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Influence of Needle-punching Parameters for the Preparation of Polypyrrole-coated Non-woven Composites for Heat Generation

Vpliv parametrov iglanja na izdelavo s polipirolom prevlečenih kompozitnih vlaknovin za generiranje toplote

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Abstract

This work deals with the preparation and characterization of electrically conductive needle-punched non-woven composites for heat generation. Electro-conductive non-woven composites were prepared through the in situ chemical polymerization of pyrrole with $FeCl_3$ (oxidant) and p-toluene sulfonic acid (dopant). A two-stage double-bath process was adopted for the in situ chemical polymerization of pyrrole. The effect of parameters such as fibre fineness, needle-punching density and depth of needle punching on a polypyrrole add-on, and surface resistivity were studied by employing the Box-Behnken response surface design. It was observed that fibre fineness was the most influential parameter of the polypyrrole add-on. The lowest surface resistivity of the polypyrrole coated sample (200 g/m², prepared with a punch density of 200 punch/cm², a punching depth of 6 mm and fibre fineness of 2.78 dtex) was found to be 9.32 kΩ/□ with a polypyrrole add-on of 47.93%. This non-woven composite demonstrated good electrical conductivity and exhibited Joule's effect of heat generation. Due to the application of a 30 V DC power supply, the surface temperature of the non-woven composite rose to 55 °C from a room temperature of 37 °C. Optical and electron microscopy images of the non-woven composites showed that PPy molecules formed a uniform coating on the non-woven surface. FTIR studies evidenced the coating of PPy on a polyester surface. These coated non-woven composites were highly electrically conductive and practically useful for the fabrication of heating pads for therapeutic use.
Keywords: non-woven, composite, surface resistivity, electroconductive textile, needle-punching, fibrous material, coated fabric

Izvleček

V delu je predstavljena priprava in karakterizacija elektroprevodnih iglanih kompozitnih vlaknovin za generiranje toplote, ki so bile izdelane in situ s kemično polimerizacijo pirola z oksidantom $FeCl_3$ in dopantom p-toluensulfonsko kislino. Uporabljen je bil dvostopenjski dvokopelni postopek. Vplivi parametrov, kot so finoča vlaken in gostota ter globina iglanja, na dodani polipirol in površinsko upornost so bili proučevani glede na odzivno površino, zasnovani z metodo Box-Behnken. Ugotovljeno je bilo, da na dodatek polipirola najbolj vpliva finoča vlaken. Najnižja površinska upornost 9,32 kΩ/□ je bila dosežena na vzorcu s 47,93-odstotnim nanosom polipirola na vlaknovini površinske mase

200 g/m², gostote iglanja 200 vbodov/cm², globine iglanja 6 mm in finoče vlaken 2,78 dtex. Ta kompozitna vlaknovina je imela dobro električno prevodnost in je izkazovala Joulov učinek generiranja toplote. Zaradi uporabe enosmernega, 30-voltnega, napajanja se je temperatura površine kompozitne vlaknovine zvišala s sobne temperature, tj. 37 °C, na 55 °C. Na posnetkih kompozitnih vlaknovin s pomočjo optičnega in elektronskega mikroskopa je vidno, da so molekule polipirola tvorile enakomerno prevleko na površini vlaknovine. Študije FTIR so potrdile prisotnost polipirola na površini poliestrskih vlaken. Te prevlečene kompozitne vlaknovine so bile električno visokoprevodne in primerne za izdelavo grelnih blazinic za terapevtsko uporabo.

Ključne besede: netkane tekstilije, kompozit, površinska upornost, elektroprevodna tekstilija, iglanje, vlaknati material, premazana tekstilija

1 Introduction

The use of metal wires or metal coating is a common practice for imparting electrical conductivity to textiles [1–3]. Also, metal fibres were used during staple spinning to manufacture electro-conductive yarns [4–5]. Those yarns were used in weaving or knitting to produce electro-conductive fabrics. However, the processing of those yarns was difficult and they lose their textile properties [6]. Many such limitations associated with processability, low mechanical strength and poor flexibility could be successfully overcome by coating/applying conducting polymers on textile substrates [7–9]. PPy has been used primarily as a conductive polymer due to its high conductivity, low toxicity [10], commercial availability and high stability in the air relative to other conducting polymers [11–13].

