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Interfacial physicochemical properties of functionalized conducting polypyrrole particles

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Abstract

Polypyrrole-coated polystyrene latex particles bearing reactive *N*-succinimidyl ester functional groups (PS-PPyNSE$_{75}$) were prepared by the in situ copolymerization of pyrrole **1** and the active *N*-succinimidyl ester-functionalized pyrrole **2** (pyrroleNSE), with initial **1**:**2** fractions of 25:75 (%) in the presence of sterically stabilized polystyrene (PS) latex particles. PS particles were prepared by dispersion polymerization leading to particles having a diameter of 600 ± 10 nm. The PS-PPyNSE$_{75}$ particles were characterized in terms of surface morphology and chemical composition. Surface analysis of the colloidal materials by X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM) indicated a substantial coating of PS by the reactive conducting copolymer. Infrared spectroscopy permitted to detect pyrroleNSE repeat units at the surface of the particles indicating that **1** and **2** did indeed copolymerise.

Reactivity of the PS-PPyNSE$_{75}$ particles has been investigated using 2-aminoethanol and 2-mercaptoethanol, two model molecules bearing functional groups borne by proteins. Incubation of the particles with these model molecules clearly showed that the particles are highly reactive towards amine and thiol groups. The functionalized particles were then tested as bioadsorbents. PS-PPyNSE$_{75}$ particles were found to be effective in attaching an aminated biotin. The Biotin-decorated PS-PPyNSE$_{75}$ latex particles were incubated with avidin with a result of a significant change in the surface composition that is in line with the attachment of the protein by specific binding to biotin.

Keywords: Polypyrrole dispersions; Reactivity; Protein

1. Introduction

The synthesis of polypyrrole-coated latex particles is very well documented [1]. The general method of preparation of these polypyrrole dispersions consists in the oxidative polymerization of pyrrole in the presence of polystyrene (PS) latex [2,3], polyurethane [4], poly(alkyl methacrylate) [5–7], polypropylene [8], etc. These studies concerned essentially homopolypyrrole either coated or embedded in the support. However, for biomedical application purposes, it is very desirable to immobilize biomolecules by covalent bonding to the carrier surface. This has the advantage to minimize physisorption which can under certain circumstances result in leaching the biomolecule of interest.

There are several ways of surface functionalization of particles and other supports [9,10]. Carboxylic acid and amine are among the most popular functional groups used for the attachment of e.g. proteins. However, activation is needed for the carboxylic acidic group whereas some conditions have to be met necessarily for the amine to be reactive towards for example the carboxylic acid or aldehyde groups [9].

Taking into consideration the advantages and shortcomings of recommended methods in the published literature on surface functionalization of particles, we proposed to modify the surface of particles by active ester

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groups such as *N*-succinimidyl ester [11–14]. This approach has also been suggested for the functionalization of carbon nanotubes in view of attaching proteins [15]. The rationale for choosing the *N*-succinimidyl ester (NSE) group is that it is well known to react readily with amines (e.g. from a protein residue) under very mild conditions to form the corresponding amides.

In our recent bio-interfacial studies, we have demonstrated that NSE-modified polypyrrole-silica nanocomposites [12] and NSE-functionalized polypyrrole-polystyrene particles [14] massively attach (up to 6–8 mg/m^2) human serum albumin (HSA). However, the amount of HSA immobilized on NSE-functionalized polystyrene–polypyrrole particles surprisingly depends on the method of preparation of the polystyrene support, that is by way of dispersion [14] or emulsion polymerization [11,13]. As the covalent protein–particle bonding is undoubtly driven by the reactivity of active *N*-succinimidyl ester groups towards amino acid residues such as lysine and cysteine, it is interesting to investigate thoroughly the reactivity of NSE-functionalized polystyrene–polypyrrole particles.

This paper reports on the preparation, characterization and reactivity of polypyrrole-coated polystyrene latex particles bearing surface *N*-ethyl succinimidyl ester groups (see Fig. 1). The dispersion polymerization of styrene was chosen for the preparation of the polystyrene core, choice dictated by a better colloidal stability obtained by this method compared to the emulsion polymerization. Poly(*N*-vinyl pyrrolidone) was used as a steric stabilizer. The reactive coatings consist of copolymers of pyrrole **1** and *N*-ethyl succinimidyl ester substituted pyrrole **2** (pyrroleNSE) in the initial 25:75 (%) ratio. This ratio was found to be optimal for HSA attachment [14]. The latex particles were characterized by FTIR, X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM). Selected batches of particles were incubated with model molecules in order to monitor the formation of interfacial bonds between the carrier (polypyrrole particle) and the target molecule. These model molecules, namely aminoethanol and mercaptoethanol, bear functional reactive groups (amine, thiol), which are contained in proteins. Furthermore, the *N*-ethyl succinimidyl ester-functionalized particles were evaluated as a support for the bioconjugate system biotin–avidin. First biotin was immobilized on the polypyrrole particles, and then the biotin-decorated carriers were incubated with avidin.

