

Conducting Poly(styrene-co-divinylbenzene)/Polypyrrole PolyHIPE Composite Foam Prepared by Chemical Oxidative Polymerization

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ABSTRACT: Conducting poly(styrene-co-divinylbenzene)/polypyrrole (PPy) polyHIPE (polymerized high internal phase emulsion) composite foams were synthesized via chemical oxidative polymerization method. The effect of solvent and dopant type on the surface morphology and electrical conductivity of composite foams has been investigated. SEM micrographs showed that the morphology of PPy thin film on the internal surface of poly(styrene/divinylbenzene) (poly(St-co-DVB) polyHIPE support foam strongly depends on the solvent and dopant type used. Incorporation of dodecylbenzene sulfonic acid-sodium salt (DBSNa) as a dopant in chloroform solvent resulted in formation of a PPy thin film with higher molecular compact structure and electrical conductivity on the support foam as compared to other solvents and another dopant used. Fourier-transform infrared spectroscopy was used to correlate the electrical conductivity of composite foams to their PPy structural parameters. As expected, the extended conjugation length of PPy in the presence of DBSNa dopant is the main reason for higher electrical conductivity of resultant composite foam. Electrical conductivity measurements revealed that the chemical aging of various conducting foams follows the first-order kinetic model, which is a representative of a reaction-controlled aging mechanism. © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 000: 000–000, 2012

KEYWORDS: chemical oxidative polymerization; polypyrrole; dopant; electrical conductivity; polyHIPE foam

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INTRODUCTION

Recently, electrically conducting polymers have attracted scientists and industry interests because of their potential applications in various electronic devices, such as EMI shielding, rechargeable batteries, corrosion protective coatings, and electrochemical sensors.^{1–6} Among conducting polymers, polypyrrole (PPy) has been studied extensively due to its reasonably high conductivity, facile synthesis, and relative good environmental stability.^{7–11} However, poor mechanical properties and low processability of conducting PPy restrict its widespread technological applications. Several methods have been developed to overcome the aforementioned drawbacks. Most research works have improved the physical properties of PPy based on its copolymerization with insulating polymers.^{12–14} The preparation of conducting composites via *in situ* polymerization of conducting polymer within a host insulating polymer is another approach to improve PPy properties.^{15–21} Recently, *in situ* polymerization of pyrrole within a porous polymeric host has produced conducting composite materials with proper mechanical/electrical properties. *In situ* polymerization of pyrrole may be accomplished by diffusing pyrrole solution into polymeric foam

containing a suitable oxidant. This approach was used to prepare conducting polyurethane/PPy^{15–18} and poly(styrene-co-divinylbenzene)/PPy composite foams.^{19–21}

Polymerized high internal phase emulsion (polyHIPE) foams are prepared by polymerizing the continuous organic phase of high internal phase emulsions (HIPEs). The resulting foam consists of a highly open-cell and interconnected structure provides a suitable substrate for *in situ* polymerization of the pyrrole within. This structure of polymeric substrate facilitates the penetration and uniform formation of conducting polymer inside polyHIPE foam.^{19–21} Recently, a few research works have been carried out on the preparation and characterization of conducting composite foams via *in situ* polymerization of monomers within polyHIPE foams. Ruckenstein et al.^{19–20} synthesized polyHIPE support foams with a maximum nominal porosity of 84% in the absence of porogen solvent. They coated the prepared foams by a thin film of PPy without using any sulfonic derivatives as a dopant. In this case, the composite foams were prepared based on support foams with low specific surface area. However, in some applications such as gas electrochemical sensors the performance of conducting foam is directly related its

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specific surface area to provide a higher surface for electrochemical reaction with adsorbed gas. Incorporation of a suitable porogenic solvent in the organic phase of HIPE increases the specific surface area and presumably improves the electrical conductivity of composite foam.^{22–23}

In this work, conducting poly(St-co-DVB)/PPy polyHIPE composite foams were synthesized by *in situ* chemical oxidative polymerization. For this purpose, highly porous polyHIPE support foam with nominal porosity of 95% was first synthesized using a porogenic solvent and then its internal surface coated by a thin layer of conducting PPy film. The effects of different dopants and monomer solvents have been investigated based on surface morphology and electrical conductivity of resultant composite polyHIPE foams. The effect of chemical aging on the foam electrical conductivity has also been investigated.

EXPERIMENTAL

Materials

All chemical reagents were purchased from Merck Co. (Darmstadt, Germany), unless otherwise stated. Styrene, divinylbenzene, 1, 2-dichlorobenzene, and pyrrole were distilled under vacuum to remove the inhibitor and stored at 5°C before use. Sorbitan monooleate (Span80), potassium persulfate (K₂S₂O₈), iron (III) chloride as oxidant, and calcium chloride dihydrate were used without any purification. Chloroform, acetonitrile, and cyclohexane, all as monomer solvents were used for pyrrole dissolution. 1,5-Naphthalenedisulfonic acid (NDSA) and dodecylbenzene sulfonic acid-sodium salt (DBSNa) as sulfonate derivatives or dopants were used without any further purification. Methanol was used as the oxidant solvent. Deionized distilled water (DDI) was prepared in the author's laboratory.

