomplished. However, most conducting polymers have at least one of the following undesirable characteristics: (1) environmental instability, (2) poor processability, and (3) poor physical properties.[5] In the last few years, a considerable amount of investigation on conducting polyheterocyclic polymers has been carried out because of their good environmental stability. Among these polyheterocyclic polymers, polypyrrole (PP) has been extensively employed.[6,7] PP was prepared either by the chemical oxidative polymerization method[6] or by the electrochemical oxidative poly-

merization method.[7] The electrochemical polymerization of pyrrole produces free-standing conducting films, whose conductivity at room temperature is as high as 10^2 S/cm.[8] On the other hand, the chemical polymerization method produces a finely divided, insoluble black powder, whose conductivity ranges from 10^{-15} S/cm to 10^1 S/cm, depending on the specific preparative conditions.[9] However, both the electrochemically prepared PP films and the chemically prepared PP powders are difficult to handle, and this restricts their potential for applications. A useful approach for the improvement of the mechanical properties of polymers has been the synthesis of hybrids, i.e., copolymers and polymer blends. A slight improvement of the mechanical properties has been, indeed, achieved by the copolymerization of pyrrole and styrene.[10] A better improvement of the mechanical properties can be obtained by formulating a conducting polymer blend in which the conducting polymer is well mixed with one or more conventional polymers. This polymer blending method involves the inclusion of electrochemically or chemically polymerized pyrrole in the matrix of a host insulating polymer. PP can be impregnated electrochemically into a host polymer that coats the electrode. Conducting polymer blends have

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been obtained by this electrochemical polymerization of pyrrole in a poly(vinyl chloride) (PVC),[11] poly(vinyl alcohol) (PVA),[12] or polyurethane (PU) matrix.[13] There are, however, limitations regarding the practical applications of the polymer blends prepared by the electrochemical method. Indeed, uniform thin films of the host polymers are necessary for this process and it is difficult to prepare them at a large scale. Although the chemical method usually produces a less conductive PP compared with the electrochemical method, it has the advantages of easier mass production and shorter reaction time. To improve the mechanical properties of PP obtained by the chemical method, conducting polymer blends were prepared by exposing an oxidant containing nonporous host polymer to pyrrole vapor.[14] Poly(methyl methacrylate) (PMMA), PVC, or PVA were employed as host polymers. Another type of conducting polymer composite was prepared by incorporating pyrrole into a nonporous polymeric matrix, followed by the exposure of the impregnated matrix to an oxidant.[15] However, the low penetration of PP into the matrix limits the utility of these procedures. The penetration of the PP in the host polymer matrix can be increased by employing a porous host polymer instead of a nonporous host polymer. A conducting polymer composite of PP–cellulose was prepared by impregnating thin porous filter papers with an aqueous oxidant solution and then contacting them with pyrrole as liquid or vapor.[16] However, in this case, the host polymer is thin and has poor mechanical properties. For the preparation of thick and large objects, the host polymer must be able to be appreciably imbibed by both the monomer and oxidant solutions, thus permitting PP to grow uniformly inside the matrix.

In this work we report a new approach to preparing thick conducting polymer composites with good mechanical properties. The concentrated emulsion polymerization method[17,18] is employed for the preparation of the host polymer. The host polymer is imbibed either with the monomer and subsequently with the oxidant or vice versa. The porous host polymer was synthesized starting from a concentrated emulsion of water dispersed in a hydrophobic continuous phase of a mixture of styrene and divinyl benzene. A concentrated emulsion has a large volume fraction of dispersed phase (about 0.8 in the present study) and the appearance of a gel. For sufficiently large volume fractions, it consists of polyhedral droplets separated by thin layers of the continuous phase.[18] This concentrated emulsion is stabilized by dissolving suitable surfactants in the continuous phase. The incorporation of PP into the porous host polymer has been achieved by the chemical oxidative polymerization method. After the well-dried porous host polymer was imbibed with a solution of pyrrole (or oxidant) and then partially dried, it was contacted with a solution of oxidant (or pyrrole). As a result, pyrrole polymerizes inside the host polymer. Both aqueous and nonaqueous solvents were employed for the oxidant, and nonaqueous solvents for pyrrole. $FeCl_3$ was used both as polymerization initiator and dopant. The effect of the order in which the host polymer was imbibed with the solutions of monomer and oxidant, as well as the effect of the concentrations of oxidant and pyrrole on the conductivity of composite was investigated. The relationship between the depth of penetration of the PP in the host matrix and the drying time after the first imbibation was also examined.