The heating effect of PPy-coated polyethylene terephthalate-lycra woven fabrics was studied by Kaynak & Håkansson [14]. Those coated fabrics exhibited reasonable electrical conductivity and effective heat generation. At an applied voltage of 24 V, the maximum temperature achieved was 40.55 °C. In another study, PPy was incorporated into cotton woven fabrics and various properties such as anti-static, anti-microbial, heat generation were investigated [15]. A conducting textile composite was prepared by embedding PPy in natural and manmade cellulosic fibres, such as cotton, viscose, cupro and lyocell, using in situ vapor phase polymerization [16]. Their various electrical properties such as voltage-current characteristics, voltage-temperature characteristics, etc., were also studied [17]. It was suggested by Sparavigna et al. and Macasaquit & Binag that 100% polyester fabrics could easily be made electrically conductive using a PPy coating, while they were practically useful for many applications, including flexible, portable surface-heating elements for medical or other applications [17–18]. An electrical resistor was formed within a fabric by sewing a highly conductive silver-coated yarn into less conductive PPy-treated knitted fabric [19]. This was found to be a useful method for enabling electrical connection to a fabric for the characterization of a fabric's resistivity and the design of a fabric resistor. In another study, it was reported that fabrics knitted with silver yarn, together with elastomeric yarn, could generate sufficient heat to warm the body. Those fabrics could be used to manufacture personal heating garments that could generate heat in relation to applied voltage [20]. In another study, when a constant voltage of 9 V was applied to cotton/PPy fabric for 10 minutes, its surface temperature rose to 90 °C, while the performance of the fabric did not deteriorate after numerous repetitive cycles [15]. Similar observations were also made by Maity et al. in the case of PPy-coated polyester-woven, spunlace and needle-punched non-woven composite fabrics prepared using in situ chemical polymerization [8]. PPy-coated polyester-woven, spunlaced and needle-punched non-woven composite fabrics demonstrated an exponential rise in surface temperature when voltage was applied, while the rise in temperature was found to be related to the duration of voltage application [8]. Among these three types of fabric structures, the needle-punched non-woven composite performed better in terms of good electrical conduction and heat generation. This was attributed to its porous and bulky structure. In another study, various polyester needle-punched non-woven composites were prepared and PPy applied to them using in situ chemical polymerization. It was shown that a porous and bulky non-woven structure facilitated the better penetration of a reagent liquor between fibre interstices with a more extensive polymerization yielding a higher polymer add-on [12, 21]. The highest PPy add-on of 16.28% was achieved for a polyester needle-punched non-woven composite (250 g/m², 200 punches/cm², 8 mm depth of penetration and 6.66 dtex fibre) [12]. These PPy-coated non-woven

composites were evaluated for electro-magnetic shielding application, but heat generation behaviour was not studied.

Most of the studies available in literature concern woven and knitted fabric composites that are relatively thin compared with needle-punched non-woven composites. There are limited works available regarding PPy-coated needle-punched structures and the evaluation of the same in terms of Joule's effect of heat generation. Fibre fineness and the process parameters of needle-punching affect the structural features of the manufactured non-woven composites. Thus the aim of the present work was to investigate the effect of fibre fineness, needle-punching intensity and needle punching depth on a PPy add-on, and the surface resistivity of manufactured non-woven composites by employing a Box-Behnken response surface design and the characterization of the same

in terms of Joule's effect of heat generation. Polyester needle-punched non-woven composites are currently used in many applications, such as home furnishing, upholstery, filtration, filler materials for winter garments, geotextiles, etc., while this functional modification of the same may open up new dimensions of its application.

2   Materials and method

2.1 *Preparation of polyester needle-punched non-woven composites using a 3^3 Box-Behnken design*

Polyester fibres were used as the raw material in the preparation of needle-punched non-woven composites on a "Dilo" needle-punching machine. The virgin polyester fibres of three different finenesses (3.33 dtex,

Table 1: Box-Behnken design parameters of the experiment and the levels thereof

Level	Punch density (punch/cm²)	Punch depth (mm)	Fibre fineness (dtex)
	A	B	C
−1	150	6	3.33
0	200	9	6.66
1	250	12	9.99

Table 2: Scheme of experiments according to the 3^3 Box-Behnken Design with five central repeat runs

Run	A: punch density (punch/cm²)	B: punch depth (mm)	C: fibre fineness (dtex)
1	0	0	0
2	1	0	1
3	0	0	0
4	0	0	0
5	0	1	−1
6	−1	0	1
7	−1	1	0
8	1	0	−1
9	0	1	1
10	1	−1	0
11	0	0	0
12	−1	0	−1
13	0	−1	1
14	0	−1	−1
15	−1	−1	0
16	1	1	0
17	0	0	0

6.66 dtex and 9.99 dtex) and a cut length of 51 mm were procured from Reliance Industries Ltd., India. A three-factor, three-level (3^3) Box-Behnken response surface design was employed for an experimental plan to prepare needle-punched non-woven samples. The parameters were fibre fineness, punch density and punch depth, the value of which are shown in Table 1. The 3^3 Box-Behnken design has only 17 experimental runs in comparison to 27 runs for the full factorial design. In this Box-Behnken design, the treatment combinations are at the midpoints of the edges of the process space and at the centre, and thus do not consider extreme treatment conditions that result in the increased consumption of energy and chemicals, and may also have a harsh effect on the substrate of non-woven composites. Thus, according to the design, 17 varieties of needle-punched non-woven samples, each of the same mass per unit area (200 g/m²), were prepared, as shown in Table 2. All these samples were coated with PPy and tested for thickness, porosity, PPy add-on, and surface resistivity.

2.2 Chemicals
The chemicals used were ferric chloride ($FeCl_3$) as the oxidant, pyrrole as the monomer and p-Toluenesulfonic acid (PTSA) as the dopant. All of the chemicals used were of laboratory grade and purchased from Sigma Aldrich, India.