Synthesis of PolyHIPE Support Foam

The aqueous internal phase of HIPE (95 vol %) containing 1 wt % CaCl₂·2H₂O as electrolyte and 5 wt % potassium persulfate as initiator was added dropwise into the organic continuous phase. The organic phase consisted of styrene and 1,2-dichlorobenzene as porogenic solvent (1 : 1 by volume ratio), divinylbenzene as crosslinker (50 vol % based on styrene), and Span80 as surfactant (20 vol % based on total monomer/porogen). The emulsification process was carried out at 400 rpm and room temperature. The prepared concentrated emulsion (HIPE) was transferred into the glass mold and polymerized at 60°C in a circulating oven for 24 h. The polymerized emulsion was then dried at 70°C for 24 h. The surfactant and unreacted initiator were extracted by methanol and water for 24 h in a Soxhlet apparatus, respectively. The same procedure was used for the polyHIPE solid foam prepared without using porogenic solvent. In this case, the organic phase includes styrene, divinylbenzene, and Span80.

Preparation of Conducting Composite Foams

The well-dried extracted polyHIPE support foam was first dipped in an oxidant solution (FeCl₃ dissolved in methanol) for 1 h. Subsequently, the oxidant-saturated support foam was dried for 20 min in air. Thereafter, the foams were dipped into monomer solutions containing 0.5 mL pyrrole dissolved in various solvents and certain amounts of dopants for 3 h at room tem-

perature. The monomer solution penetrates within the foam, and polymerization takes place in the presence of support foam. The composite foam was dried under vacuum for 48 h before conductivity measurement.

Foam Characteristics

Solvent Absorption. The solvent absorption capacity of the polyHIPE supports (1 × 1 × 0.4 cm³) was measured by immersing the polymeric foams within 10 mL of various solvents at room temperature for 24 h. The increase in weight due to solvent absorption was measured gravimetrically. The absorption capacity was calculated as solvent uptake per unit mass of dry foam.

Chemical Structure. The infrared spectra of polymeric foams were studied by using a Fourier-transform infrared instrument (FTIR, Shimadzu, Japan) apparatus. All spectra in the range of 400–4000 cm⁻¹ with a 2 cm⁻¹ spectral resolution were obtained from compressed KBr pellets in which the conducting foam samples were dispersed within.

Surface Area and Intercellular Pore Size

The specific surface area of the support foam was calculated from N₂ adsorption/desorption data at 77 K by using a Brunauer-Emmett-Teller (BET) model on a gas adsorption analyzer (Belsorp, Japan). The sample in the form of small particles was degassed at 100°C overnight under vacuum before data collection. The average intercellular pore size of support foam was obtained by BJH method.

Electrical Conductivity Measurement. For electrical conductivity measurement, a thin slab (1 × 1 × 0.4 cm³) was cut from the conducting composite foam. Two flat edges of the slab were painted with silver paste to adhere thin copper sheets to the edges. The sheets act as two electrodes for subsequent two-point direct current (DC) conductivity measurements.

Foam Morphology. The morphology of the foams was studied by means of scanning electron microscopy (SEM, XL30, Philips, Netherland) apparatus. Samples from polyHIPE foams were fractured in liquid nitrogen. The fracture surface was coated with a thin layer of gold before microscopy observation.

RESULTS AND DISCUSSION

Support Foam Characteristics

The characteristics of polyHIPE support foam can influence the composite foam properties. The highly open-cell porous structure of support foam improves the monomer solution penetration within the foam and facilitates the effective polymerization of pyrrole on the foam internal surface. The solvent absorption/adsorption capacity of the support foams is shown in Table I. The absorption capacity followed the order chloroform > cyclohexane > acetonitrile. A very small difference between the solubility parameter of the chloroform and the poly(St-co-DVB) foam, respectively, 18.95 and 18.60 (MPa)^{1/2}, resulted in the highest solvent absorption capacity.²⁴ For the polyHIPE2 support foam with higher specific surface area, the absorption capacity is much higher than that of the polyHIPE1 prepared without using porogenic solvent.

Table I. Effect of Solvent Type on Its Absorption Capacity and PPy Formation on the PolyHIPE Support Foam

Solvent	Solubility parameter (MPa) ^{1/2}	Donor number(DN) (kcal mol ⁻¹)	Solvent/ PolyHIPE (gr/gr) ^a	PPy/ PolyHIPE (gr/gr)
Chloroform	18.9	0.0	79 (133)	2.1
Acetonitrile	24.4	14.1	30 (95)	1.0
Cyclohexane	16.8	4.8	35 (112)	13.6