2. Experimental

2.1. Synthesis of N-succinimidyl ester-functionalized pyrrole

The synthesis of *N*-succinimidyl ester-functionalized pyrrole was reported in detail in Ref. [11,12].

2.2. Synthesis of uncoated polystyrene latex particles

The sterically stabilized polystyrene latex particles were prepared using the procedure described by Lascelles and Armes [3]. Briefly, isopropyl alcohol (400 mL) was added to a 2-L round-bottomed flask fitted with a condenser and a mechanical stirrer. The poly(*N*-vinylpyrrolidone), PNVP (Fluka, molecular weight 360,000), steric stabilizer (7.5 g) was then added and allowed to dissolve. The reaction vessel was heated to 75 °C under a nitrogen blanket for 6 h. A solution of azo-*iso*-butyronitrile initiator (0.75 g, Aldrich) pre-dissolved in styrene monomer (83.3 mL, Aldrich product purified by passing through a column of activated neutral alumina) was then added at once using a syringe. The mechanical stirrer was set at a constant speed of 2000 rpm. The polymerization was allowed to proceed for 24 h before cooling to room temperature. The resulting latex particles were purified by repeated centrifugation-redispersion cycles, replacing successive supernatants with distilled water.

2.3. Synthesis of surface functionalized polypyrrole-coated polystyrene latex particles

$FeCl_3 \cdot 6H_2O$ oxidant (0.9 g, Aldrich) was dissolved in 13 mL of an aqueous dispersion of the PS latex particles (1 g dry weight), in a screw-cap bottle with a magnetic stirrer. Pyrrole 1 (Fluka, purified by passing through a column of activated basic alumina (Acros)) and 2 were premixed in 25:75 (2.2×10^{-3}:6.7×10^{-3} mol) molar ratios and then added via syringe. The polymerization was allowed to proceed for 24 h. The coated latex particles were then purified by repeated centrifugation-redispersion cycles (successive supernatants being replaced by deionized water) in order to remove the unwanted inorganic products ($FeCl_2$ and HCl) produced during the pyrrole polymerization. The as-prepared conducting polymer particles are abbreviated PS-PPyNSE$_{75}$ where *75* stands for the initial molar fraction of PyrroleNSE.

2.4. Reactivity of PS-PPyNSE$_{75}$ towards molecules and proteins

2-Aminoethanol, 2-mercaptoethanol (Aldrich products), biotin ethylenediamine (Biot-NH_2) and avidin (Sigma products) were used as received. All measurements were performed at room temperature and pressure. A phosphate buffer solution (PBS) was prepared by dissolving 1.18 g of potassium phosphate KH_2PO_4 and 4.32 g of sodium phosphate $NaHPO_4$ in 1 L of H_2O to give a solution of pH 7.4.

The reactivity was investigated by the reaction of PS-PPyNSE$_{75}$ with R-NH_2 and R-SH. In this reaction the nucleophilic amine (or thiol) undergoes acylation with the succinimidyl ester group to produce the amide product. Amine (or thiol) predissolved in PBS solution were added to

