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# Modification of surface energy and electrical and thermal properties of spherical polypyrrole nanoparticles synthesized by CTAB for potential application as a conductive ink

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## Abstract

Polypyrrole (PPy) fine powder was synthesized via a chemical route in the presence of various concentrations of the cationic surfactant cetyltrimethylammonium bromide. The effect of the concentration of the surfactant used on electronic, structural, thermal, and surface properties of the final product was investigated. The sample prepared with the highest concentration of surfactant yielded mostly the best electronic, thermal, and physical characteristic properties, a lot closer to the materials used in conductive inks. The electrical conductivity of the sample underwent an increase of 320%. Fourier transform infrared spectroscopy indicated that all surfactant has been excluded at the final stage of the synthesis, and the results indicated higher doping levels for higher concentrations of surfactant used. Surface energy showed a dramatic increase, and spherical nanoparticles were seen in scanning electron microscopy images. The PPy powder became soluble in polar solvents, becoming highly hydrophilic and indicating good mass retention upon thermal degradation.

**Keywords:** Polypyrrole; CTAB; Conductivity; Surface energy; Spherical nanoparticles; Thermal stability

## Introduction

Conducting polymers have attracted much interest not only within the research community but also within the electronic materials industry. This is due to the advantages that they pose, such as light weight, ease of fabrication, and cost-effectiveness, compared to other conventional conductors and that have been utilized in transparent EMI shielding, inkjet printing, antistatic coating, display devices and electrodes, sensing, information processing, actuation, polymer battery fabrication, and supercapacitors [1-3]. Nevertheless, there are still drawbacks to these materials, namely their insolubility and intractable properties, indicating that modifications are required in order to be able to overcome these drawbacks.

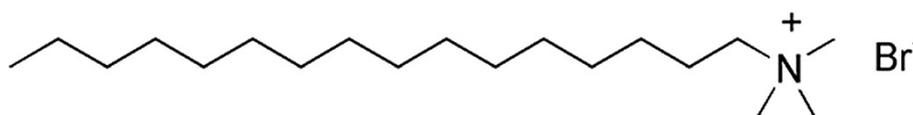
Polypyrrole, among all conducting polymers, is one of the most attractive and has therefore been the subject of

various comprehensive studies as an organic conductor. This is because of its facile chemical synthesis, versatility, high conductivity, low oxidative potential, and good thermal as well as environmental stability [4,5]. PPy has been synthesized in the form of nanoparticles with various shapes in the presence of different types of surfactants and has been utilized in a wide range of optoelectronic devices and applications such as batteries, electrodes, light-emitting diodes, actuators, electromagnetic shielding of circuits, and gas biosensors [1,6,7]. The material used in inkjet printing must possess certain properties, i.e., low viscosity for facile and rapid refilling and high surface energy for preventing the ink from dripping from the nozzle. Surface energies in the range 28 to 350 mJ/m<sup>2</sup> have been reported [8-11]. Surfactants have been employed as emulsifiers in emulsion polymerization, and products of different morphologies with various properties and characteristics have been obtained [12,13]. Furthermore, in order to prevent clogging of the device nozzle by the suspension, only very small particle sizes are allowed; the size of the nanoparticles must be small in respect to the printer nozzle diameter.

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**Figure 1** Chemical structure of CTAB.

While anionic surfactants can also act as counter ions and become incorporated in the backbone of the resulting polymer, cationic surfactants are excluded from the material at the final stage of the synthesis [14,15]. In order to explore the various properties that are imparted upon the synthesis of PPy using varying concentrations of cationic surfactants, in this study, we chose to synthesize polypyrrole using different amounts of cetyltrimethylammonium bromide (CTAB; Figure 1). The effect of the concentration of a cationic surfactant (CTAB) on the surface energy and the electrical and thermal properties of PPy was investigated. At certain concentrations, spherical nanoparticles of PPy formed and the thermal properties of the material were significantly enhanced. This method resulted in profound changes of the electrical properties of PPy. Furthermore, significant changes in the surface energy of PPy were observed, which perhaps hint on the possibility to fine-tune conjugated polymer properties with surfactant concentration. Thus, the material's properties were tuned in a fashion which renders them as better candidates for application in the field of conducting inks.