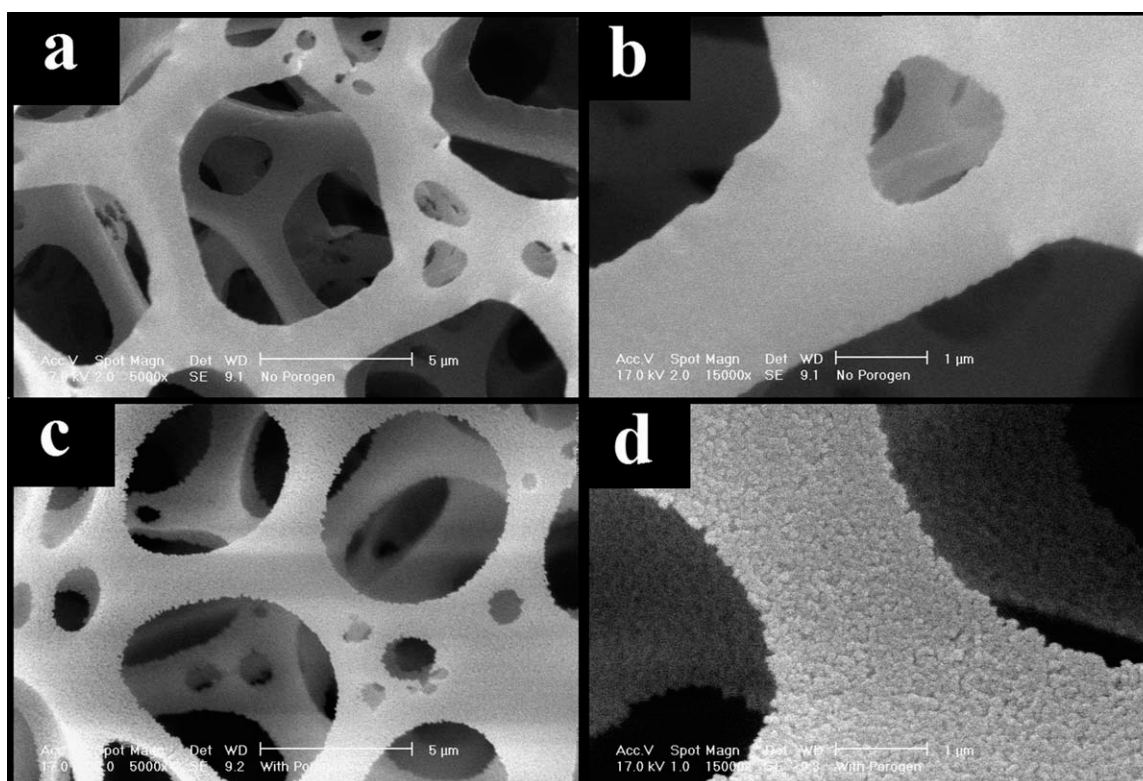
^aThe values inside the parentheses correspond to the volume of adsorbed solvent per unit volume of the dry foam (cm³/ cm³). All the support foams were prepared by using the porogenic solvent.

Figure 1 shows the SEM micrographs of the poly(St-co-DVB) support foams prepared without and with 1,2 dichlorobenzene as porogen. The polyHIPE1 support has a smooth cell wall surface having no observable nanopores on it [Figure 1(b)]. For the polyHIPE2 support, the phase separation occurred within the continuous organic phase during the polymerization process which resulted in nanopore formation [Figure 1(d)]. The existence of nanopores considerably increased the foam specific surface area from 3 to 56 m²/g and thereby the foam solvent adsorption capacity. Figure 2(a) shows the N₂ adsorption/desorption isotherm and the hysteresis loop of the polyHIPE2. The foam demonstrates a type II adsorption isotherm and a type H4 hysteresis loop. The pore size distribution is quite broad with pore size ranged from 1.5 to 96 nm in the foam cell wall [Figure 2(b)]. The mean nanopore radius is close to 19 nm.

The narrow hysteresis loop and broad pore size distribution indicate a good interconnectivity between nanopores.²⁵ The specific surface area calculated from BET treatment based on N₂ adsorption/desorption data for the polyHIPE2 is close to 56 m² g⁻¹, which is much higher than that of the polyHIPE1 prepared without using porogen in the emulsion foam. Therefore, the polyHIPE2 support foam with higher specific surface area was used to synthesize the conducting poly(St-co-DVB) polyHIPE/PPy composite foams.

Conducting Composite Foam

Effect of Solvent Type. The solvent medium for polymerization of pyrrole has a strong effect on the surface morphology and electrical conductivity of resulting polyHIPE composite foams. The conductivity values of the composite foams prepared using various solvents in the absence of sulfonate derivatives (dopants) are shown in Table II. The highest electrical conductivity, 1.54 × 10⁻² S cm⁻¹, is obtained for the composite polyHIPE foam prepared using cyclohexane as monomer solvent, whilst the lowest conductivity, 5.67 × 10⁻⁴ S cm⁻¹, corresponds to the foam prepared with chloroform solvent. The aprotic solvent can facilitate the oxidative chemical polymerization of pyrrole. In fact, the polymerization of pyrrole proceeds through the radical cations in which the solvent molecules may attack the radical cations in the reaction media. Thus, the use of solvent with lower basicity character or donor number increases the life of radical cation, polymerization degree, and polymer conductivity.²⁶ In addition, the low conducting efficiency obtained by

**Figure 1.** SEM micrographs of poly(St-co-DVB) polyHIPE support foams prepared: (a,b) without using porogen and (c,d) with using 1,2 dichlorobenzene porogen.