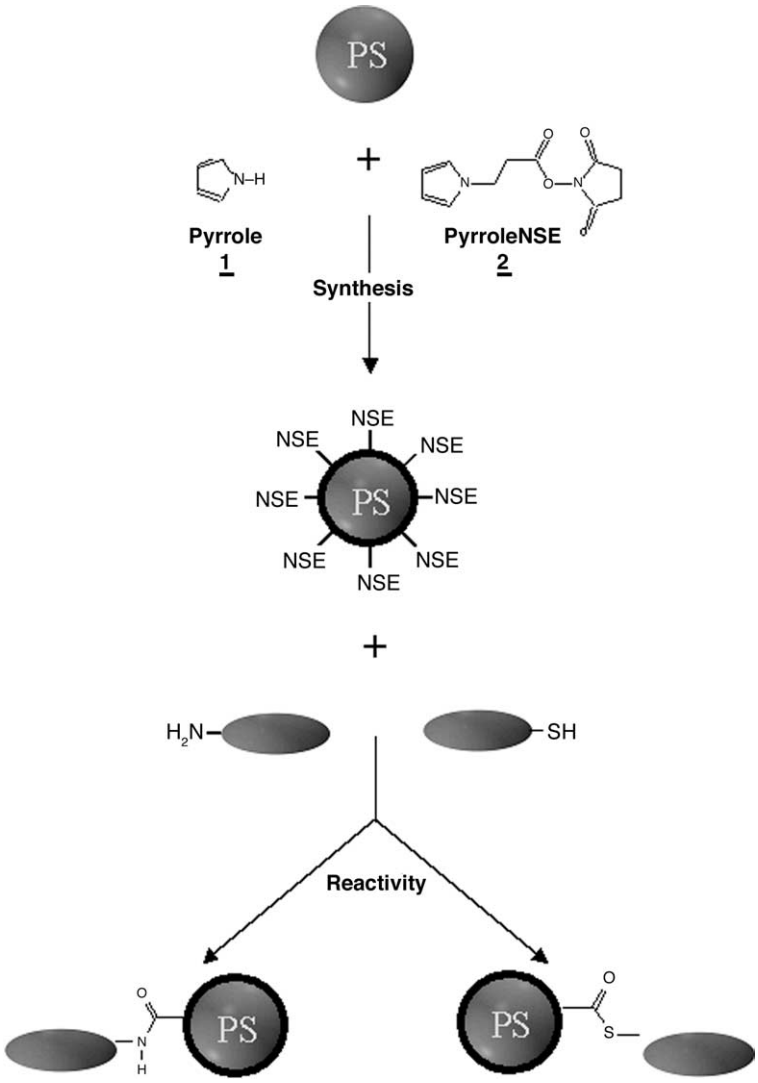

Fig. 1. Schematic representation of the synthesis and reactivity of a polypyrrole-coated, PNVP-stabilized polystyrene latex particles bearing surface reactive *N*-ester succinimidyl (NSE) groups. Chemical reactivity towards amine- and thiol-bearing molecules is shown.

the PS-PPyNSE$_{75}$ latex particles (0.15 g dry weight, total volume 2 mL) and the mixture was left to react for 16 h. After incubation, the samples were centrifuged and washed thoroughly with distilled water to remove all the free and/or loosely bound amine (or thiol) molecules. The products were characterized by FTIR and XPS spectroscopy.

A solution of biotin ethylenediamine in PBS buffer pH 7.4 was added to a latex suspension (0.15 g dry weight). The final biotin ethylenediamine concentration was 5×10^{-4} and 10^{-2} M. The reaction was carried out by gentle mixing for 16 h at room temperature. The product was isolated by several centrifugation/redispersion cycles to remove unreacted Biot-NH$_2$ and dried under vacuum. Then various concentrations of avidin (0.1; 0.5; 1; 5 mg/mL) in PBS buffer solution were added to a suspension of the Biot-NH$_2$-modified PS-PPyNSE$_{75}$ particles (0.15 g). The reaction was allowed to proceed for 12 h. The product was purified and analyzed by XPS and FTIR spectroscopy.

2.5. Analytical techniques

Scanning electron micrographs were obtained with a Cambridge 120 that is completely controlled from a computer workstation. The filament is a zirconated tungsten and the accelerating voltage was set at 20 kV. All specimens were coated with gold prior to analysis in order to avoid or limit static charging effects.

FT-IR spectra of PS particles and PS-PPyNSE$_{75}$ latexes (KBr disks) were recorded using a Nicolet Magna 550 Series II instrument. Spectra were typically averaged over 20 scans at 4 cm^{-1} resolution.

X-ray photoelectron spectroscopy (XPS) measurements were performed using a Thermo VG ESCALAB 250 instrument equipped with a monochromatic Al Kα X-ray source (1486.6 eV). The X-ray spot size was 650 μm. The pass energy was set at 150 and 40 eV for the survey and the narrow scans, respectively. Additional high resolution C1s

regions were recorded using a pass energy of 10 eV. Charge compensation was achieved with a combination of electron and argon ion flood guns. The energy and emission current of the electrons were 4 eV and 0.35 mA, respectively. For the argon gun, the energy and the emission current were 0 eV and 0.1 mA, respectively. The partial pressure for the argon flood gun was 2×10^{-8} mBar. These standard conditions of charge compensation resulted in a negative but perfectly uniform static charge. Data acquisition and processing were achieved with the Avantage software, version 1.85. Spectral calibration was determined by setting the main C1s component at 285 eV [16]. The surface composition was determined using the manufacturer's sensitivity factors. The fractional concentration of a particular element A (% A) was computed using:

$$\%A = \frac{(I_A/s_A)}{\sum(I_n/s_n)} \times 100$$

where I_n and s_n are the integrated peak areas and the sensitivity factors, respectively.

3. Results and discussion

3.1. Morphology, surface chemical composition, and stability of reactive PS-PPyNSE particles

SEM images of PS and PS-PPyNSE$_{75}$ particles are shown in Fig. 2. The uncoated PS particles (Fig. 2(a)) are spherical, nearly monodisperse ($d=600\pm10$ nm, polydispersity index = 1.003), and have a smooth surface. In contrast, PS-PPyNSE$_{75}$ particle surface (Fig. 2(b)) consists of small, raised granular polypyrrole nodules that were previously observed for thick homo-polypyrrole coated onto micrometer-sized PS core [3].