EXPERIMENTAL

Materials

Styrene (Aldrich), divinyl benzene (Polysciences), and pyrrole (Aldrich) were distilled and stored in a refrigerator. Azobisisobutyronitrile (AIBN, Alfa)

Table I   Representative Compositions in the Preparation of Porous Host Polymers

	Polymer 1	Polymer 2	Polymer 3
Continuous phase			
Styrene	5 g	5 g	5 g
Divinyl benzene	1 g	1 g	1 g
Initiator (AIBN)	0.05 g	0.05 g	0.05 g
Surfactant (sorbitane monooleate)	1 mL	1 mL	1 mL
Dispersed phase			
Water	20 mL	25 mL	30 mL

was purified by recrystallization in methanol. Sorbitane monooleate (Span80, Fluka), iodine (Baker Chem.), ferric chloride (Aldrich), and ferric chloride hexahydrate (Aldrich) were used as received. All solvents were of reagent grade and were used as received. Water was deionized and distilled.

Preparation of the Host Polymer (Porous Crosslinked Polystyrene)

A small amount of a mixture of styrene and divinyl benzene containing AIBN and sorbitane monooleate was placed in a flask (250-mL capacity) equipped with a mechanical stirrer and an addition funnel. A set of amounts involved is listed in Table I. Water was placed in the addition funnel. The concentrated emulsion was prepared at ambient temperature by dropwise addition of water to the stirred mixture of styrene and divinyl benzene containing AIBN and sorbitane monooleate.[17] The polymerization was carried out in a temperature-controlled oven at 50°C for 24 h. Subsequently, the water of the dispersed phase was eliminated by keeping the polymer in the oven at 100°C for 3 days.

Preparation of the Conducting Polymer Composite

Two different procedures were used to incorporate PP in the porous host polymer. The experiments were conducted at ambient temperature.

The First Procedure

A well-dried porous host polymer was dipped in the oxidant solution until it saturated (about 15 min). Subsequently, the solution imbibed host polymer was partially dried by exposing it to air at room temperature. The partially dried host polymer was contacted with a large amount of pyrrole–organic solvent solution for 2 h. The latter solution penetrates in the pores of the host polymer and there polymerization takes place. Because of some diffusion of the oxidant outside the host polymer, some polymerization occurs also outside. Before measuring the conductivity of the composite, it was dried in air for 1 day.

The Second Procedure

A well-dried host polymer was saturated with a pyrrole–organic solvent solution (about 15 min). The saturated host polymer was partially dried in air. The partially dried host polymer was contacted with a large amount of the oxidant solution for 2 h for

polymerization to take place. The conductivity of the composite was measured after drying in air for 1 day.

The Absorption Test

To determine the maximum absorption capacity of the porous host polymer ($0.3 \times 2 \times 3$ cm), the host polymer was immersed in various solvents at room temperature for various time intervals. The increase in weight due to absorption was determined with a Mettler balance. The rate of desorption of the solvent from the host polymer in air with time after the first imbibation was also measured by using a Mettler balance.

Conductivity Measurements

Measurements were carried out by using the standard four-point probe method on thin sheets of composites ($0.3 \times 0.75 \times 2.5$ cm) at room temperature.

Electron Microscopy

The morphologies of the porous host polymer and conducting composite were examined by scanning electron microscopy (SEM, Amray 100A). A thin layer of gold was deposited on the cross section of the sample prior to observation.

Penetration Observation

The penetration of PP inside the composite was examined by employing a well-dried pellet of porous host polymer (diameter 1.6 cm, length 4 cm). After the formation of the composite, its cross section, obtained by cutting the sample, was subjected to observation.

RESULTS AND DISCUSSIONS

Three porous host polymers, whose compositions are listed in Table I, were prepared by the concentrated emulsion polymerization method. The maximum amounts of various liquids absorbed by the porous polymer 2 are listed in Table II. These data indicate that the host polymer is highly porous and can absorb amounts of liquid greater than three times its own weight. The highest absorption is for chloroform, namely 9.5 g chloroform per gram host polymer. The desorption in air at room temperature of the absorbed liquid from its host polymer is plot-

Table II   Absorption of Various Liquids in the Host Polymer[a]

	Water	Pyrrole	Acetonitrile	Methanol	Ether	Chloroform
Absorption (grams liquid/ grams host polymer)	4.4	4.6	4.1	3.8	3.5	9.5

[a] Polymer 2 was used in these experiments.

ted in Figure 1. As expected, the desorption rate of methanol is faster than those of water and pyrrole; most of the methanol was evaporated from the host polymer in 40 min. This figure also shows that absorption increases with increasing porosity of the polymer. The mechanical strength of the host polymer decreases, however, with increasing porosity.