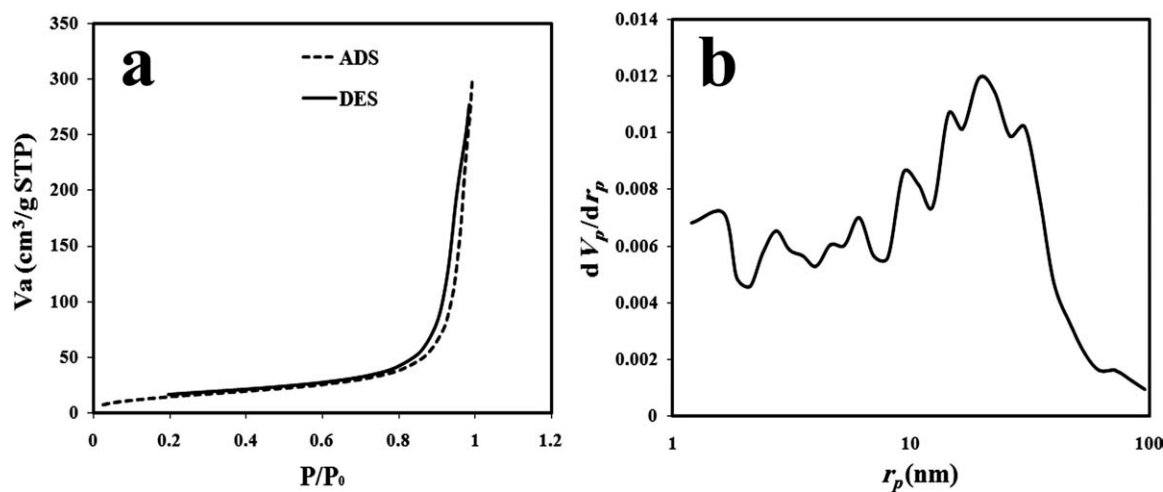


Figure 2. (a) The N_2 isotherm and (b) the pore size distribution of the polyHIPE2 support foam.

using chloroform can be explained by morphological arguments.

Figure 3 shows the SEM micrographs of the free dopant composite foams prepared by using various solvents. As shown, the nature of the solvent influenced the characteristics and surface morphology of the PPy film on the foam surface. The pyrrole polymerization occurs at the interface between the pyrrole solution and $FeCl_3$ adsorbed on the polyHIPE surface. It is obvious that the differences in surface morphology are directly connected to the PPy particles that converge on the void surface. The morphology of composite foam prepared using chloroform is much like that of the uncoated polyHIPE support foam [Figures 1(d) and 3(b)]. Using chloroform resulted in conducting film coating that fills the craters of the polymeric substrate, and subsequently, the formation of PPy film with discrete loose structure and low electrical conductivity on the void surface. When acetonitrile was used as the monomer solvent, the larger and more compact PPy particles seems to form on the nanopore surface. This more compact PPy structure may be responsible for a higher electrical conductivity. The highest electrical conductivity was observed for the composite foam prepared by using cyclohexane. The use of cyclohexane led to a PPy film that fits the surface roughness of the foam support. However, the formation of a thin layer of highly compact PPy film on the internal surface of support foam considerably increased the electrical conductivity [Figure 3(f)].

Although using the cyclohexane resulted in the highest foam electrical conductivity, the use of chloroform in the presence of a suitable dopant showed a remarkable conductivity enhancement.

Effect of Dopant Type. The electrical properties of conducting composite foams can significantly depend on the conjugation length of the conducting polymer used. In fact, the increase of conjugation length leads to increasing the PPy electrical conductivity.^{27–28} The incorporation of dopant in the monomer solution may result in critical change of the conjugation length. The structure of the conducting PPy chains contains the sulfonic derivatives, i.e., NDSA and DBSNa as anionic dopants are

shown in Figure 4. These anions are incorporated into the polymer structure to maintain electroneutrality.¹⁷ Table II indicates the effect of various monomer solvents and dopants on the electrical conductivity of resulting composite foams. The composite foam prepared using cyclohexane in the absence of sulfonic compounds showed the highest conductivity as compared to the other solvents used. On the contrary, using cyclohexane in the presence of sulfonic compounds such as DBSNa resulted in lower electrical conductivity (Table II). This behavior can be concerned to lower solubility of the anionic dopants in the non-polar cyclohexane solvent. In fact, the lower solubility restricts the incorporation of the sulfonic dopant into the PPy chains and consequently decreases the electrical conductivity. Nevertheless, the highest conductivity improvement corresponds to the composite foam synthesized by using chloroform and DBSNa dopant, when compared to the undoped and NDSA doped-composite foams (Table II).

Figure 5 shows the infrared spectra of the P(St-co-DVB) polyHIPE/PPy composite foam prepared using various dopants in the chloroform. The bands located close to 1540 cm^{-1} and 1450 cm^{-1} in the spectrum of the conducting foams correspond to the asymmetric and stretching vibration of the pyrrole ring, respectively. As the conjugation length increases, the asymmetric to symmetric vibration intensity ratio decreases.^{27–28} Consequently,

Table II. Electrical Conductivities of Poly(St/DVB)/PPy PolyHIPE Composite Foams Prepared by Using Various Solvents and Dopants

Dopant	Conductivity $\times 10^3$ (S cm^{-1})		
	Chloroform	Acetonitrile	Cyclohexane
Cl (no dopant)	0.567 (0.040)	3.976 (0.881)	15.444 (0.781)
NDSA	1.274 (0.108)	5.369 (2.014)	5.270 (0.047)
DBSNa	83.330 (40.404)	1.742 (0.089)	0.076 (0.021)

^aThe values outside and inside the parentheses correspond to the conductivity values of the composite foams with aging time of 1 day and 30 days, respectively.