FT-IR spectra, in the 1850–1350 cm^{-1} region, for PS and PS-PPyNSE$_{75}$ particles, and the PyrroleNSE monomer are shown in Fig. 3. The characteristic features of polystyrene (especially at 1453 and 1494 cm^{-1}) and the steric stabilizer PNVP (at 1605 cm^{-1}) are clearly visible in the two spectra. In addition, one can notice the appearance of new peaks in the PS-PPyNSE$_{75}$ spectrum: one characteristic of the pyrrole repeat units (at 1560 cm^{-1}) and three others (at 1739, 1781 and 1815 cm^{-1}) which are assigned to the succinimidyl ester groups of PyrroleNSE units as deduced from the spectrum of the pure PyrroleNSE monomer. This is a strong supporting evidence for the effective incorporation of this ester-functionalized monomer in the conjugated copolymer.

The stability of the NSE groups at the particle surface has been investigated using IR spectroscopy. Fig. 4 shows IR spectra of aged PS-PPyNSE$_{75}$ particles. One observes that during, at least, two weeks following the particle synthesis, the NSE groups are still present and stable at the surface of the particles. However, one month later, a slow but gradual

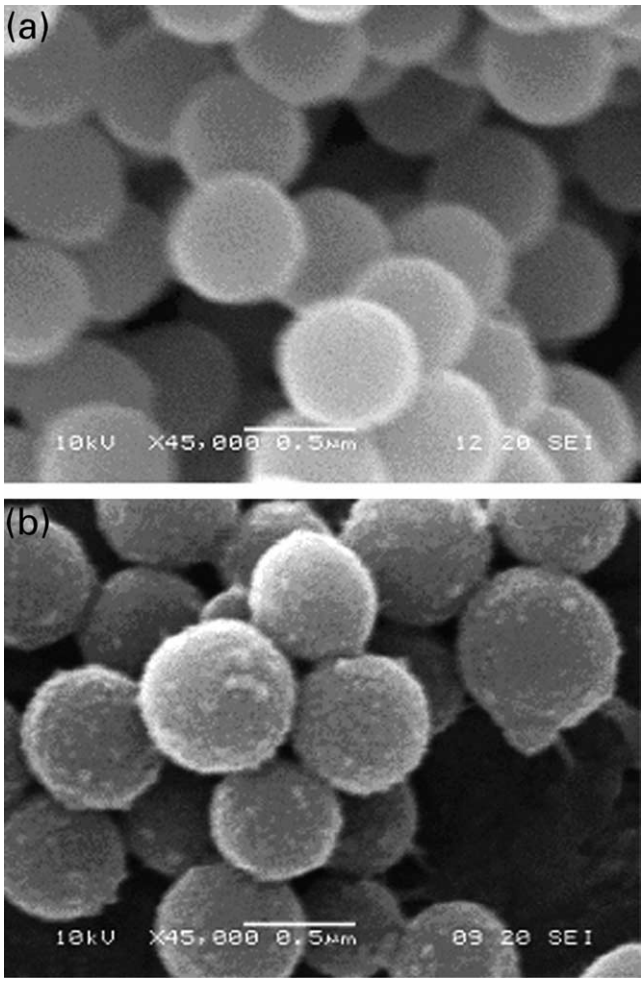

Fig. 2. SEM pictures of (a) uncoated PS particles; (b) PS-PPyNSE$_{75}$.

conversion of the NSE groups occurs back to the former carboxylic acid groups.

The pH-dependence of the stability of freshly

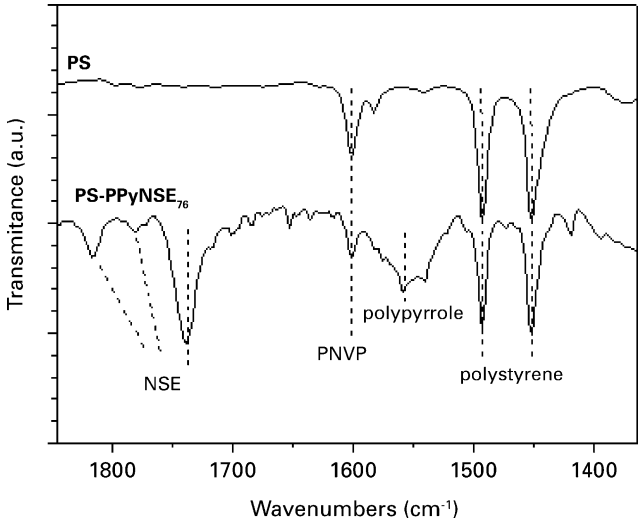

Fig. 3. FTIR spectra of PS latex particles, PyrroleNSE monomer and PS-PPyNSE$_{75}$ in the 1350–1850 cm^{-1} regions.