2.3 Method of preparation of PPy-coated non-woven composites
Polyester needle-punched, non-woven samples were hydrolysed for the better fixation of PPy on a polyester surface. It was reported that the hydrolysis of polyester fabrics with 10% sodium hydroxide (NaOH) solution for 20 minutes at boiling (95 °C) at a 1:40 material to liquor ratio was the optimum condition of hydrolysis for achieving maximum add-on without significant loss of fabric strength [8]. Therefore, all polyester non-woven composites were hydrolysed employing the same process conditions.

After hydrolysis, the non-woven composites were neutralized with 0.1 N acetic acid solutions. A two-stage double-bath process was then employed for the in situ chemical polymerization of pyrrole. In the first stage, the non-woven fabric was soaked with the monomer in a monomer bath. In the second stage, the in situ polymerization of pyrrole was conducted in an oxidant bath. A monomer bath was prepared by dissolving pyrrole in 0.5 M concentration in de-ionized water. Material to liquor ratio of monomer bath was 1:40. An oxidant bath was prepared by dissolving $FeCl_3$ and PTSA in de-ionized water. The amount of FeCl3 and PTSA used were 0.5 M and 0.05 M, respectively. Material to liquor ratio was 1:40. Hydrolysed samples were first allowed to soak in a monomer bath for 15 minutes at room temperature. The oxidant bath was cooled to 5 °C in a cryostat and pyrrole socked non-woven samples were taken from monomer bath and dipped into the oxidant bath for in situ polymerization. The time of the in situ polymerization was 1 hour. Polymerized samples were then taken out from the oxidant bath, thoroughly rinsed with cold water and dried in an oven at 80 °C for 4 hours before measurement.

2.4 Measurement of surface resistivity
The surface resistivity of the PPy-coated non-woven composites was measured inside a control chamber at 0% relative humidity using a concentric ring probe electrode according to the test procedure described in ASTM D-257 standard [22]. The concentric ring probe electrode was fabricated according to the aforementioned standard with 12.5 mm outer radius of the inner ring electrode and 32 mm inner radius of the outer ring electrode, as shown in Figure 2. Surface resistivity measured from such an electrode probe can be expressed using equation 1.

$$\rho = R_s \times \frac{2\pi}{\ln \left(\frac{R_2}{R_1} \right)} = R_s \times K \qquad (1)$$
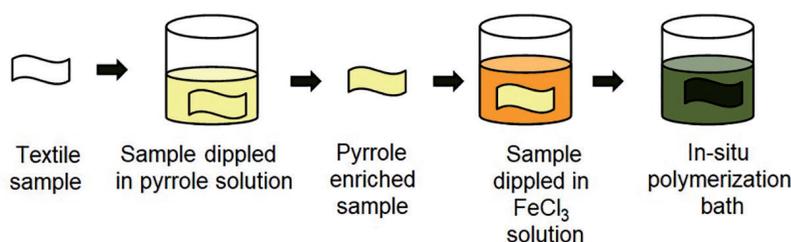

Figure 1: In situ chemical polymerization of pyrrole onto polyester non-woven composites using the double-bath process [13]

where, ρ represents surface resistivity (Ω), R_s represents the resistance of the sample (Ω), R_1 represents the outer radius of the inner ring (mm), R_2 represents the inner radius of the outer ring (mm) and K represents a geometric constant.
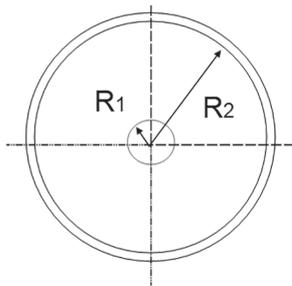

Figure 2: Schematic diagram of concentric ring electrode probe

2.5 Measurement of electrical characteristics

The higher the electrical conductivity of non-woven composites, the higher the Joule's effect of heat generation will be because generated heat $H \propto I^2$, where I represents the current flowing through the conductor. Thus, the measurement of voltage-current (V-I) characteristics will help to predict the heat generation behaviour of the non-woven composites. For this measurement, composite specimens were cut into 15 cm ×12.5 cm pieces. Two copper plates measuring 12.5 cm × 2.5 cm were placed on the two opposite edges of the specimen as electrodes. The length of the electrodes (D) was 12.5 cm, as shown in Figure 2. The distance between the two electrodes (L) was maintained at 10 cm. Electrode plates were then connected with a variable DC power supply (U), as shown in Figure 3. Due to the applied DC voltage across the samples, current flowed in the circuit that was measured by connecting an ammeter, while the surface temperature was measured by a non-contact type infra-red thermometer.
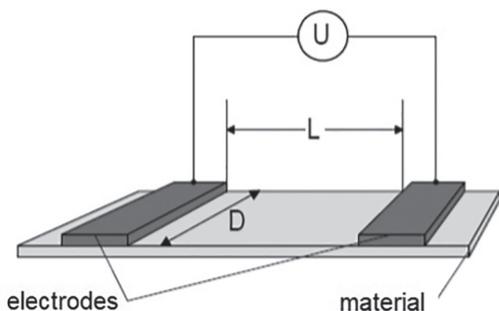

Figure 3: Parallel plate method to measure electrical characteristics of non-woven composites

2.6 Optical microscope analysis

Morphological analysis of uncoated and PPy-coated composites was carried out using an optical microscope (Nikon Eclipse E200) at a magnification of 10×.