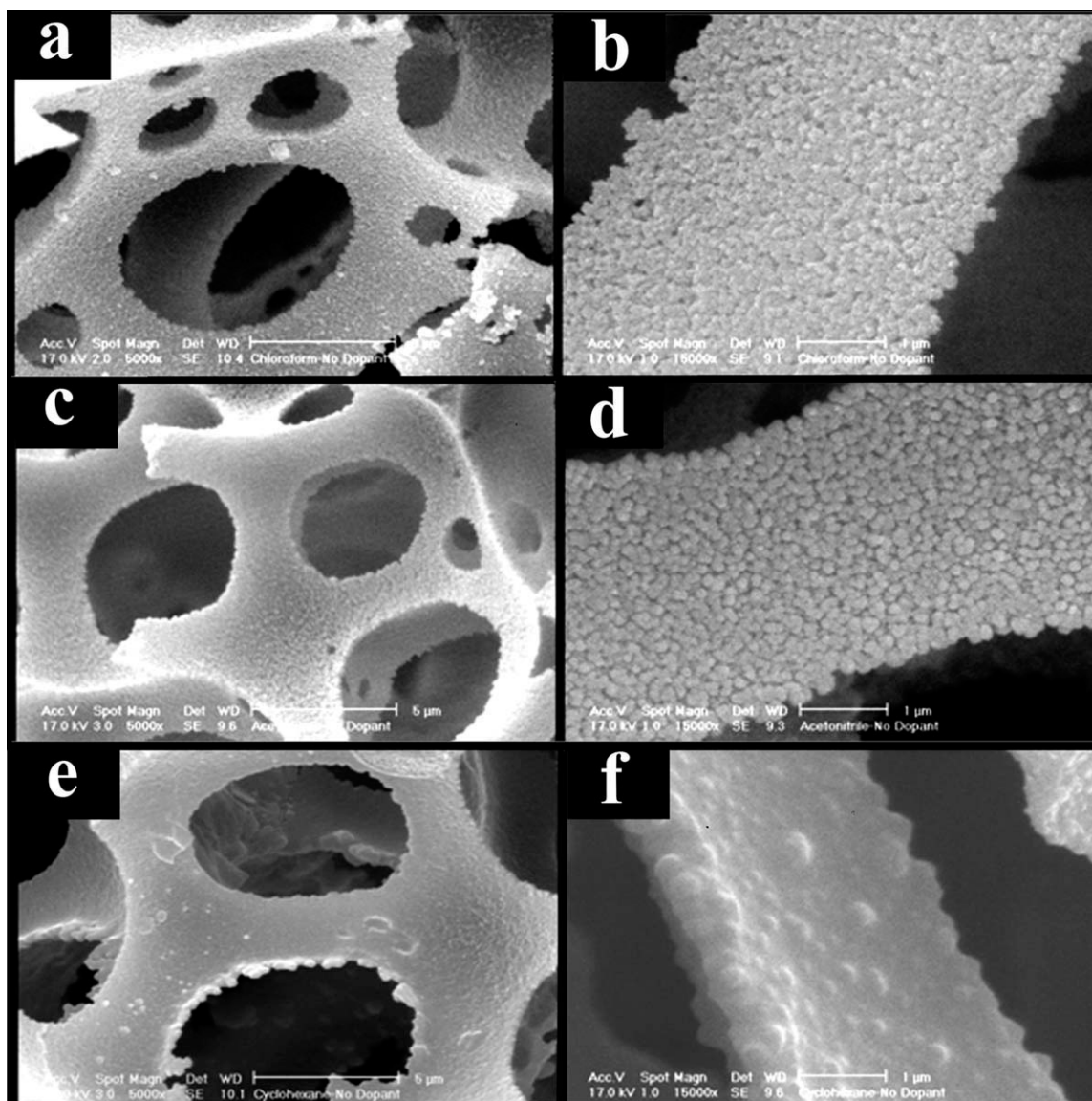


Figure 3. SEM micrographs of undoped-composite foams prepared by using various solvents: (a,b) chloroform; (c,d) acetonitrile; and (e,f) cyclohexane.

the intensity ratio of the vibration bands, i.e., A_{1540}/A_{1450} , can be evaluated as a relative measurement criterion of conjugation length. The electrical conductivity and the relative intensity of ring stretching bands for undoped and doped-composite foams are listed in Table III. The results showed that the DBSNa considerably improved the electrical conductivity of the composite

foam because of its lower A_{1540}/A_{1450} value. The formation of PPy film in the presence of DBSNa on the hydrophobic surface of support foam seems to have a more extended conjugation length. Higher conjugation length can be attributed to a lower number of conjugation-interrupting linkages and planar configuration of the pyrrole monomer during bond formation.