## Experimental

### Materials and methods

The pyrrole monomer was purchased from Sigma-Aldrich (St. Louis, MO, USA) and was used as received since it was almost colorless, hinting the absence of moisture in the monomer. CTAB, methanol, and ferric chloride were purchased from Merck Chemicals (Darmstadt, Germany). All the reactants were precooled to 0°C before synthesis. FeCl<sub>3</sub> (0.21 mol) was dissolved in 60 ml of methanol; then, CTAB (if any) was dissolved in 40 ml of methanol and was subsequently added to the solution of FeCl<sub>3</sub>. The mixture was stirred magnetically for 30 min, and a homogenous light brown solution yielded. Then, 6 ml of pyrrole was added to the above mixture, and the reaction was allowed to proceed for 4 h. The whole procedure including the reaction was carried out in the atmosphere of nitrogen to avoid any contact with oxygen or moisture as oxygen causes various defects in the backbone of the resulting polymer leading to loss in conductivity [15,16]. The product was then filtered and washed with methanol numerous times until the washings were clear and no oxidant or surfactant remained. The filtrate was subsequently dried at 60°C for 24 h. The reaction conditions were optimized so that the highest conductivity was

achieved. The reaction was carried out at 0°C, the molar ratio of oxidant to monomer was kept at 2.39, and the molar ratio of monomer to surfactant was varied from 20:1 to 3:1 (see Table 1) [15,17,18].

The smallest concentration of surfactant was considerably higher than CMC and attempts were made so as to increase the concentration of CTAB over the widest possible range so that an optimal ratio could be obtained, but it was not physically feasible to further increase the concentration due to limitations of stirring and reactant solubility in methanol.

### Instruments

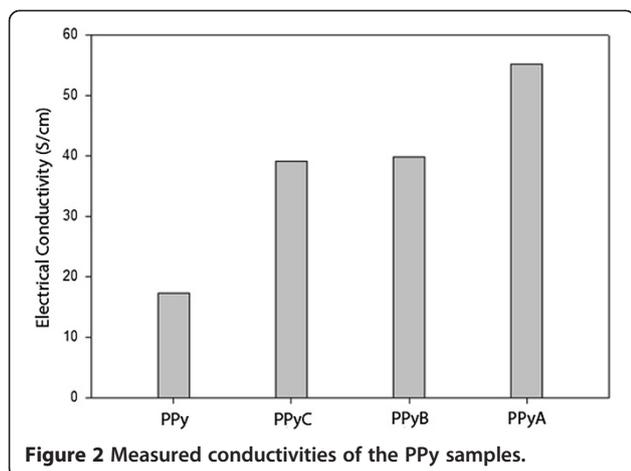
The electrical conductivity measurements of the samples were carried out using a standard four-point probe van Der Pauw setup [19]. Pellets of 1 cm in diameter and 0.15 cm in thickness were prepared by a pellet press using 3 tons of pressure. The probe spacing was kept at 2 mm, and each test was carried out multiple times in different directions at room temperature to make sure that the values were repeatable.

IR spectroscopy was carried out using a Nicolet 6700 FT-IR Spectrometer (Thermo Fisher Scientific, Waltham, MA, USA). The range varied from 400 to 4,040 cm<sup>-1</sup>. Specified amounts of polymer were dispersed in KBr and were subsequently pressed into pellets. UV-vis spectroscopy was performed using a Cary 100 UV-vis spectrometer (Varian, Santa Clara, CA, USA). X-ray diffraction (XRD) measurements were conducted using a Philips X'Pert XRD instrument (Eindhoven, The Netherlands). The field emission scanning electron microscopy (FESEM) images were obtained at 15 kV using a Hitachi SU6600 microscope (Chiyoda-ku, Japan). A uniform layer of gold was deposited on the specimen prior to the test.

Contact angle measurements were carried out using a standard goniometer/tensiometer. Young's equation was then utilized for surface energy calculations. Details of

**Table 1** Reaction concentrations and yields of the PPy samples

|      | PPy/CTAB | [CTAB] | [Py]  | Yield (g) |
|------|----------|--------|-------|-----------|
| PPyD | None     | -      | 0.600 | 1.11      |
| PPyC | 20       | 0.030  | 0.600 | 1.165     |
| PPyB | 7        | 0.085  | 0.600 | 1.466     |
| PPyA | 3        | 0.200  | 0.600 | 0.966     |



this method will be discussed at more length within the later sections. For measurements of the surface energy of the samples, droplets of water, dimethyl sulfoxide (DMSO), and ethylene glycol were dropped on the specimen. For solubility observations, the specimen was first finely ground in a mortar, then sonicated for 30 min in the solvent to obtain good dispersion, subsequently magnetically stirred for several hours, and finally allowed to settle for 48 h so that all unsolved particles would settle down.