2.7 Analysis of samples using FTIR

FTIR analysis of untreated uncoated and PPy-coated composites was carried out using a Bruker 10074570 spectrometer. Spectra were recorded in the range of 4,000 cm^{-1} to 600 cm^{-1} by accumulating 24 scans at a resolution of 4 cm^{-1}.

3 Result and discussion

3.1 PPy add-on and surface resistivity of electro-conductive composites

According to the Box-Behnken design, 17 experimental runs were performed to prepare 17 varieties of PPy-coated non-woven composite samples. Thirty observations were made for all these PPy-coated composite samples for the purpose of measuring PPy add-on and surface resistivity. Average values are shown in Table 3. It is evident that the highest PPy add-on of about 47.93% was achieved in the case of sample no. 14. The surface resistivity of this sample was found to be the lowest among all samples (9.32 kΩ/□). In a previously reported study, PPy add-on of 16.28% was achieved for polyester needle-punched non-woven composite (250 g/m^2, 200 punches/cm^2, punching depth of 8mm and 6.66 dtex fibre) [12]. Better PPy add-on may be achieved due to alkali hydrolysis and the use of finer fibres for needle-punching [7].

3.2 Effect of the process parameters on surface resistivity of PPy-coated composites

The effect of the process parameters of needle punching, such as punch density, punching depth and fibre fineness, on the surface resistivity of coated composites was investigated by employing the Box-Behnken design and using a response surface methodology. The main effects and interaction effects of these parameters were analysed by employing analysis of variance (ANOVA), as shown in Table 4. The "Model F-value" of 4.15 implies model significance relative to noise. There is only a 3.00 % chance that a "Model F-value" this large could occur due to noise. Values of "Prob > F" of less than 0.05 indicate model terms are significant. The lowest p-value (0.0812) is observed for punch

Table 3: Surface resistivity and PPy add-on of PPy-coated needle-punched non-woven composites

Run	Factor A: punch density (punch/cm²)	Factor B: punching depth (mm)	Factor C: fibre fineness (dtex)	Average surface resistivity (kΩ/□)	Average PPy add-on (%)
1	0	0	0	406	14.2
2	1	0	1	384	13.3
3	0	0	0	368	14.2
4	0	0	0	286	14.2
5	0	1	−1	38	15.2
6	−1	0	1	479	18.0
7	−1	1	0	702	15.7
8	1	0	−1	155	17.4
9	0	1	1	364	8.9
10	1	−1	0	713	19.2
11	0	0	0	358	14.2
12	−1	0	−1	998	16.2
13	0	−1	1	171	13.7
14	0	−1	−1	9	47.9
15	−1	−1	0	269	7.3
16	1	1	0	316	8.0
17	0	0	0	296	13.0

density(A), which is the most influential parameter of all. Among interaction effects, AB interaction was significant in that it indicated their combined effect during the needle-punching process. The "lack of fit F-value" of 17.80 implies that lack of fit is significant. There is only a 0.82% chance that a "lack of fit F-value" this large could occur due to noise. The coefficient of determination (R^2) is found to be 0.8060, which implies that the fit of the model is sufficient for predicting the response. The model equation in the coded unit is shown in equation 2.

$$\rho = 342.91 - 109.70A + 19.70B + 12.16C + 270.27A^2 \quad (2)$$
$$-113.17B^2 - 109.18C^2 - 207.50AB + 187.08AC$$

This equation is good at predicting the theoretical surface resistivity of PPy-coated composites with known process parameters.

Table 4: Significance of PPy-coated needle-punched composite samples for the surface resistivity

Source	Sum of squares	DF	Mean square	F-value	Prob > F	
Model	8.044×10⁵	9	1.006×10⁵	4.15	0.0300	Significant
A	96277.11	1	96277.11	3.98	0.0812	
B	3104.33	1	3104.33	0.13	0.7295	
C	1183.17	1	1183.17	0.05	0.8306	
A²	3.076×10⁵	1	3.086×10⁵	12.71	0.0074	Significant
B²	53925.39	1	53925.39	2.23	0.1739	
C²	50189.93	1	50189.93	2.03	0.1878	
AB	1.722×10⁵	1	1.722×10⁵	7.11	0.0285	Significant
AC	1.400×10⁵	1	1.400×10⁵	5.78	0.0429	Significant

Source	Sum of squares	DF	Mean square	F-value	Prob > F	
Residual	1.937×10^5	8	24206.63			
Lack of fit	1.833×10^5	4	45837.45	17.80	0.0082	
Pure error	10303.22	4	2575.81			
Core total	9.981×10^5	16				