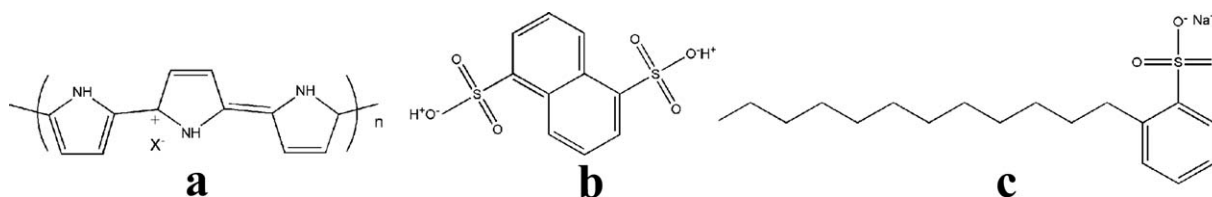


Figure 4. Chemical structure of (a) PPy, (b) NDSA, and (c) DBSNa.

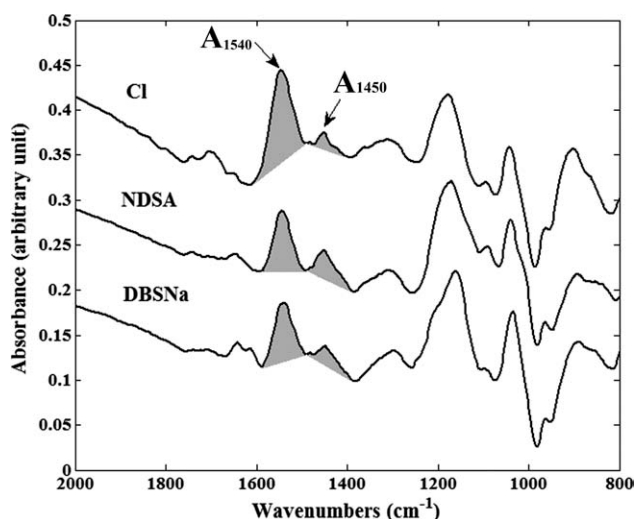


Figure 5. FTIR spectra of the composite foams prepared by using different dopants in the chloroform (vibration peaks at 1540 and 1450 cm^{-1} are highlighted in the spectra).

It is well known that the morphology of conducting polymer and its bulk properties including electrical conductivity may be tailored by employing suitable anionic surfactants as dopants, such as sulfonate derivatives of aromatic compounds.²⁹ Figure 6 shows the SEM micrographs of the doped-composite foams prepared by using chloroform and different dopants, i.e., DBSNa

Table III. Electrical Conductivity of Composite Foams Synthesized by Using Different Dopants (in All Cases the Chloroform is the Monomer Solvent)

Dopant	PPy/ PolyHIPE (gr/gr)	Conductivity $\times 10^3$ (S cm^{-1})	$A_{1540}/$ A_{1450}
Cl (no dopant)	2.1	0.567	7.64
NDSA	7.0	1.274	2.62
DBSNa	6.4	83.33	1.96

and NDSA. Morphological studies show that the presence of dopants in the polymerization mixture strongly influences the morphology of the synthesized PPy film on the internal surface of support foam. In fact, the molecular structure of dopant influences the solubility of PPy particles in organic solvent. In the case of poly(St-*co*-DVB)/DBSNa-PPy composite foam, the long alkyl chain of DBSNa reduces the PPy interchain interaction and increases the mobility along the polymer chain. Thus, a higher solubility of PPy chains in a relative acidic solvent, i.e., chloroform, was expected. The enhanced solubility of PPy in the presence of DBSNa facilitates the formation of a highly compacted PPy film on the foam surface. Compact structure of PPy-DBSNa film results in more easy transfers of electrical charge carriers, and thereby higher foam electrical conductivity in comparison with the composite foam doped by NDSA (Table III). DBSNa contains aromatic rings that improve regular parallel configuration of the PPy chains on the support foam surface.