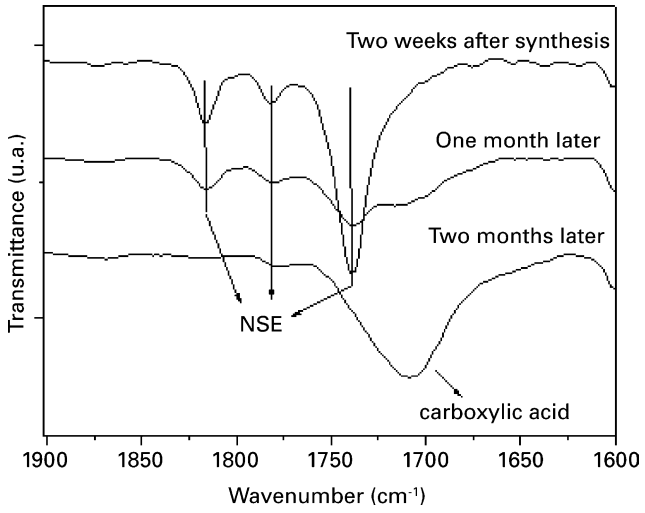

Fig. 4. FTIR spectra (1600–1900 cm^{-1}) of aged PS-PPyNSE$_{75}$ latex particles.

synthesized PS-PPyNSE$_{75}$ latex was also investigated. Fig. 5 shows the IR spectra of the particles after suspension at indicated pH. When the pH decreases below 5, the NSE characteristic IR band (at 1739 cm^{-1}) vanishes while a new one appears (at 1705 cm^{-1}) due to the carboxylic acid groups. Hydrolysis of the NSE groups can be monitored by plotting the intensity ratio A_{NSE}/A_{sty} of the bands (at 1739 cm^{-1}) related to NSE against the bands due to styrene (at 1453 and 1494 cm^{-1}). The inset in Fig. 5 shows the evolution of A_{NSE}/A_{sty} ratio versus pH and clearly indicates that above pH 5, no significant change appears in the spectra. This suggests that the conducting copolymer is chemically stable in the pH range that is found in physiological media.

Fig. 6 shows XPS survey spectra of PS and PS-PPyNSE$_{75}$. The main peaks are C1s, N1s and O1s centered

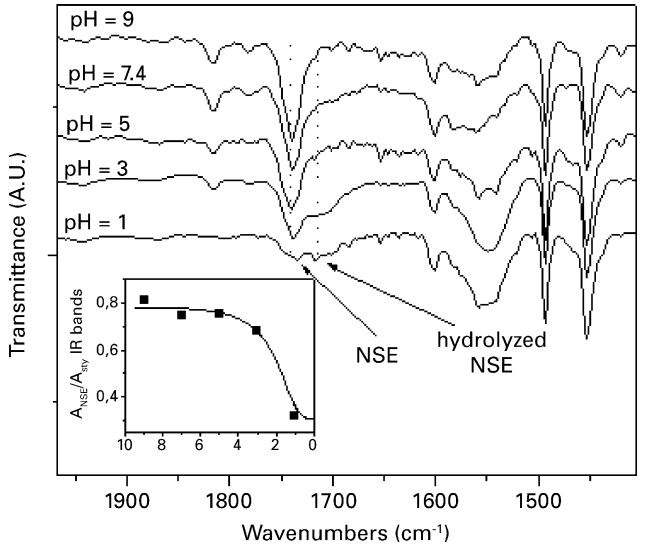

Fig. 5. pH-Dependence of the FTIR spectra of PS-PPyNSE$_{75}$ latex particles (1400–1960 cm^{-1}). Plot of A_{NSE}/A_{sty} ratio vs pH is shown in the inset.

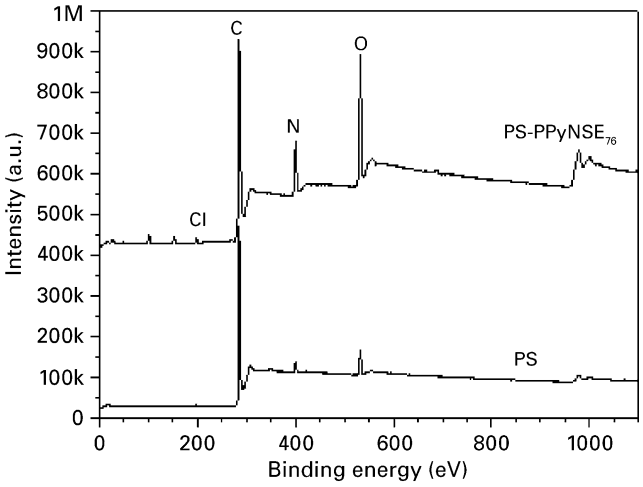

Fig. 6. XPS survey scans of PS and PS-PPyNSE$_{75}$ latexes.