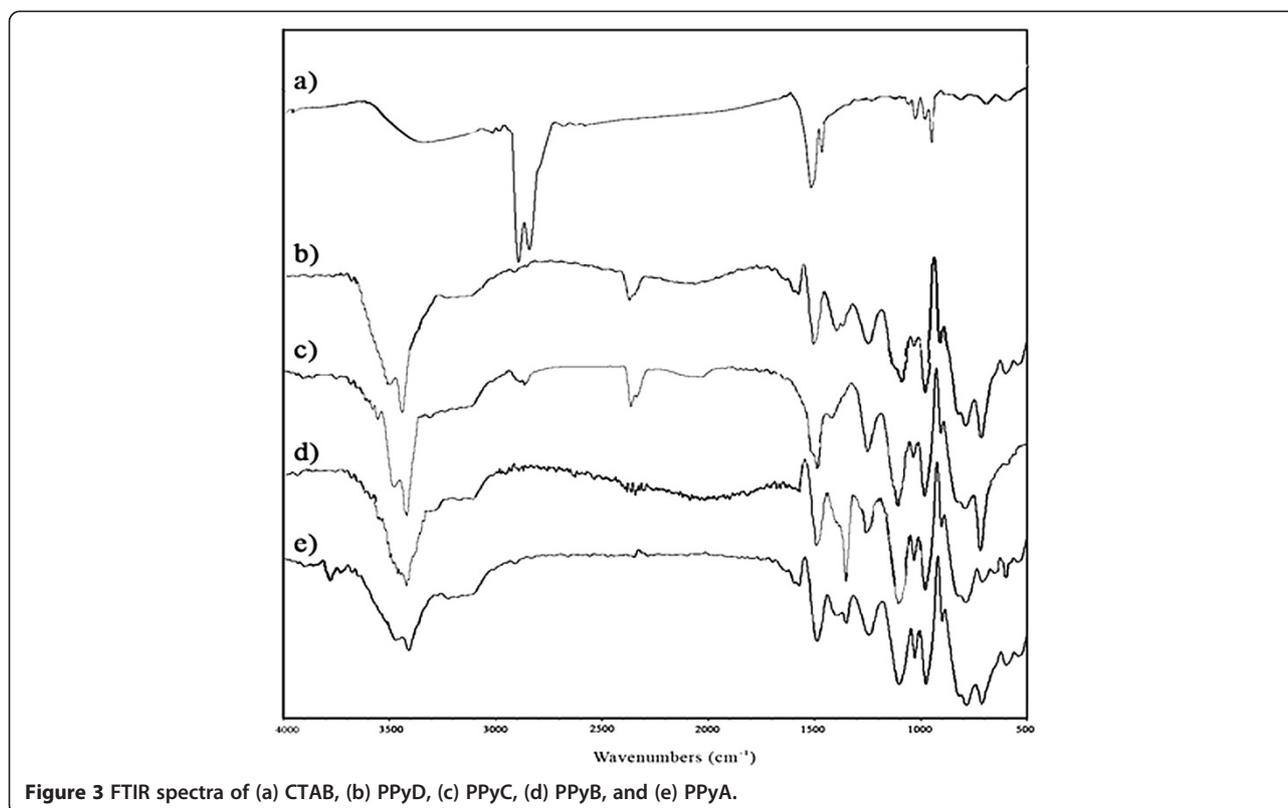
The thermal stability of conductivity and loss of surface energy was tested by keeping the samples at 190°C or

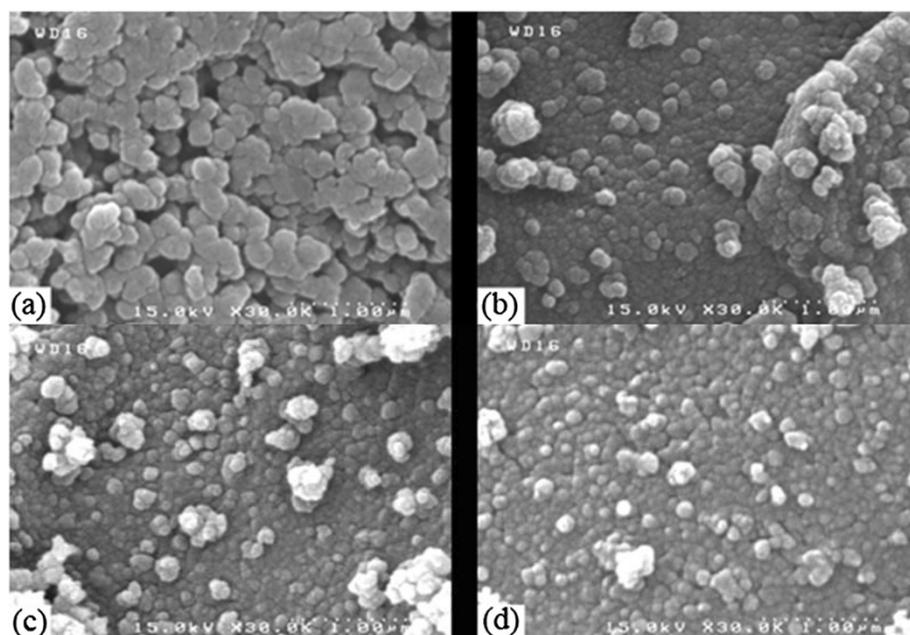
210°C for 24 h. Then, the pellets were examined in terms of their conductivity, mass loss, and surface energy, all of which will be discussed more thoroughly within the later sections.