The chemical oxidative polymerization method was employed to incorporate PP in the host polymer. SEM studies were carried out to investigate the morphologies of the host polymer and of the conducting composite. The host polymer, shown in Figure 2(a), contains pores larger and smaller than 10 μm. According to previous studies,[17,18] the size of the dispersed phase is in the micron or submicron range. The larger pores observed here might form during drying at 100°C. These holes might constitute the tunnels through which the solutions of pyrrole and oxidant penetrate in the host polymer. The PP forms in the host polymer, producing a conducting composite, which becomes stronger and harder than

Figure 1 The amount of liquid against time during drying in air at ambient temperature.

the host polymer. In the composite, shown in Figures 2(b) and 2(c), the PP coats the inside surface area of the host polymer; in addition, some particles of PP are attached to the surface of the host polymer.

Table III lists the conductivity of the polymer composites, which were prepared by changing the solvent in the pyrrole solution. The value of the conductivity is not affected in a major way by the nature of the solvent and is of the order of 10^{-1} S/ cm. The effect of the oxidant and its solvent on the conductivity of the composites is shown in Table IV. The ferric chloride, which is known to be a very effective oxidant,[9] generates a better conducting composite than iodine. The conductivities of the polymer composites prepared by using a ferric chloride hexahyrate–acetone solution and a ferric chloride–acetonitrile solution were 3.9 × 10^{-3} and 2.7 × 10^{-1} S/cm, respectively. These experimental results indicate that the nature of the solvent employed for the oxidant affects the conductivity of the composite. In the literature,[9,19,20] it was already noted that the reaction medium plays an important role. Many solvent-related factors, such as the solubility of $FeCl_3$, the solvent basicity, the dielectric constant of the solvent, and the Fe^{3+}–Fe^{2+} redox couple, are expected to affect the reaction of ferric chloride with pyrrole. The texture of the PP is, therefore, expected to depend on the reaction medium, which thus affects the conductivity of the composite. The porosity of the host polymer also plays a part, since it can determine whether the growth of the PP occurs such as to generate a conducting pathway.