at 285, 399.7 and 532 eV, respectively. In the case of PS-PPyNSE$_{75}$, Cl2p peak (198 eV) from the chloride dopants is also detected. N1s and O1s peaks are relatively intense by comparison to those from the uncoated PS latex particles. For example, the (O+N)/C atomic ratio is 0.09 and 0.36 for PS and PS-PPyNSE$_{75}$, respectively. The significant increase in the (O+N)/C atomic ratio is due to the presence of four oxygen and two nitrogen atoms per pyrroleNSE repeat unit from the conducting copolymer shell. We have shown recently that (O+N)/C atomic ratio increases gradually as the fraction of pyrroleNSE monomer increases in the comonomer feed ratio [14].

The high resolution C1s spectra are shown in Fig. 7 for the uncoated and polypyrroleNSE-coated PS particles. For PS, the C1s region exhibits a sharp main component centered at 285 eV and minor components centerd at 286.3 (C–N, C=N, C–O), 288 (N–C=O) and 291.5 eV (shake-up satellite). The latter component is characteristic of the aromatic pendent phenyl group from styrene repeat units. For PS-PPyNSE$_{75}$ particles, the C1s region experiences a

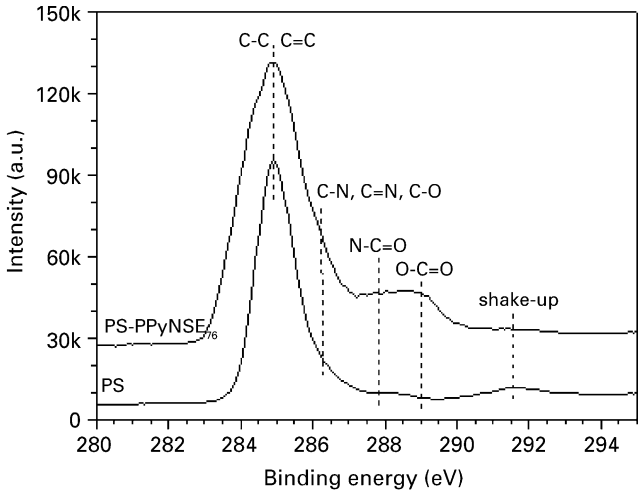

Fig. 7. High resolution C1s spectra of PS and PS-PPyNSE$_{75}$.

dramatic change resulting in the widening of the main component centered at 285 eV, due to the introduction of C–C/C–H, C–N and C=N bonds particularly. It is noteworthy that the ester carbon from the pyrroleNSE repeat units induces a distinct component at 289 eV that is not observed at the surface of the underlying PS latex particles. Moreover, the shake-up satellite, fingerprint of polystyrene, vanishes following coating by the conducting shell: the shake-up satellite fraction decreases from 4.3 to 2.7% on coating PS by $PPyNSE_{75}$. This significant decrease in the proportion of the shake-up satellite actually occurs only when polypyrrole coating is uniform and quite thick [17]. For patchy polypyrrole coatings, the polystyrene shake-up satellite remains very well detected [18,19].

Taking into consideration the main C1s components, one can use the sum of the fractions of the N–C=O and O–C=O C1s components (abbreviated by C_{NSE}) as a chemical descriptor for monitoring the surface functionalization of polypyrrole-coated PS by the NSE groups. The C_{NSE} fraction (determined by C1s peak fitting, not shown here) increases from 3.5 to 15.7% on deposition of the $PPyNSE_{75}$ at the surface of PS. Although PNVP stabilizer contributes to the component at 288 eV due to its N–C=O pendent group, the increase of C_{NSE} fraction is a clear indication of the incorporation of pyrrole-NSE repeat units in the conducting copolymer overlayer surrounding the PS particles.

3.2. Reactivity of PS-$PPyNSE_{75}$ particles towards model molecules

The reactions of PS-$PPyNSE_{75}$ particles with 2-ethanolamine (R-NH_2, R=OH–CH_2–CH_2–) and 2-mercaptoethanol (R-SH) were conducted in buffered media by incubating a batch of freshly synthesized PS-$PPyNSE_{75}$ particles with R-NH_2 or R-SH for 12 h. The dried products were then characterized by IR and XPS.