## Results and discussion

### DC conductivity

The reaction conditions, i.e., temperature, concentration of reactants, time, and oxidation potential, have been optimized so that the highest initial conductivity was obtained [16,20]. The results of the conductivity measurement are illustrated in Figure 2. The sample prepared with no surfactant exhibits a mean conductivity of 17.3 S cm<sup>-1</sup>. After incorporating even a slight amount of surfactant (sample, PPyC), a steep increase in conductivity was seen (126%). This trend, however, was not observed for the higher level of CTAB concentration (PPyB). An increase of 1.75% in conductivity from PPyC to PPyB was obtained. For PPyA, in which the highest concentration of CTAB has been used, a further significant increase was observed; the conductivity of PPyA is 28% higher than that of PPyB, indicating a significant increase of more than threefold in magnitude compared to the conductivity of the plain PPy sample. Fourier transform infrared (FTIR) spectroscopy and XRD analyses support the observed enhancement in conductivity. Moreover, following the addition of CTAB, a higher crystallinity was obtained, which facilitates





**Figure 4** FESEM images of (a) PPyD, (b) PPyC, (c) PPyB, and (d) PPyA samples.

improvements in charge transfer mechanism and, hence, would result in increased conductivity [21,22]. Also, less cross-links in PPyA means less destruction of the electronic structure as the formation of cross-links requires breakage and reduction of a double bond to a single bond, thereby decreasing its  $\pi$ - $\pi$  conjugation length which brings about charge localization on the backbone, therefore leading to a reduction in conductivity. Also, cross-links are known to act as electron trap sites, serving as defects in the backbone disturbing mobility and the electronic structure.

The FTIR spectra of the polypyrrole samples prepared with different amounts of CTAB are illustrated in Figure 3. The spectra exhibit all of the characteristic peaks of polypyrrole such as symmetric and asymmetric stretching vibrations of the heterocyclic ring at about 1,537 and 1,448  $\text{cm}^{-1}$ , respectively, N-H stretching vibration at approximately 3,420  $\text{cm}^{-1}$ , N-H out-of-plane vibration at 770  $\text{cm}^{-1}$ , and the other significant peaks located at 1,036 and 1,160  $\text{cm}^{-1}$ , respectively. From the spectra, it is obvious that no characteristic peak of CTAB is seen; therefore, no residual surfactant remains within the samples. Since the reaction media was carefully controlled in order to exclude any water or oxygen, only tiny peaks at 1,702 and 1,635  $\text{cm}^{-1}$  which are attributed, respectively, to carbonyl defects and moisture are seen. We believe that this is due to the residual water in KBr and the oxygen uptake of pyrrole [23].

#### FTIR

By comparing the FTIR spectra of CTAB and the samples, it is obvious that no characteristic peaks of CTAB

are seen in the final polymer, indicating that it has been completely washed out. Although it is seen that the cationic surfactants did not remain in the final structure, there is some evidence that the chemical structure of the samples prepared in the presence of CTAB has been subjected to profound changes. The increase in conductivity can be attributed to the  $\pi$ -electron delocalization which causes a slight shift in the position of the peaks located at 3,418, 1,537, and 1,160  $\text{cm}^{-1}$ , to undergo a red shift to 3,415, 1,536, and 1,159  $\text{cm}^{-1}$ , respectively. The peak at 1,088  $\text{cm}^{-1}$  which is assigned to the in-plane deformation vibration of  $\text{N}^+\text{H}_2$  exhibits a slight increase which hints higher degrees of protonation of the polypyrrole backbone [14,18,21,24].

#### Morphology

The scanning electron microscopy (SEM) images of the samples prepared at different concentrations of CTAB are illustrated in Figure 4. The samples prepared with higher amounts of surfactant exhibit less compact morphology and they seem more regular, whereas the sample prepared without the surfactant exhibits a very dense and compact structure. As the concentration of the incorporated CTAB

**Table 2** Physical characteristics of solvents for substitution into Equation 1

| Solvent         | $\gamma$ (mJ/m <sup>2</sup> ) | $\gamma^f$ (mJ/m <sup>2</sup> ) | $\gamma^+$ (mJ/m <sup>2</sup> ) | $\gamma^-$ (mJ/m <sup>2</sup> ) |
|-----------------|-------------------------------|---------------------------------|---------------------------------|---------------------------------|
| Water           | 72.6                          | 21.6                            | 25.5                            | 25.5                            |
| Ethylene glycol | 48.3                          | 29.3                            | 1.9                             | 47.0                            |
| DMSO            | 44.0                          | 36.0                            | 0.5                             | 32.0                            |