3.3 Effect of process parameters on the surface resistivity of PPy-coated composites

The effect of process parameters on the surface resistivity of PPy-coated needle-punched non-woven composites were analysed using 3-D response surface plots. The combined effect of punch density and punching depth on surface resistivity of the PPy-coated composites is shown in Figure 4. It is evident that, as punch density increases, the surface resistivity of the composites initially decreases and then increases. As punch density increases, the non-woven structure became more compact with small pores. As a result, PPy molecules were trapped inside fibre interstices and could not be released after washing, resulting in higher a PPy add-on, as well as a decrease in electrical resistivity. However, after achieving the lowest value of surface resistivity, it increased with an increase of punch density. This is due to the formation of large holes and needle-marks in the non-woven structure due to rigorous punching that allows PPy molecules to wash out easily from fibre interstices. As the punching depth increases, surface resistivity increases. This is because a low punching depth results in a gentle action, which in turn results in a uniform and bulky non-woven structure that is able to entrap PPy molecule in fibre interstices. This would be difficult to achieve in a compact structure prepared with a high punching depth.

The combined effect of punch density and fibre fineness on surface resistivity of the PPy-coated needle-punched non-woven composites is shown in Figure 5. It is evident that if fibre fineness increases at a high punch density, surface resistivity increases, which means that non-woven composites prepared with finer fibres have a lower electrical resistivity. Finer fibres have a higher specific surface area than that of coarser fibres. As as a result, finer fibres can hold more PPy, resulting in lower surface resistivity. If the accessible area of the fibre surface is increased, PPy molecules can be deposited on the surface, which is believed to have happened here. At a low punch density, however, surface resistivity decreases slightly as fibre fineness increases because, at a low punch density, thicker fibres are difficult to entangle to form a uniform non-woven structure, which leads to poor PPy add-on and higher resistivity. Punch density has the same effect on surface resistivity as discussed above.
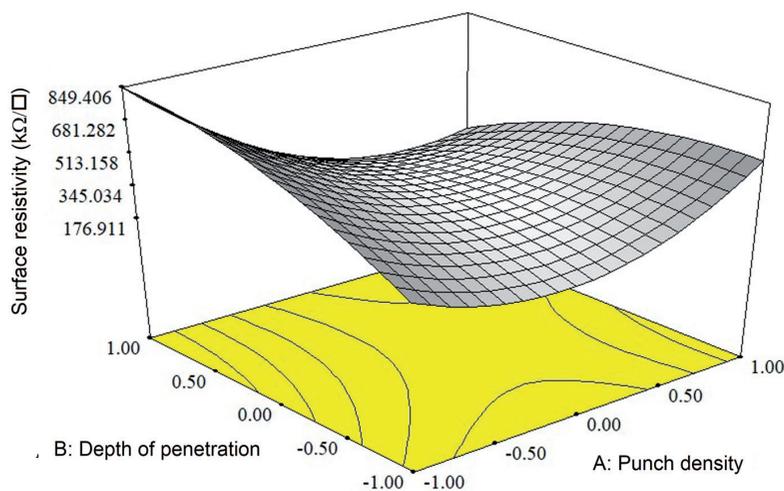

Figure 4: Effect of punch density and punching depth on the surface resistivity of PPy-coated needle-punched non-woven composites
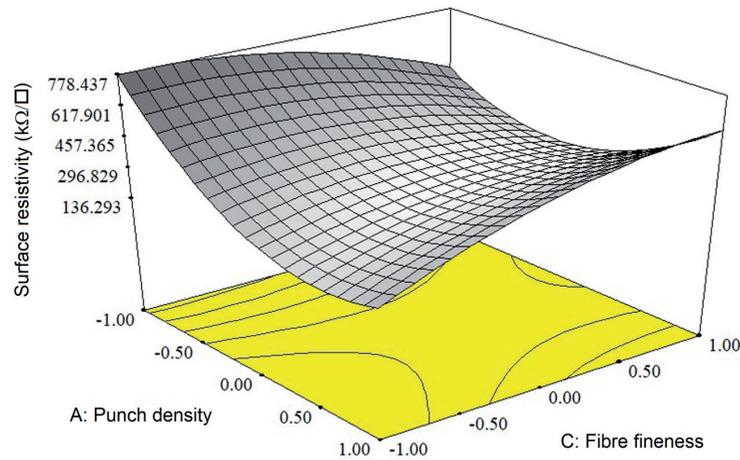
Figure 5: Effect of punch density and fibre fineness on the surface resistivity of PPy-coated needle-punched non-woven composites

3.4 Voltage-temperature (V-T) characteristics

The electro-conductive composites were tested for their voltage-temperature characteristics by applying a different range of direct current (DC). The surface temperature of the composites was measured using a non-contact type infra-red thermometer after an interval of three minutes of voltage application. The results obtained for sample no. 14 are shown in Figure 6. It is evident that as the applied voltage was gradually increased, the surface temperature increased due to Joule's effect. According to Joule's law, the heating effect produced by an electric current (I) through a conductor of resistance (R) for a time (t) is given using equation 3.

$$H = I^2 Rt \tag{3}$$

Joule's law states that the amount of heat production in a conductor is directly proportionate to the square of electric current flowing through it, directly proportionate to the resistance of the conductor, and directly proportionate to the time for which electric current flows through the conductor. Thus, an exponential trend of rising temperature was observed due to the application of DC voltage to the samples. For this sample, the surface temperature was raised by 18 °C from room temperature by applying of 30 V. Similar exponential trends were also observed in the case of PPy-coated polyester needle-punched non-woven by Maity et al. [8].