As the chemical polymerization of the pyrrole proceeds, the host polymer darkens. When pyrrole (or oxidant) solution containing host polymer is imbibed with the oxidant (or pyrrole) solution, PP films grow on the outside surface, generating a layer that retards the diffusion of pyrrole (or oxidant) in the host polymer. For this reason the host polymer imbibed with the first solution is dried before being imbibed with the second solution. By controlling the drying ti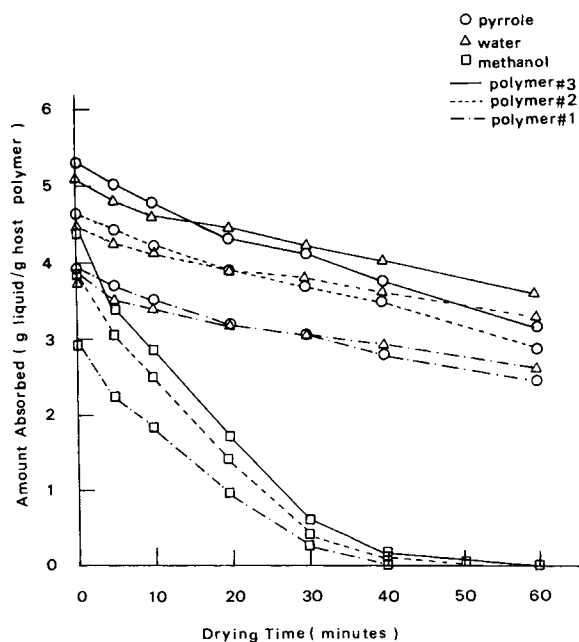me, one can influence the absorption of the second solution. Figure 3 shows a poor penetration of the PP in the composite for short drying times.
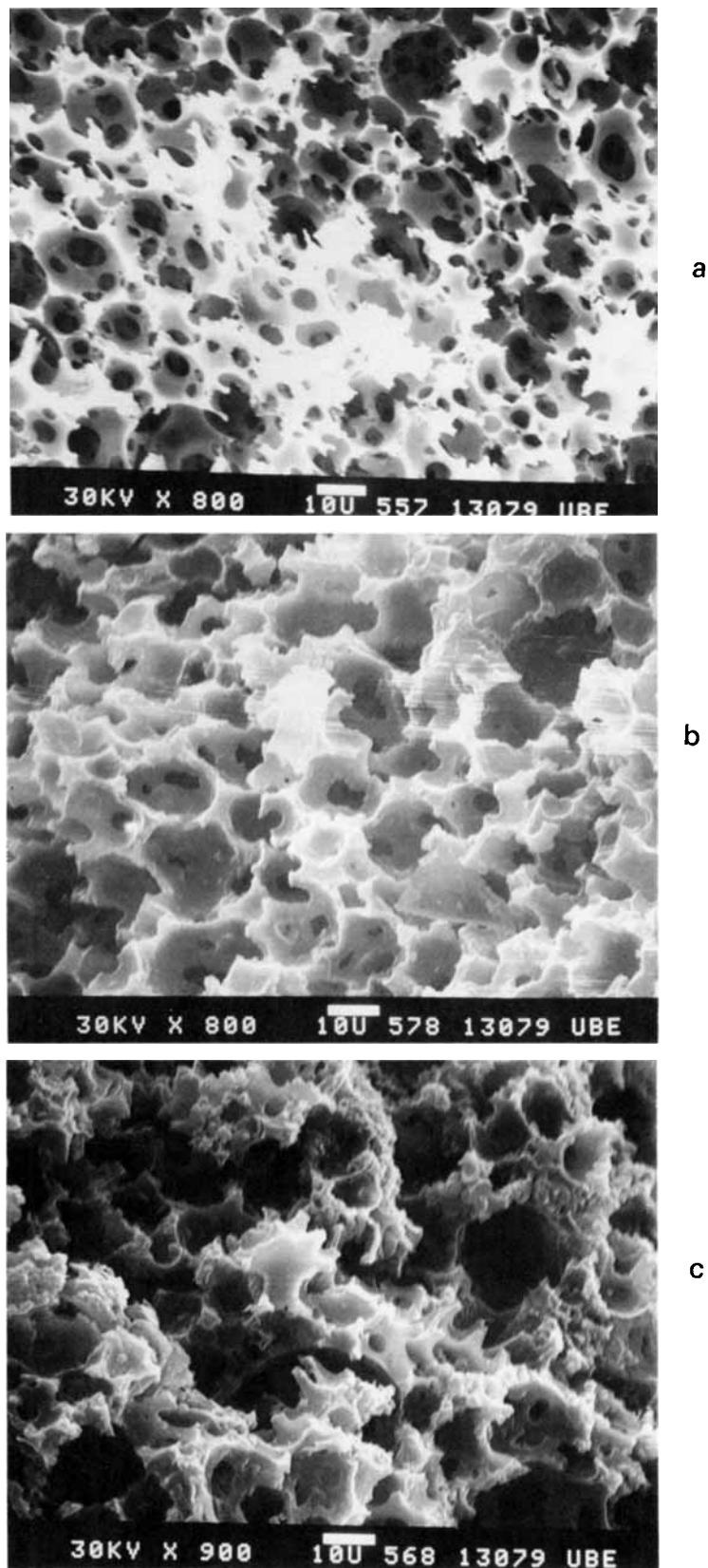
Figure 2 Scanning electron micrographs of the host polymer (a) and composites (b, c). The composites were prepared by the second procedure. The oxidant solution for composite (b) was $FeCl_3 6H_2O$ (4 g) in water (10 mL) and for composite (c) $FeCl_3$ (2 g) in methanol (10 mL). The drying time of the host polymer imbibed with pyrrole–ether solution (4 g pyrrole in 10 mL ether) was 20 min.

**Table III   Effect of the Solvent Employed
for Pyrrole on the Conductivity
of Polymer Composite[a]**

Solvent	Conductivity (S/cm)
Cyclohexane	2.1×10^{-1}
Chloroform	3.0×10^{-1}
Methanol	1.4×10^{-1}
Ether	3.1×10^{-1}

[a] The polymer composites were prepared by the first procedure. The drying time after imbibing with the oxidant solution (2 g $FeCl_3$/10 mL methanol) was 20 min. The concentration of pyrrole was 2 g/10 mL solvent.

Figure 3   Penetration of PP in the polymer composites. Polymer composites (a) were prepared by the first procedure by changing the drying time of the host polymer imbibed with the oxidant solution. The concentrations of pyrrole and oxidant were 4 g pyrrole/10 mL ether and 4 g $FeCl_36H_2O$/10 mL H_2O, respectively. The drying time was (a) 0 min, (b) 20 min, (c) 40 min, (d) 50 min, (e) 90 min, and (f) 150 min, respectively. Polymer composites (b) were prepared by the second procedure by changing the drying time of the host polymer imbibed with the pyrrole solution. The drying time was (a) 0 min, (b) 20 min, (c) 30 min, (d) 40 min, and (e) 60 min, respectively.

The poor penetration is partly due to the poor miscibility between the pyrrole and oxidant solvents employed. Figures 4 and 5 show the effect of the penetration of PP on the conductivity of the composites, prepared as in Figures 3(a) and 3(b), respectively. In both cases the conductivity is low for short drying times, due to the low penetration and nonuniform distribution of the PP in the composites. Figures 4 and 5 indicate that the amount of PP included in the composite is 1–2 g PP/gram host polymer for the first procedure and 3–5 g PP/gram host polymer for the second procedure.