**Table 3 Contact angle measurements and surface energy calculations of PPy samples**

|      | Water (deg) | Glycol (deg) | DMSO (deg) | Surface energy (mJ/m <sup>2</sup> ) |
|------|-------------|--------------|------------|-------------------------------------|
| PPyD | 68          | 30.4         | 20.83      | 36.18                               |
| PPyC | 62          | 39.47        | 23.35      | 42.27                               |
| PPyB | 46.54       | 42           | 21.80      | 52.22                               |
| PPyA | 37          | 41           | 20.55      | 60.78                               |

increases, less agglomeration and better dispersion are obtained. Moreover, although PPyA and PPyB are roughly similar in morphology, for PPyA, one can see regular spherical nanoparticles of smaller mean size diameter as compared to PPyB nanoparticles (see Figure 4). In some regions, PPyA samples with diameters as small as 80 nm are observed; this particle size is more conventional in inkjet printing [10,11]. The packing of the nanoparticles can be attributed to the agglomeration of the particles due to the removal of the surfactant at the final stage of the synthesis by rinsing with methanol [24].

**Contact angle measurements, surface energy, and solubility**

The interaction between the forces of cohesion and the forces of adhesion determines whether or not wetting occurs. If complete wetting does not occur, then a bead of liquid will form, with a contact angle which is a function of the surface energy of the substrate [9,25,26]. There are multiple ways of measuring the surface energy of a substance; a rather facile way is to measure the values of the contact angle that the substrate makes with certain solvents, and then by utilizing Young's equation (Equation 2), the surface energy would be determined. The physical character values of the solvents, Equation 1, are given in Table 2.

$$(1 + \cos\theta)\gamma_L = 2\left(\sqrt{\gamma_s^f \times \gamma_L^f} + \sqrt{\gamma_s^+ \times \gamma_L^-} + \sqrt{\gamma_s^- \times \gamma_L^+}\right), \tag{1}$$

where  $\theta$  is the contact angle produced by the liquid on the solid surface;  $\gamma_L$ ,  $\gamma_L^f$ ,  $\gamma_L^-$ , and  $\gamma_L^+$  are given in Table 2. The equation above is applied to each solvent, respectively,

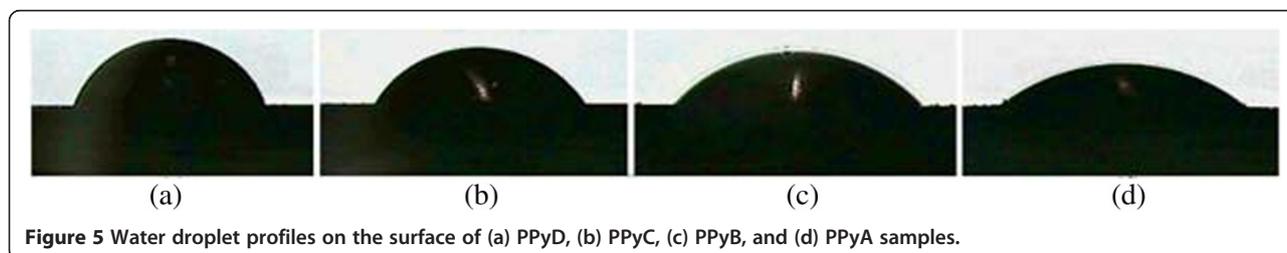
using the values of  $\gamma_s^f$ ,  $\gamma_s^+$ , and  $\gamma_s^-$  for each solvent, given within the table.

$$\begin{aligned} \gamma_s &= \gamma_s^f + \gamma_s^{AB} \\ \gamma_s^{AB} &= 2\sqrt{\gamma_s^+ \times \gamma_s^-} \end{aligned} \tag{2}$$