3.5 Voltage-current characteristics of electro-conductive non-woven composites

Ohmic conductors show liner V-I characteristics. To verify the nature of the PPy-coated needle-punched non-woven composites, current flow through the
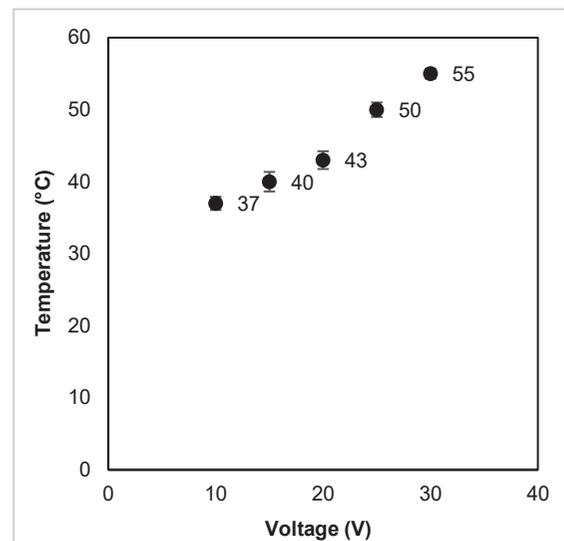

Figure 6: Voltage-temperature characteristics (V-T) of PPy-coated polyester needle-punched non-woven composite (200 g/m², 200 punch/cm², punching depth of 6mm, 3.33 dtex fibre)

samples due to the application of voltage was measured using an ammeter connected to the circuit. The results obtained for sample no. 14 are shown in Figure 7. V-I characteristics obtained as such follow a linear trend of I = 0.0383V with a coefficient of determination of $R^2 = 0.998$, which means current is directly proportionate to applied voltage such as an Ohmic conductor. The electrical resistance of the sample is 1/0.0383 = 26 Ω. Similar linear V-I characteristics were also observed in the case of PPy-coated composites up to the $FeCl_3$ concentration of 9 g/l used during the in situ polymerization [8, 16]. However, an exponential trend was observed if a higher concentration of $FeCl_3$ was used.
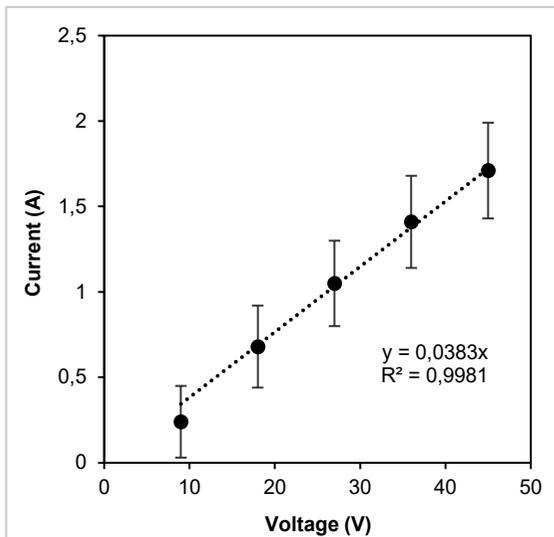
Figure 7: Voltage-current (V-I) characteristics of PPy-coated polyester needle-punched non-woven composite (200 g/m², 200 punch/cm², punching depth of 6 mm, 3.33 dtex fibre)

Figure 8: Effect of time on heat generation behaviour of PPy-coated polyester needle-punched non-woven composite (200 g/m², 200 punch/cm², punching depth of 6 mm, 3.33 dtex fibre)

3.6 Time-temperature characteristics of the electro-conductive non-woven composites

The time duration of the application of voltage may affect the heat generation behaviour of the electro-conductive non-woven composites [8]. To investigate this, fixed DC voltage (20 V) was supplied to the samples and allowed to heat up for an extended period. The results for sample no. 14 are shown in Figure 8. It is evident that temperature increased with time up to six minutes and then levelled off at a temperature of 49 °C. It can thus be concluded that these conductive non-woven composites heated up quickly due to the application of a fixed voltage and
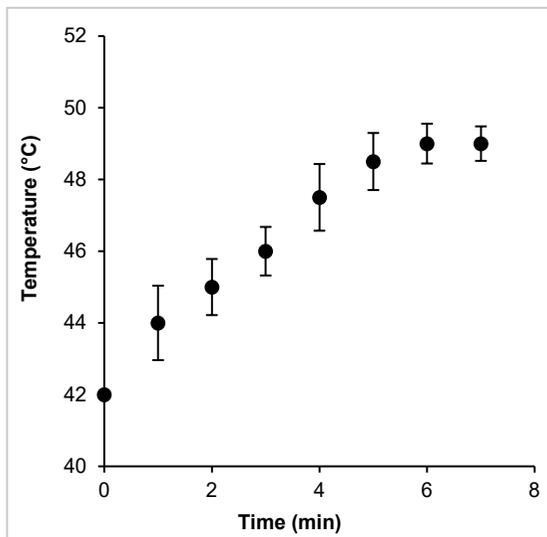
then levelled off at a particular temperature. Such behaviour of the non-woven composites is suitable for the fabrication of a heating pad or garment.