Figure 6, in which instead of an aqueous oxidant solution a methanol solution is employed, shows a better penetration of PP in the composite, compared with that of Figure 3. This is due to the better miscibility of the oxidant solvent and pyrrole solution. Comparing Figures 6(a) and 6(b), one can conclude that the composite prepared by the first procedure is better penetrated by PP for short drying times than the composite prepared by the second procedure. The conductivities of the composites, prepared
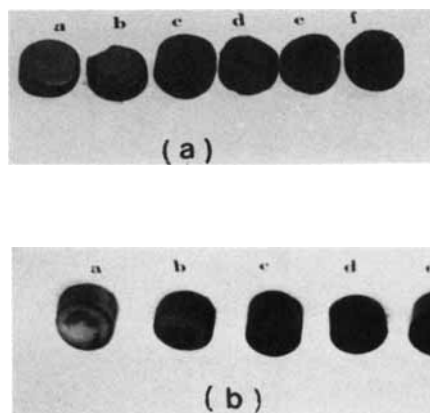
as in Figures 6(a) and 6(b), are plotted against drying time in Figures 7 and 8. The conductivity of the composites in Figure 7 is by an order of magnitude higher than that of the composites in Figure 8. Both kinds of composites incorporate, however, comparable amounts of PP in the host polymer. Consequently, the solvent employed and the order in which the two solutions imbibe the host polymer affect both the penetration of PP in the composites and the conductivity of the composite. The order of

**Table IV   Effect of the Oxidant and the Solvent
Employed for the Oxidant on the Conductivity of the
Polymer Composite[a]**

Oxidant Solution	Conductivity (S/cm)
$FeCl_36H_2O$ in acetone (4 g/10 mL)	3.9×10^{-3}
$FeCl_36H_2O$ in water (4 g/10 mL)	4.1×10^{-2}
$FeCl_3$ in acetonitrile (2 g/10 mL)	2.7×10^{-1}
$FeCl_3$ in methanol (2 g/10 mL)	6.5×10^{-1}
I_2 in acetonitrile (0.2 g/10 mL)	5.3×10^{-4}

[a] The polymer composites were prepared by the first procedure. The drying times for the nonaqueous oxidant solution (2 g $FeCl_3$/10 mL solvent) and aqueous oxidant solution (4 g $FeCl_36H_2O$/10 mL H_2O) were 20 and 60 min, respectively. The concentration of pyrrole was 2 g/10 mL ether.
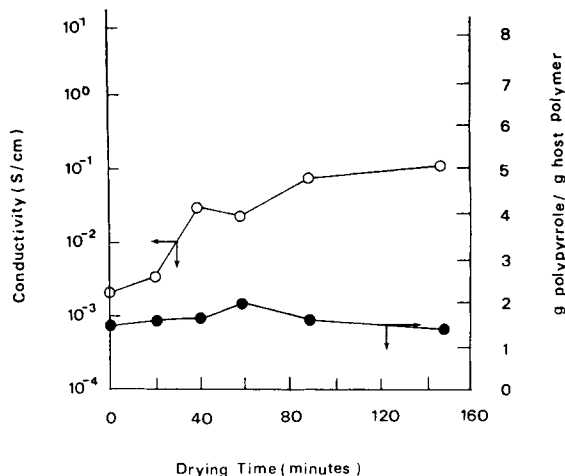
Figure 4 Conductivity of composite and weight ratio of PP to host polymer against drying time of the host polymer imbibed with the oxidant solution in air. The polymer composites were prepared by the first procedure with the compositions of Figure 3(a).
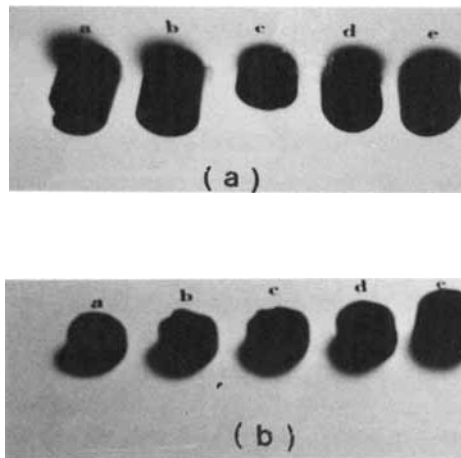

Figure 6 Penetration of PP in the polymer composites. Polymer composites (a) were prepared by the first procedure by changing the drying time in air of oxidant solution imbibed host polymer. The concentrations of oxidant and pyrrole were 2 g $FeCl_3$/10 mL methanol and 4 g pyrrole/10 mL ether, respectively. The drying time of the oxidant solution imbibed host polymer was (a) 0 min, (b) 5 min, (c) 10 min, (d) 20 min, and (e) 40 min, respectively. Polymer composites (b) were prepared by the second procedure by changing the drying time of pyrrole solution imbibed host polymers. The drying time was (a) 0 min, (b) 5 min, (c) 10 min, (d) 20 min, and (e) 30 min, respectively.

contacting also affects, in some cases, the amount of PP incorporated in the composite. However, the amounts of PP incorporated in the composites do not affect their conductivity.