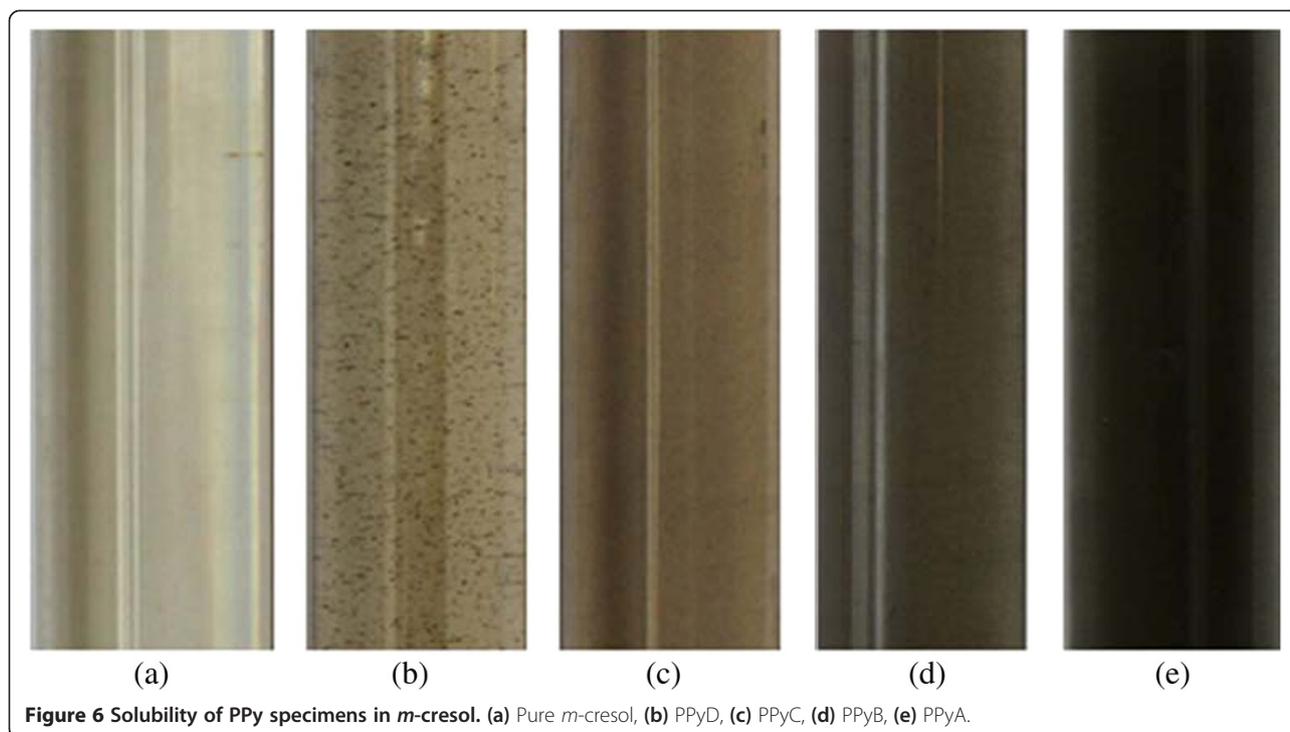
In Equation 2,  $\gamma^f$  is the apolar energy component; the other two are the electron acceptor energy ( $\gamma^+$ ) and the electron deliver energy ( $\gamma^-$ ) [27].

The values of the surface energy for different samples are given in Table 3. The surface energy of the polymer samples increases with the concentration of surfactant used during the synthesis. This may be attributed to the size reduction of the particles and, hence, the relative accumulation of the material on the surface, leading to an increase in the surface energy of the PPyA sample. A gradual increase in hydrophobicity was observed as the concentration of the incorporated CTAB increased (see Table 3). It is also worth mentioning that the samples prepared using high concentrations of CTAB show a higher affinity for polar solvents. The trend of the increase of hydrophobicity is displayed in Figure 5. The solubility of the specimens was tested in various solvents. As it is generally believed, PPy is a highly intractable polymer due to its low solubility and processing. Based on Figure 6, as the concentration of the surfactant increases, a significant increase in solubility occurs and the corresponding solution of the polypyrrole in *m*-cresol appears darker, whereas the color of the solution containing the plain PPy shows almost no sign of the polymer and its color is the characteristic color of *m*-cresol. In addition, the samples prepared with high amounts of surfactant, i.e., PPyA and PPyB, were also soluble in solvents such as methanol, water, and ethanol. Lower levels of cross-linking can be the cause of this effect. Moreover, since PPyA is the most hygroscopic of all samples, one can infer that it contains less cross-link. CTAB micelles serving as tiny reaction sites prevent chains of separate micelles to fuse, thus decreasing the probability of the occurrence of cross-links [20]. In addition, following the increase in surface energy, solubility is enhanced since the resulting material is a better match to the solvent in terms of surface energy.

Solution casting was also made possible for surfactant-containing samples. A solution of the polymer is prepared,