Fig. 8 shows XP spectra, in the 160–240 eV regions, of PS-$PPyNSE_{75}$ after addition of various amounts of R-SH. There is a distinct increase in the uptake of the sulfur containing molecules by the polypyrrole carrier as detected by the change in the relative S2p and S2s peak intensities. Because sulfur is a unique elemental marker for R-SH, the S/N atomic ratio determined by XPS can be used to monitor the uptake of these molecules. This is shown in the inset of Fig. 8 where the S/N ratios are plotted against the logarithm of the initial R-SH concentration. One observes a progressive increase of this ratio indicating the immobilization of increasing amounts of R-SH molecules at the particle surface as the concentration of R-SH is higher in the reaction mixture. Unfortunately, there is no such clear change when the R-NH_2 molecule (which does not contain elemental marker) was immobilized on the carrier surface, and we shall present only the IR monitoring of the reaction of this specific molecule with the polypyrrole support (see below).

The IR spectra are shown in Fig. 9 for PS-$PPyNSE_{75}$ after reactions with the model molecules at 10^{-2} M initial concentration. The NSE and pyrrolidone band intensities decrease significantly on addition of either R-NH_2 or R-SH, thus underlying the release of the NSE groups upon reaction of the latex particles with model molecules. Simultaneously, one can notice the appearance of the peaks corresponding to the formation of the interfacial amides (at 1685 cm^{-1}) and thiol ester (at 1709 cm^{-1}), products of the condensation of the latex particles with the model molecules.

3.3. Biorecognition properties of biotin-decorated PS-$PPyNSE_{75}$ particles

The reaction of PS-$PPyNSE_{75}$ particles with Biot-NH_2

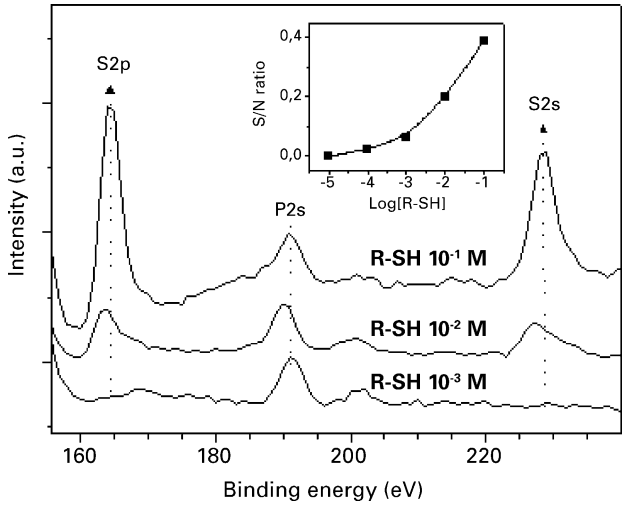

Fig. 8. XP spectra (160–240 eV region) of PS-$PPyNSE_{75}$ after reaction with mercapto ethanol (R-SH) at indicated initial concentration. Inset shows the surface *S/N* atomic ratio plotted against the logarithm of the R-SH concentration.

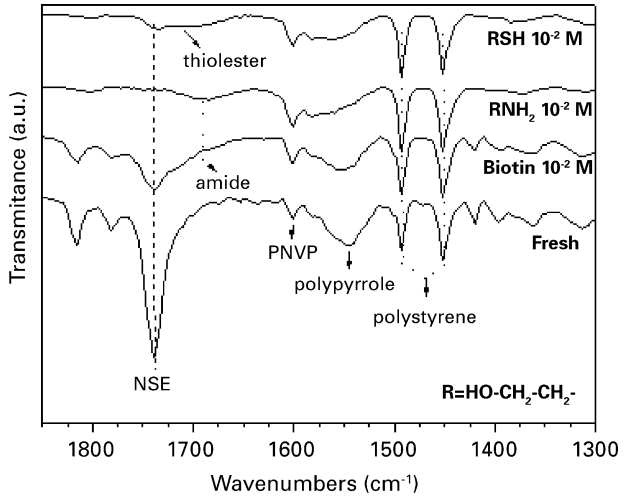

Fig. 9. FTIR spectra (1300–1850 cm^{-1}) of fresh and treated PS-$PPyNSE_{75}$ particles. Note the significant attenuation of the NSE sharp peak as a result of incubation with Biot-NH_2, R-NH_2 and R-SH at indicated concentrations.

brought similar results than those obtained using the model aminated molecules. Indeed, XP spectra of the dried product showed the appearance of S2p and S2s peaks, elemental maker of biotin. The IR spectrum of biotin-decorated PS-PPyNSE$_{75}$ particles, shown in Fig. 9, shows a very weak NSE band at 1739 cm^{-1}, in comparison to that of fresh PS-PPyNSE$_{75}$. This is due to the release of the NSE groups upon reaction. Simultaneously, an amide band appeared therefore indicating the covalent grafting of biotin to the particles.