The effect of the concentrations of oxidant and pyrrole on conductivity for both the first and second procedure and nonaqueous solvents is worth investigating. The results for the first procedure are presented in Figures 9 and 10. In Figure 9, the conductivity is about 10^{-1} S/cm and is unaffected by the

concentration of the oxidant. The oxidant concentration does not affect the amount of PP formed in the composite either. Figure 10 shows that the
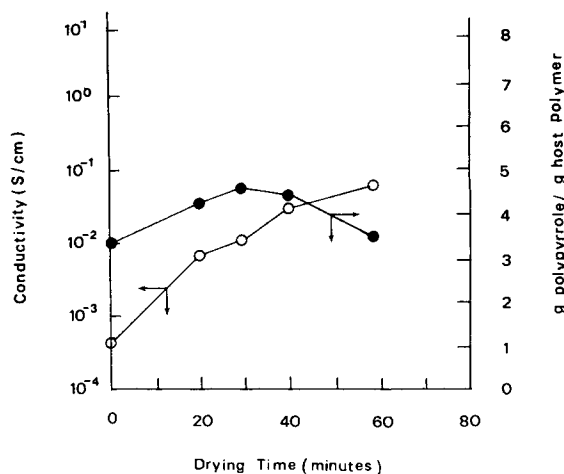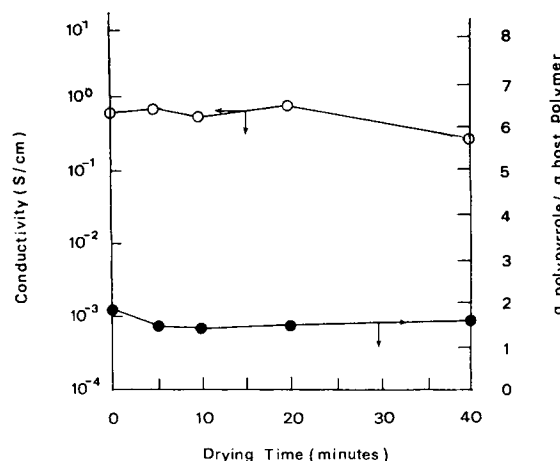

Figure 5 Conductivity of composites and weight ratio of PP to host polymer against drying time in air of pyrrole solution imbibed host polymer. The polymer composites were prepared by the second procedure with the compositions of Figure 3(b).

Figure 7 Conductivity of composites and weight ratio of PP to host polymer against drying time in air of oxidant solution imbibed host polymer. The polymer composites were prepared by the first procedure with the composition of Figure 6(a).
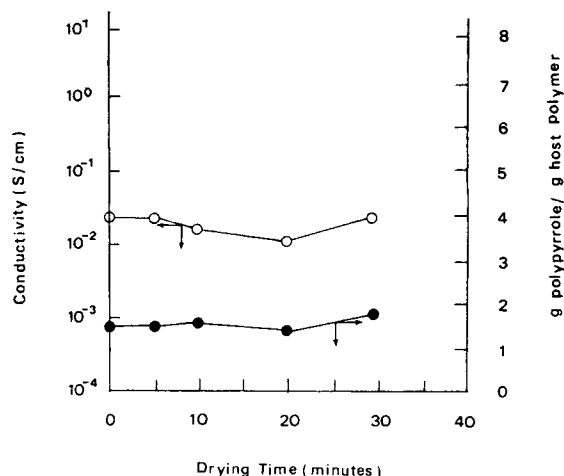
Figure 8 Conductivity of composites and weight ratio of PP to host polymer against drying time in air of pyrrole solution imbibed host polymer. The polymer composites were prepared by the second procedure with the composition of Figure 6(b).
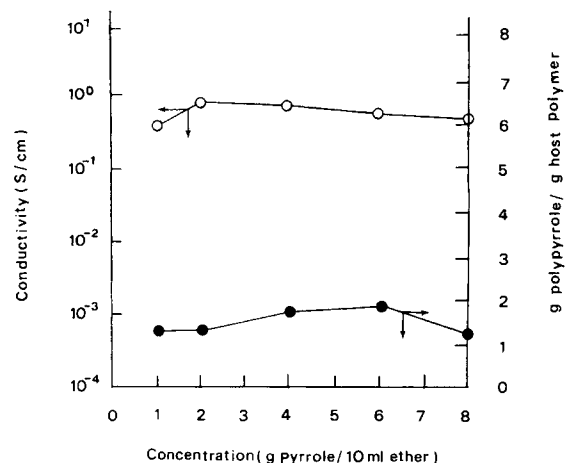