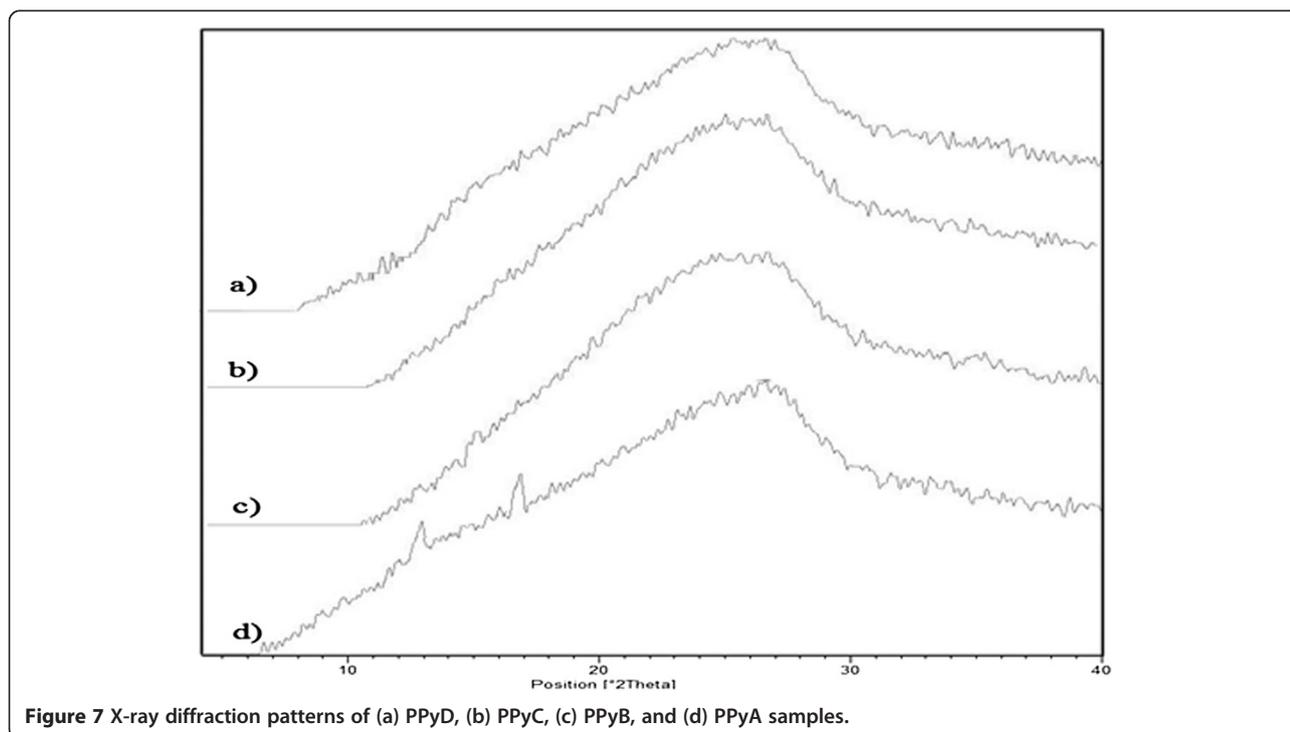
**Figure 5 Water droplet profiles on the surface of (a) PPyD, (b) PPyC, (c) PPyB, and (d) PPyA samples.**



and then either drops of the polymer solution were poured on a glass substrate or the lamellae themselves were dipped in the polymer solution. The solvent is then evaporated and a very thin but uniformly dark layer of polymer is formed on the glass substrate.

#### Crystallography

The X-ray diffraction patterns of the samples are illustrated in Figure 7. All spectra seem to follow a specific pattern, and no significant deviation can be detected for any sample. The characteristic amorphous scattering peak of PPy is seen



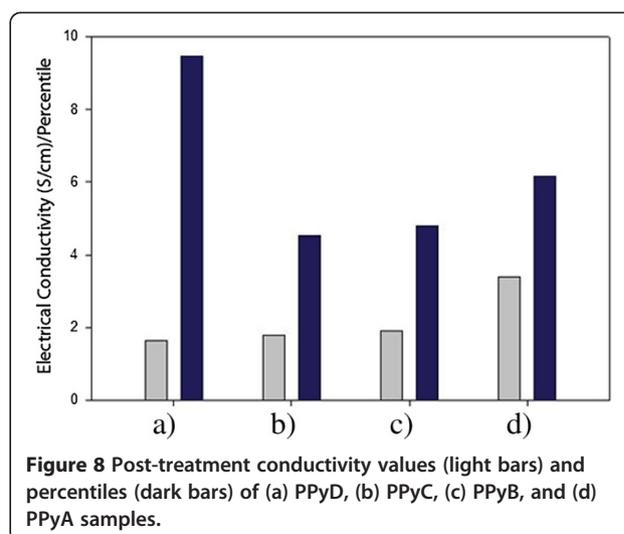
at  $2\theta \cong 5$  to 30; this peak undergoes no change for any sample. The spectrum of PPyA displays two perfectly outstanding peaks at  $2\theta = 12.87$  and  $2\theta = 16.88$ . We attribute the peak at  $2\theta = 16.88$  and the peak at  $2\theta = 12.87$  to polymer crystallite structures. As it is vividly clear, the intensity of the spectrum for PPyA has increased in comparison, which hints increased crystallinity [13,24]. For high-surfactant samples, the presence of crystallinity, meaning ordered polymer structures, enhances the overlay of chains. This results in lowered bandgap and facilitates interchain charge hops, increasing conductivity. Therefore, the overall structure transition from amorphous to crystalline signals a rise in the charge transfer mechanism, bringing about the large increase in the conductivity obtained.

### Ageing and thermal analysis

Testing the material's behavior after thermal treatment was not readily done, since all samples were almost completely stable thermally and all kept 94% of their original weight after heating at 90°C, 120°C, 150°C, and 170°C each for 6 h (first run). Also, at temperatures above 210°C, some samples underwent combustion; thus, it was not feasible to observe the changes in the material's weight in a wide temperature range. As a result, in order to test material's behavior, a specific temperature needed to be set while expanding heating time. Thus, two sets of unaged samples were selected and were kept at 190°C (second run) and 210°C (third run), each for 24 h to observe mass loss [28] (Table 4).