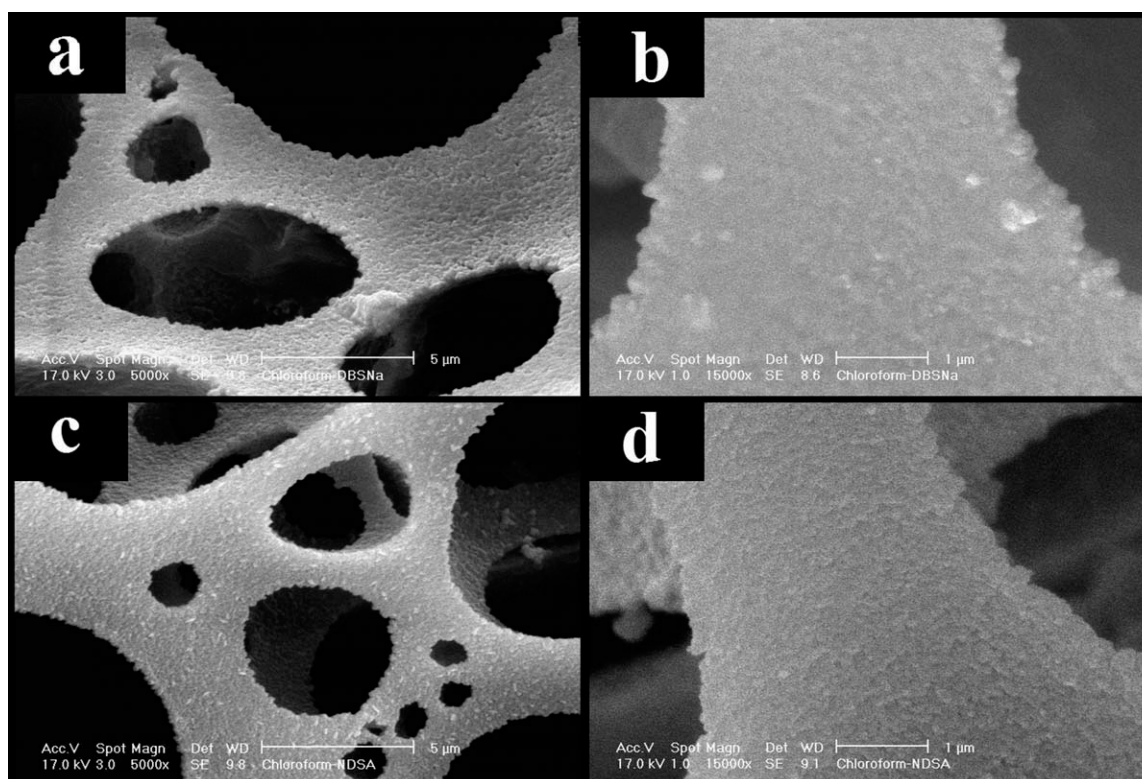


Figure 6. SEM micrographs of poly(St/DVB)/PPy poly(HIPE) foams prepared in the presence of different dopants: (a,b) DBSNa and (c,d) NDSA (in all cases the chloroform is the monomer solvent).

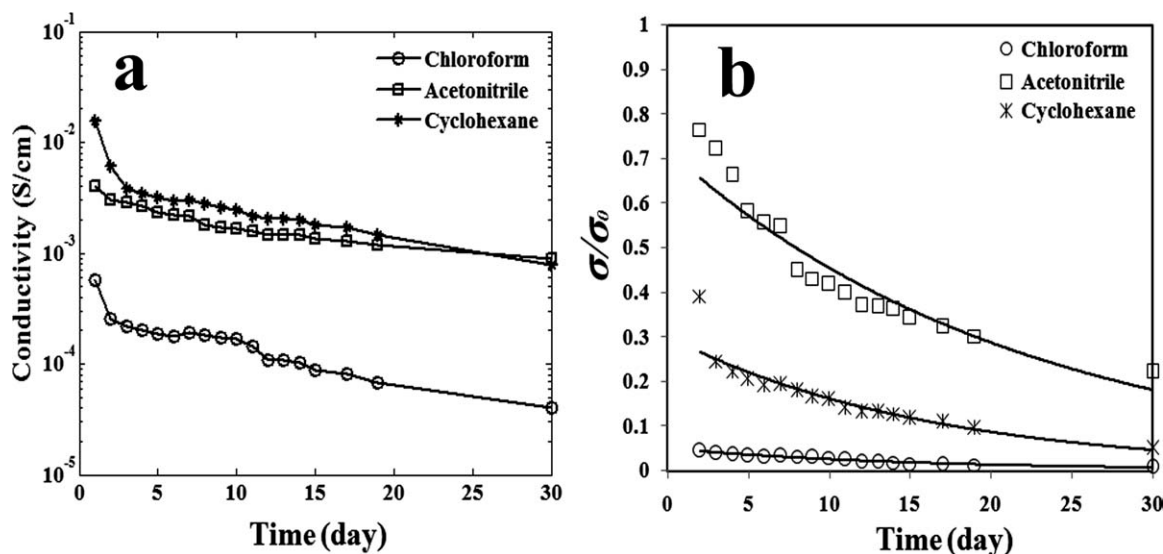


Figure 7. Effect of solvent type on the electrical conductivity and conductivity ratio of the undoped-composite foams versus aging time.

In other words, the greater degree of planarity of the PPy chains in the presence of DBSNa molecules resulted in higher electrical conductivity of the resulting composite foam. On the other hand, the number of sulphonate groups of a dopant molecule can influence the foam conductivity. Increasing the number of sulfonate groups causes the conductivity to decrease.³⁰ In this case, a larger number of sulfonate groups in NDSA dopant may lead to stronger attraction of positive charges in PPy chain. This stronger attraction increases both the chain irregularity and the degree of charge localization in the PPy–NDSA film, thereby lowering the electrical conductivity of the composite foam. Interestingly, the amount of coated PPy in the presence of dopant is higher than the PPy coated without using any dopant (Table III). This behavior indicates that a part of dopant can be

incorporated into the PPy chain structure; similarly as the Cl⁻ arise from the oxidant.