Figure 10 Conductivity of composites and weight ratio of PP to host polymer against pyrrole concentration. The composites were prepared by the first procedure. The saturated host polymer with oxidant solution (2 g $FeCl_3$/10 mL methanol) was dried for 20 min in air, followed by imbibation in the pyrrole solution (pyrrole in 10 mL ether).

monomer concentration does not affect the conductivity and the amount of PP included in the composite. Figures 11 and 12 present the conductivities of composites prepared by the second procedure. They are by an order of magnitude smaller than those obtained by the first procedure and are low at low concentrations of oxidant and pyrrole. The amount of PP included in the composite increases with the concentration of oxidant (Fig. 11). No re-

lationship between the amount of PP in the composite and conductivity was, however, found. The lower conductivities of the composites prepared by the second procedure might be due to the lower penetration and nonuniform distribution of PP in the composite, detected in the penetration experiments (Fig. 6).
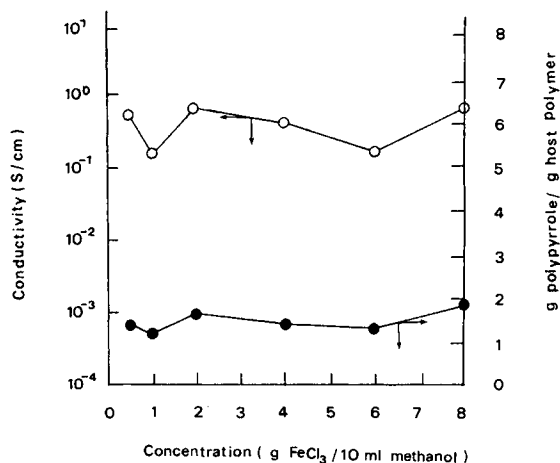

Figure 9 Conductivity of composites and weight ratio of PP to host polymer against oxidant concentration. The composites were prepared by the first procedure. The host polymer imbibed with oxidant solution ($FeCl_3$ in methanol) was dried for 20 min in air, followed by imbibation with pyrrole solution (4 g pyrrole/10 mL ether).
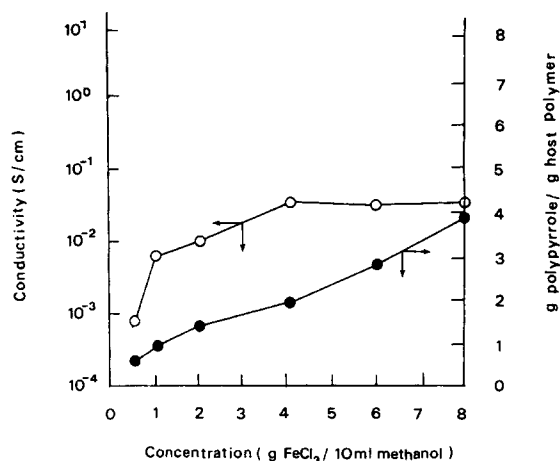

Figure 11 Conductivities of composites and weight ratio of PP to host polymer against concentration. The composites were prepared by the second procedure. The host polymer imbibed with pyrrole solution (2 g pyrrole/10 mL ether) was dried for 20 min in air and then imbibed with oxidant solution (2 g $FeCl_3$/10 mL methanol).
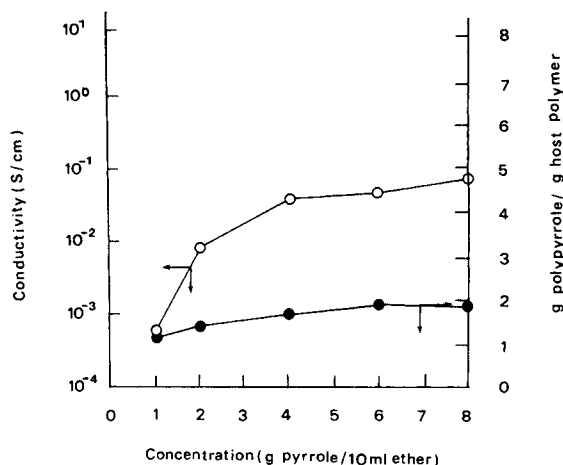
Figure 12 Conductivity of composites and weight ratio of PP to host polymer against pyrrole concentration. The composites were prepared by the second procedure. The host polymer imbibed with pyrrole solution (pyrrole in ether) was dried for 20 min in air and then imbibed in oxidant solution (2 g $FeCl_3$/10 mL methanol).