The biotin-decorated PS-PPyNSE$_{75}$ particles were then evaluated as bioadsorbents of the model protein, avidin. The reaction was conducted in buffered media during 12 h and the particles were then washed by several centrifugation/re-dispersion cycles to remove unreacted avidin. The XP spectra of the dried products showed strong modifications after adsorption in comparison to the spectra obtained prior to the reaction, with most visible changes appearing in the C1s spectra, as shown in Fig. 10. The immobilization of the protein is indicated by two major features: one observes a relative increase of intensity of the shoulder at 286 eV resulting in a broadening of the main peak, and the intensity of the one centered at 288 eV increases also with the avidin concentration in the reaction mixture. These two peaks, which are strongly present in the spectrum of pure avidin, are due to the peptidic residues. The progressive immobilization of avidin on the surface of biotin-decorated PS-PPyNSE particles is better evidenced by plotting the N–C=O C1s fraction for various concentrations of avidin in the reaction mixture, shown in Fig. 11. One observes that the surface chemical composition of the particles gets closer to that of the supported avidin.

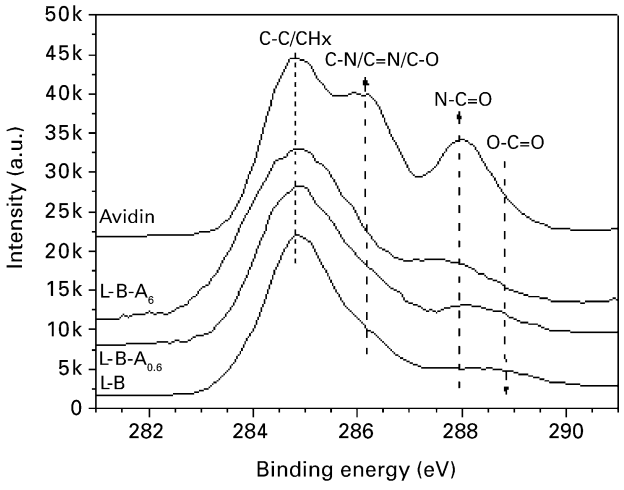

Fig. 10. High resolution C1s spectra of biotin-decorated PS-PPyNSE$_{75}$ (L-B) before and after incubation with avidin (L-B-A$_x$) at indicated concentrations x (in mg/mL), and the control pure avidin powder. Arrows indicate the decreasing or increasing trend of the relative intensity of the specific C1s component.

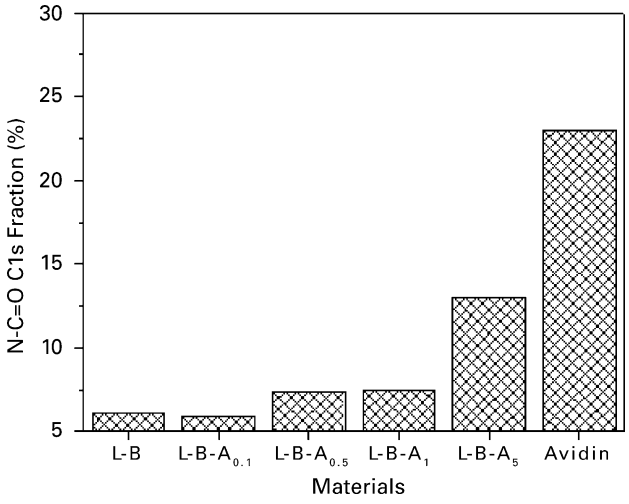

Fig. 11. Fraction (in %) of the N–C=O C1s component determined for biotin-decorated PS-PPyNSE$_{75}$ (L-B) before and after incubation with avidin (L-B-A$_x$), and the control pure avidin powder (A). The initial concentration x of avidin is given in mg/mL.

4. Conclusion

Novel polypyrrole–polystyrene composite particles bearing reactive surface *N*-succinimidyl ester functional groups were prepared in aqueous solution by copolymerization of pyrrole and *N*-esterified pyrrole (pyrroleNSE) using $FeCl_3$ in the presence of polystyrene particles. PS-PPyNSE$_{75}$ particles were characterized in terms of size, morphology and chemical structure. SEM indicated a narrow size distribution of the particles with a diameter of 600 nm. XPS and FTIR spectroscopy confirmed the existence and the chemical stability of the desired ester group at the surface of the colloidal particles. The reactivity of these NSE-functionalized particles was investigated using model molecules bearing amine and thiol groups which can be found in protein residues. Furthermore, the reactive particles proved to be effective for the covalent immobilization of biotin and the recognition of avidin by biotin-decorated particles. The PS-PPyNSE$_{75}$ latexes could thus be used for the covalent attachment of proteins with controlled surface proportion making these supports suitable for biomedical applications.

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