Aging of Conducting Composite Foams. In general, the environment stability of PPy is related to its physical aging and the extent of reaction of the polymer backbone with oxygen or water that exists in the surrounding foam atmosphere. Figures 7(a) and 8(a) show the electrical conductivity of composite foams prepared by using various monomer solvents and dopants versus aging time at room temperature, respectively. The undoped-composite foam with oxidant anions (Cl⁻) reacts easily with environment oxygen; hence, a fast decay in electrical conductivity was observed [Figure 7(a)]. The PPy film is sensitive to moisture because of leaching the counter ion.³¹ In addition, the

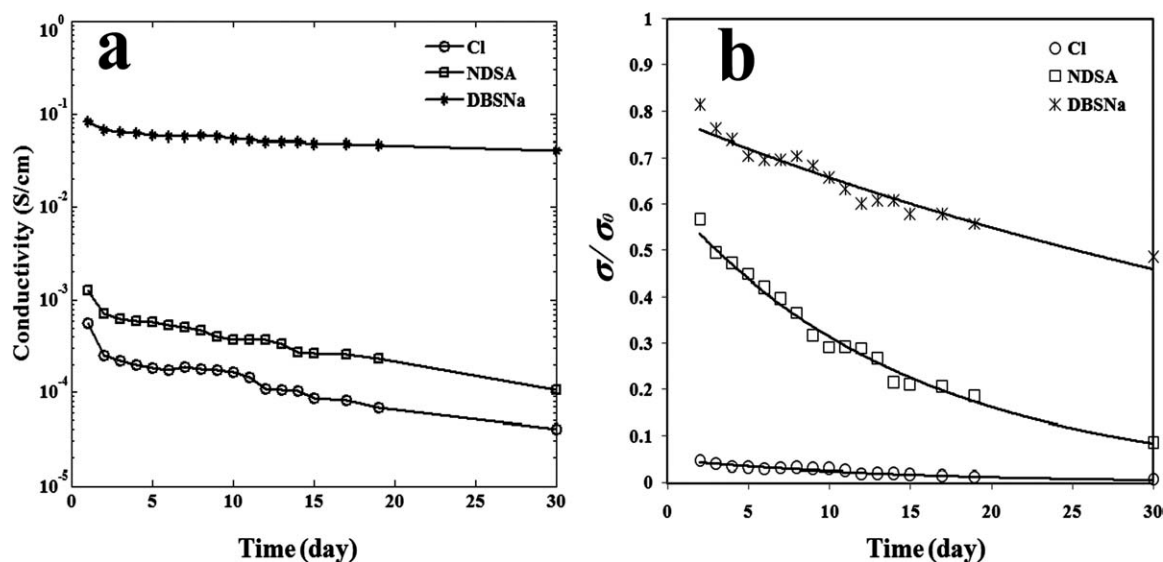


Figure 8. The effect of dopant type on the conductivity and conductivity ratio of composite foams versus aging time (in all cases the chloroform is the monomer solvent).

morphological aspects may influence the degradation behavior of PPy film. In the absence of sulfonate derivatives, the spreading PPy thin layer on the support foam facilitates the oxygen attack and conducting polymer degradation. The high stability of doped-composite foams originates from less structural defects exists on these conjugated systems. The presence of aromatic dopant during polymerization enhances the PPy chains regularity and presumably preserves the electrical properties against the environmental oxygen and moisture [Figure 8(a)].

The electrical conductivity ratio ($\frac{\sigma}{\sigma_0}$) of the composite foams was plotted as a function of aging time [Figures 7(b) and 8(b)]. The aging mechanism of all composite foams follows a first-order kinetic aging model according to following equation:

$$\sigma = \sigma_0 e^{-kt} \quad (1)$$

where σ_0 and σ are the initial electrical conductivity and conductivity at any time, respectively. This suggests that the reaction of oxygen and moisture with PPy backbone is the limiting step in electrical conductivity decay of the composite foams. This type of degradation mechanism has been used successfully for predicting the PPy degradation.³² As mentioned earlier, the use of dopants has strong effect on the morphology of PPy film coated on the foam surface. A relative stability of DBSNa doped PPy film [Figure 8(a)] may be due to the protected smooth surface of conducting film that inhibits the oxygen and moisture penetration. For the undoped PPy film (PPy-Cl), a highly porous structure without any protecting layer facilitates the penetration of oxygen and air moisture within the conducting layer. This leads to lower stability of undoped PPy in comparison with the PPy doped with sulfonate derivatives.

CONCLUSIONS

Conducting poly(St-co-DVB)/PPy polyHIPE composite foams were successfully synthesized via oxidative chemical polymerization using various monomer solvents and dopants. Conductivity measurements showed that the foam electrical properties strongly depend on the solvent and dopant type used. The observed differences between the electrical conductivities of the composite foams were connected to the morphological and chemical structure of the PPy film formed on the polyHIPE support foam. Highly compact structure of undoped PPy film prepared by using cyclohexane solvent resulted in a higher electrical conductivity. The highest conductivity and environmental stability of the DBSNa-doped-composite foam can be attributed to the more extended conjugation length and smooth surface of doped PPy film formed in the presence of chloroform solvent. The aging of conducting foams follows a first-order kinetic aging model that indicates the reaction of oxygen and air moisture is the limiting step in the PPy film degradation.

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