Figure 14 Conductivity of composites and weight ratio of PP to host polymer against pyrrole concentration. The composites were prepared by the first procedure. The host polymer imbibed with oxidant solution (2 g $FeCl_36H_2O$/10 mL H_2O) was dried for 50 min in air and then imbibed with pyrrole solution (pyrrole in 10 mL ether).

Figures 13 and 14 present the effect of the concentrations of oxidant and pyrrole on the conductivities of composite when an aqueous medium is employed for the oxidant. At low concentrations of oxidant and pyrrole, the conductivity has low values. In Figure 13 the conductivity increases with increasing oxidant concentration, without appreciable changes in the amount of PP. In Figure 14 the con-
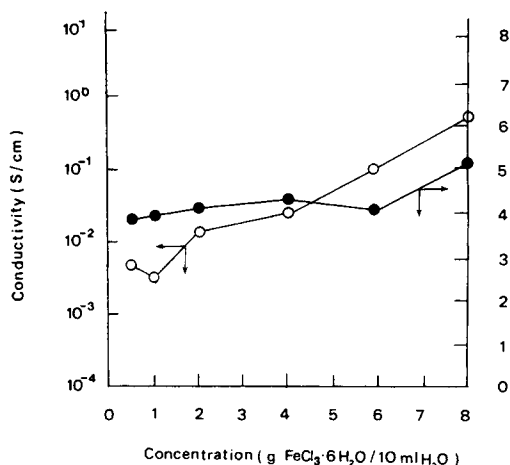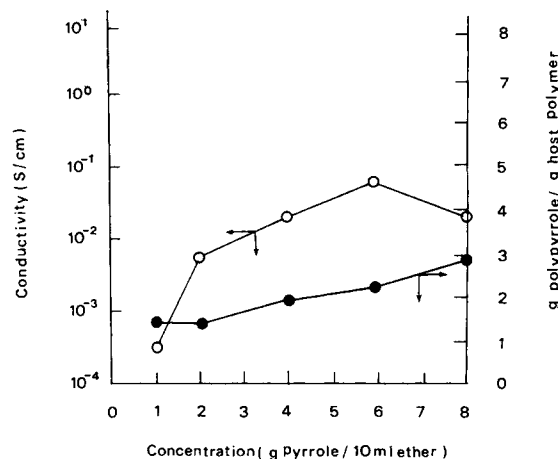
ductivity does not vary with the increase in the concentration of pyrrole.

As shown in Table V, the amount of oxidant in the host polymer increases with increasing porosity of the host polymer. For the range of porosities investigated, there is, however, no major change of the conductivity with porosity.

CONCLUSION

A new method for preparing thick, conducting polymer composites is suggested. In the first step a highly porous host polymer is prepared. The concentrated emulsion method is employed to prepare a porous crosslinked polystyrene host polymer. In the second step the host polymer is imbibed with a solution of pyrrole or a solution of oxidant ($FeCl_3$). In order to facilitate the subsequent uniform distribution of a second solution, the host polymer thus imbibed is subjected to drying. After drying, the second solution (solution of oxidant or pyrrole) is allowed to imbibe the host polymer. As a result, pyrrole polymerizes to polypyrrole, with the latter uniformly distributed inside the host polymer. Morphology studies with SEM indicate that the polypyrrole grows on the inner surface of the host polymer in the form of polypyrrole films. Some polypyrrole particles are also attached to the surface of the host polymer. A higher penetration and more uniform distribution of polypyrrole in the composite is obtained by employing

Figure 13 Conductivity of composites and weight ratio of PP to host polymer against oxidant concentration. The composites were prepared by the second procedure. The host polymer imbibed with pyrrole solution (4 g pyrrole/10 mL ether) was dried for 40 min in air and then imbibed with oxidant solution ($FeCl_36H_2O$ in 10 mL H_2O).

Table V   Effect of the Porosity of the Host Polymer on the Conductivity of Polymer Composites[a]

Host Polymer	Cond. (S/cm)	g Oxidant solution/g Polymer	g PP/g Polymer
1	1.3×10^{-1}	0.94	1.61
2	6.5×10^{-1}	1.22	1.70
3	5.5×10^{-1}	1.42	1.77

[a] The polymer composites were prepared by the first procedure. The concentrations of oxidant and pyrrole were 2 g $FeCl_3$/10 mL methanol and 4 g pyrrole/10 mL ether, respectively. The drying time of the host polymer imbibed with the oxidant solution was 20 min.

nonaqueous solvents (instead of an aqueous solvent) for the oxidant. Imbibing first with the oxidant solution and later with the pyrrole solution, higher conductivities have been obtained.

We believe that the present method can be employed to fabricate large and light-weight conducting material, which can be used for electromagnetic shielding and electrostatic charge protection.

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