3.7 Optical microscopy image analysis of PPy-coated polyester

Optical and electron microscopic images of uncoated and PPy-coated polyester fibres (taken from the coated composite) are shown in Figures 9 and 10, respectively. It is evident that all individual fibre surfaces were coated with PPy molecules. As as a result, the fibres changed to greenish-black, similar to the colour of PPy. Uniform PPy coating was achieved on the surface of polyester fibres using granular morphology.
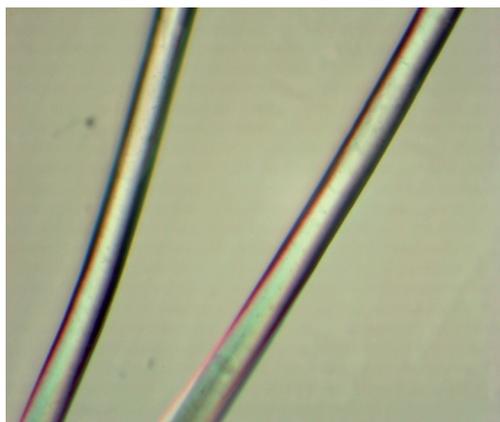
a)
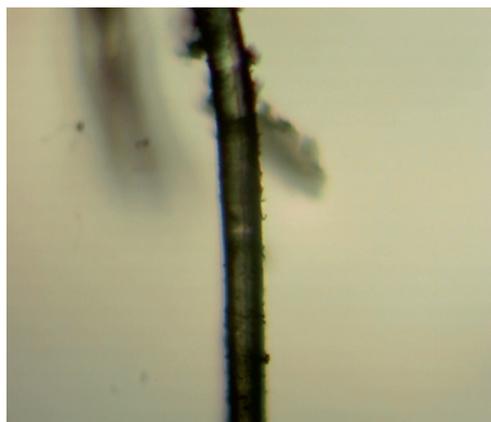
b)

Figure 9: Optical microscopy image (10×) of uncoated polyester fibre (left) and PPy-coated polyester fibre (right)
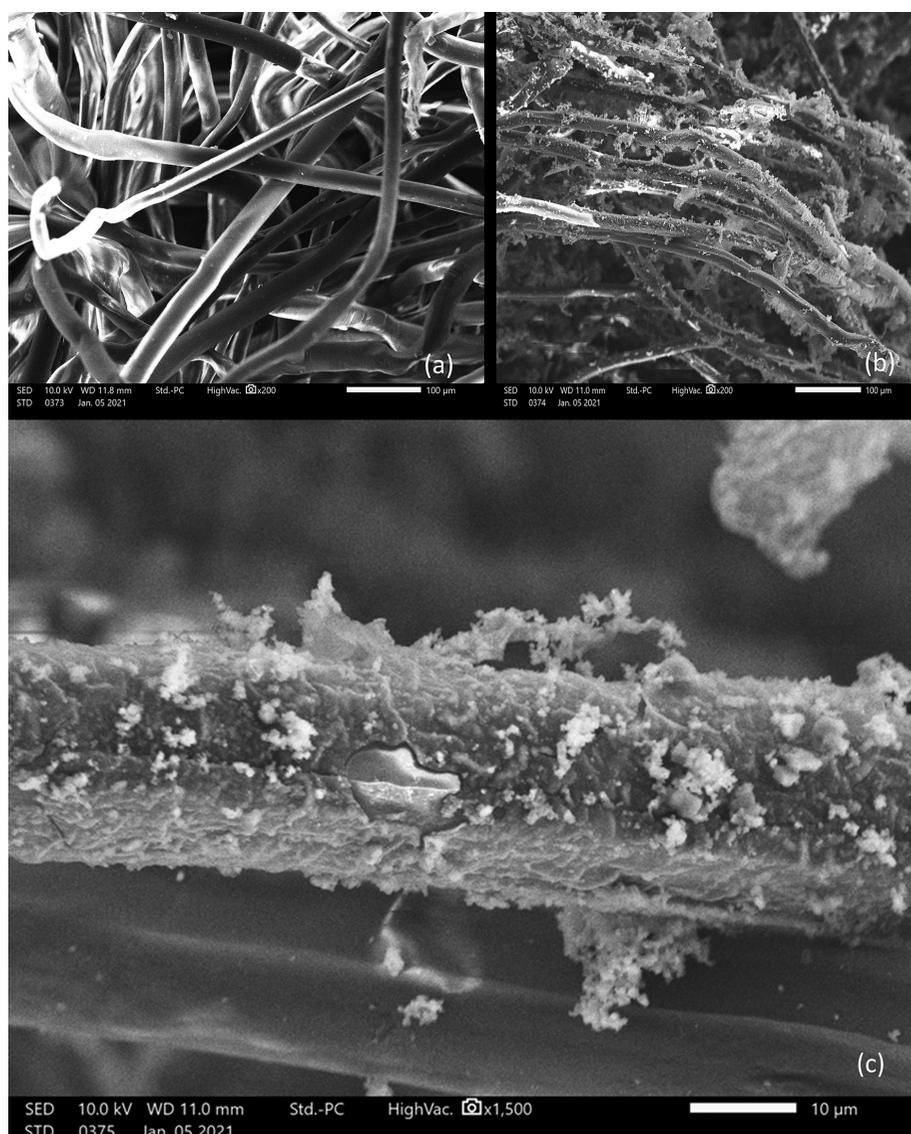
Figure 10: Electron Microscopy image of (a) uncoated polyester fiber 200× (top left); (b) PPy coated polyester fiber 200× (top right) and (c) PPy coated polyester fiber 1500× (at bottom).