The results indicate that after the second run, the sample prepared in the highest concentration of CTAB (PPyA) retains most of its weight. PPy prepared with no surfactant loses up to 30% of its mass while PPyA loses only 10% of its initial mass. However, in the third run, incorporation of the surfactant invariably results in the decrease of mass retention; nevertheless, PPyA still shows better properties compared to PPyB and PPyC. The percentage of degradation of PPyB and PPyA was roughly the same, so as the concentration of CTAB gradually increases, mass loss plateaus indicate that further increase in the concentration of CTAB would not bring about further increase or improvements of the properties. However, PPyA was the most brittle in comparison, and the pellets prepared from it were observed to be relatively more fragile.

The conductivity values after thermal treatment are illustrated in Figure 8. As compared to plain PPy, the



polymers prepared in the presence of the surfactant will face stronger decreases in the stability of conductivity; however, the sample prepared with the highest concentration of CTAB (PPyA) shows the lowest decay in conductivity among all the surfactant-assisted synthesized samples, and it also possesses the highest final value of conductivity after thermal operations. The reduction in the thermal stability of PPyA relative to PPyD may be attributed to the reduction of cross-links in the structure of polypyrrole, thereby exacerbating thermal degradation. However, although larger portions of conductivity were lost compared to plain PPy, the samples prepared with the surfactant all showed higher final values of conductivity in comparison, 1.77 S cm<sup>-1</sup> for PPyC as compared to 1.635 S cm<sup>-1</sup> for PPyD. After thermal treatment, the surface energy and hydrophilicity of the samples changed dramatically, but they did not follow a sensible trend (see Table 5). However, all samples prepared with the surfactant showed a decrease in surface energy, but the plain polymer showed an increase which may be attributed to the activation of surface groups due to thermal treatment [20]. After heat treatment, among all samples, PPyC has the highest affinity for water and it maintains almost 95% of its original surface energy. However, the values of surface energy show that surface energy first decreases with the increase in content of the

**Table 4 Percentile of mass retained after thermal ageing**

|      | First set (%) | Second set (%) | Third set (%) |
|------|---------------|----------------|---------------|
| PPyD | 96.42         | 77.11          | 75.91         |
| PPyC | 93.57         | 89.14          | 61.49         |
| PPyB | 94.20         | 90.20          | 66.29         |
| PPyA | 93.42         | 91.55          | 72.99         |

**Table 5 Post-treatment contact angle measurements and surface energy calculations of the PPy samples**

|      | Water (deg) | Glycol (deg) | DMSO (deg) | Surface energy (mJ/m <sup>2</sup> ) |
|------|-------------|--------------|------------|-------------------------------------|
| PPyD | 48.7        | 23.7         | 11.3       | 43.4                                |
| PPyC | 46.0        | 39.5         | 22.1       | 40.2                                |
| PPyB | 50.8        | 31.0         | 12.0       | 25.5                                |
| PPyA | 50.0        | 34.0         | 19.1       | 41.7                                |

surfactant incorporated during the synthesis and then rises back up slightly [28].

## Conclusions

Three different concentrations of CTAB were used for the synthesis of polypyrrole. Considering that CTAB is a cationic surfactant and referring to the FTIR data, it was eminent that all CTAB has been completely excluded from the polymer material. Further, FTIR, XRD, and SEM analyses prove that profound changes in the properties and, hence, the structure of PPy have occurred. Solubility is a crucial factor for inkjet printing, and the polymer is now soluble in many common solvents such as methanol which allows making PPy solutions with various weight percentages. The surface energy of the material enhanced and was within the range suitable for inkjet printing. The conductivity of PPy increased by more than threefold, from  $17.30 \text{ S cm}^{-1}$  for plain PPy to  $55.23 \text{ S cm}^{-1}$  for PPyA, which is a dramatic increase for a cationic surfactant. A pure, highly conductive organic material which is solvent processible was yielded. Weight retention upon thermal degradation increased with the increase of the concentration of the surfactant used, and while the final value of post-treatment conductivity for PPyA was the highest among all samples, the ratio of post-treatment conductivity to initial conductivity for plain PPy was the highest. As the concentration of CTAB used increased, less agglomeration and better dispersion resulted, and for PPyA, sphere-like nanoparticles of PPy were observed. With the increase of the surfactant concentration, the size of the nanoparticles reduced, thus reducing the probability of nozzle clogging upon application. Considering the changes and the enhancements that have taken place, the resulting material poses a suitable material for application in inkjet printing and optoelectronics [8].

## Competing interests

The authors declare that they have no competing interests.

## Authors' contributions

The current study was carried out by KV under the supervision of YSJ. Both authors read and approved the final manuscript.

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