3.8 Fourier transform infrared (FTIR) spectroscopy analysis

The FTIR spectra of polyester fibre and PPy-coated polyester fibres are shown in Figure 11. The FTIR spectra reveal various functional groups and chemical bonds that are present in polyester fibre and PPy polymers to identify chemical modifications and intermolecular interaction between them. In the case of uncoated polyester, observed peaks at wavenumbers of 2,948 cm^{-1} and 2,915 cm^{-1} are attributed to aliphatic C-H stretching vibration, while the band at 1,455 cm^{-1} is attributed to CH_2 deformation and the band at 1,374 cm^{-1} is attributed to the -COO-stretching of the ester group. In the case of PPy-coated polyester, the band at 1,541 cm^{-1} is attributed to the -C=C ring stretching vibration of PPy, while the band at 1,458 cm^{-1} is attributed to CH_2 deformation and the band at 1,304 cm^{-1} is attributed to the -CN stretching of PPy. The bands at 1,168 cm^{-1}, 1,028 cm^{-1}, 900 cm^{-1}, and 784 cm^{-1} are attributed to the characteristics of bending (C-H) vibration of PPy [10]. The band at 1,374 cm^{-1} due to the -COO-stretching of ester group in uncoated polyester vanished after PPy coating. These observations reveal significant intermolecular interaction between PPy and polyester fibres. This intermolecular interaction confirms the affinity of PPy molecules to polyester surface to achieve a durable coating.
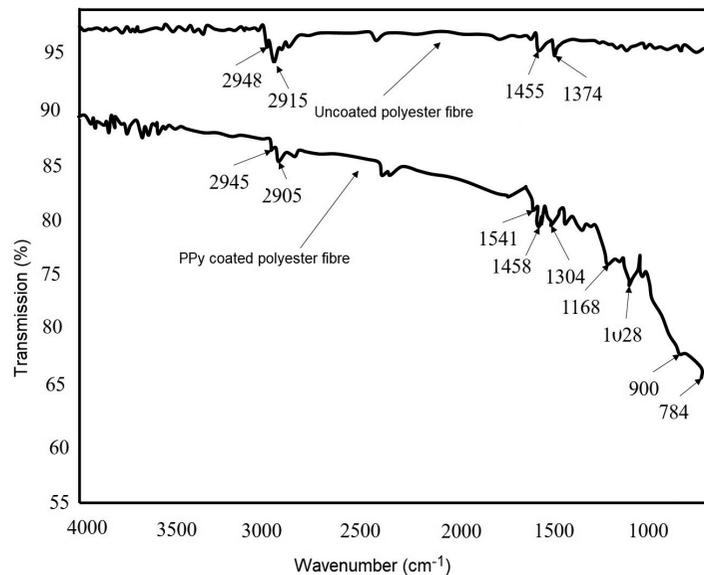
Figure 11: FTIR spectra of untreated and PPy-coated polyester fibre

4 Conclusion

The effects of parameters, such as needle-punching density, needle-punching depth and fibre fineness were investigated by employing the 3^3 Box-Behnken design and a response surface methodology. The results revealed that punch density was the most influential effect, while AB interaction (punch density-needle punching depth) was important for achieving the lowest surface resistivity. The highest PPy add-on of about 47.93% was achieved in the case of 200 g/m² non-woven fabric prepared with 200 punch/cm², a punching depth of 6 mm and 3.33 dtex fibre. The surface resistivity of this composite sample was found to lowest among all samples (9.32 kΩ). Voltage-temperature characteristic followed an exponential trend and the surface temperature of the PPy-coated composite was raised by 18 °C from room temperature by applying 30 V. Voltage-current characteristic followed a linear trend such as ohmic conductors with a linear resistance of 26 Ω. The composites heated up quickly within six minutes and reached a stable temperature of 49 °C when a contact supply of 20 V was applied. Optical and electron microscopy images revealed the uniform but granular morphology of PPy molecules over the polyester composite surface. FTIR studies revealed intermolecular interaction between PPy molecules and polyester fibres. Such PPy-coated needle-punched non-woven composites can be a potential material for the fabrication of a non-metallic, light-weight, flexible heating pad for therapeutic use and for heating garments for cold weather.

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