

PERCOLATION-GOVERNED FORMATION OF CONDUCTIVE NETWORKS IN POLYANILINE-FUNCTIONALIZED TEXTILE COMPOSITES

REDKO, YANA^{1*} AND HUDZENKO, NATALIYA²

¹ Kyiv National University of Technologies & Design, Mala Shyianovska (Nemyrovycha-Danchenka) str. 2, 01011 Kyiv, Ukraine

² Leibniz Institute for Composite Materials GmbH, Erwin-Schrödinger-Str. 58, 67663 Kaiserslautern, Germany

ABSTRACT

Conductive polyaniline (PANI)-based textiles are developed using a heterocoagulation-controlled deposition mechanism. In contrast to conventional in situ polymerization, this approach exploits the interaction between oppositely charged PANI particles and textile substrates to form specialized percolation networks. Polyamide substrates form highly interconnected networks with excellent conductivity, while cotton nonwovens exhibit less regular pathways. The heterocoagulation approach allows for fine-tuning of the layer structure, lowers the percolation threshold, achieves high conductivity while maintaining textile flexibility, and enhances durability against washing, dry, and wet rubbing. This methodology presents a systematic approach for controlling the structure–property–percolation relationship in conductive textiles, enabling potential applications in sensors, antistatic fabrics, and flexible electronics.

KEYWORDS

Polyaniline; Heterocoagulation; Electrically conductive textiles; Percolation networks; Textile materials; Surfactant-assisted deposition.

INTRODUCTION

Surface modification of textile fibers is widely used to tailor their functional properties and expand the application potential of textile materials. Previous studies have demonstrated that modification of polyacrylonitrile fibers and polyamide textile substrates can significantly influence their surface characteristics and functional performance [1] [2]. Such approaches enable the development of advanced textile materials with enhanced electrical and physicochemical properties. Among various strategies, the formation of electrically conductive networks in polymer-modified textiles has attracted increasing attention for applications in antistatic materials, sensors, and flexible electronic systems.

Conducting polymers, particularly polyaniline (PANI), have attracted considerable interest in the textile industry due to their light weight, flexibility, tunable electrical properties, and potential for wearable electronics [3–9]. Traditionally, PANI has been deposited onto textile substrates by in situ polymerization or simple coating methods, achieving moderate conductivity. However, existing methods often fail to control the formation of continuous conductive networks on fiber surfaces, resulting in limited reproducibility, high percolation thresholds,

and mechanical instability [3–6]. In particular, in situ polymerization methods do not fully exploit the role of particle-substrate interactions or the influence of surface charge on percolation network formation [10]. An alternative approach involves surface deposition mechanisms, where conductive polymer particles or nuclei are selectively assembled on textile fibers through interfacial interactions. In particular, heterocoagulation, based on the electrostatic attraction between oppositely charged particles and substrates, offers a promising avenue for directing the organization of the conductive phase on the fiber surface [11–13].

The key concept underlying charge transport in heterogeneous conducting systems is the percolation theory, which describes the transition from an insulating to a conducting state after reaching a critical concentration of conducting domains [14] [15]. Beyond the percolation threshold, the appearance of percolation networks – continuous, interconnected structures spanning the volume of the material, provides long-distance electron transport. In textile composites, the formation of such networks is strongly influenced by the structural and raw material composition, the nature of the fiber surface, spatial confinement, and the method of deposition of the conductive phase [16] [17]. Therefore, controlling the

* Corresponding author: Redko Y., e-mail: 82yanet@gmail.com

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formation of the percolation network remains a central problem yet it is rarely addressed explicitly in terms of deposition mechanism–driven network topology in textile systems in the development of electrically functional textile materials. Thus, the aim of the work is to determine how the heterocoagulation-controlled deposition of PANI on textile substrates of different raw material composition and structure affects the formation of percolation networks and the electrical conductivity of the material. This will allow establishing structure–property–percolation relationships to ensure optimal electrical conductivity with a minimum content of active conductive polyaniline.

To achieve this aim, the following objectives were defined:

1. To synthesize electrically conductive textile materials by depositing polyaniline onto polyamide knitted fabrics and cotton nonwoven substrates under surfactant-assisted heterocoagulation conditions.
2. To evaluate the effect of anionic surfactants on polyaniline deposition efficiency and electrical conductivity, in comparison with systems synthesized without surfactants.
3. To analyze electrical conductivity as a function of aniline concentration, identifying percolation thresholds and conductivity transitions for different textile substrates.
4. To compare percolation behavior in structurally ordered (polyamide knitted) and disordered (cotton nonwoven) textile systems, highlighting the influence of fiber structure on conductive pathway formation.
5. To establish structure–property–percolation relationships, demonstrating that electrical conductivity is governed primarily by the formation and connectivity of percolation networks rather than by the total amount of deposited polyaniline.

EXPERIMENTAL

Polyamide (PA) continuous filament yarn with a linear density of 15.6 tex was used as the base material. A knitted polyamide fabric produced from this yarn was employed as one textile substrate. A cotton nonwoven material was used as a second substrate for comparison.

Conductive textile materials were prepared by oxidative polymerization of aniline directly in the treatment bath in the presence of textile substrates and surfactants. Aniline oxidation was carried out using ammonium peroxydisulfate as an oxidizing agent. The molar ratio of oxidizer to aniline was maintained at 1:1.3. An anionic surfactant, sulfonol (sodium alkylbenzenesulfonate, ASAS), was used to control particle charge and deposition behavior.

The treatment process was performed at a bath temperature of 18–22 °C with a treatment time of 15–

30 min. Under these conditions, nanodispersed polyaniline particles were formed in situ and deposited onto the textile substrates through a heterocoagulation mechanism, driven by electrostatic attraction between oppositely charged particles and fiber surfaces [18]. After treatment, the conductive textile samples were thoroughly rinsed with distilled water and dried under ambient conditions.

The electrical resistance of the treated textile materials was measured using a two-electrode compensating method, which allows determination of both bulk and surface resistance (R) with an ohmmeter. Electrical resistance measurements were performed at several locations on each textile sample in order to account for the inherent structural heterogeneity of textile materials. For each experimental condition, multiple measurements were carried out and the reported values correspond to the average results. The variability of the measurements is expressed as standard deviation (\pm SD). Electrical conductivity (σ) was calculated from the measured resistance values.

Color fastness of the conductive textile materials was evaluated according to standard test methods: washing fastness (DSTU ISO 105-S06:2009), perspiration fastness (DSTU ISO 105-E04:2009), and dry and wet rubbing fastness (DSTU ISO 105-X12:2009).

The amount of polyaniline deposited on the textile substrates was assessed indirectly by measuring the optical density (D) of the treated samples dissolved in sulfuric acid. The optical density was assumed to be proportional to the polyaniline content, and, consequently, to the initial aniline concentration in the treatment bath.

RESULTS AND DISCUSSION

In this study, a controlled heterocoagulation deposition mechanism was applied to deposit polyaniline (PANI) onto textile substrates. The approach is based on electrostatic attraction between oppositely charged PANI particles and textile fibers in the presence of anionic surfactants. Polyamide knitted fabrics and cotton nonwoven materials were selected as model substrates to evaluate the influence of fiber surface charge, substrate structure, surfactant concentration, and aniline content on conductive network formation and percolation behavior.

Effect of surfactants on polyaniline deposition

During aniline oxidation in the treatment bath, the use of ionic and nonionic surfactants allows the formation of colloidal dispersions of emeraldine salt with different surface charges. For heterocoagulation-controlled deposition, both the sign and magnitude of the ξ -potential of PANI particles and the textile surface are crucial.

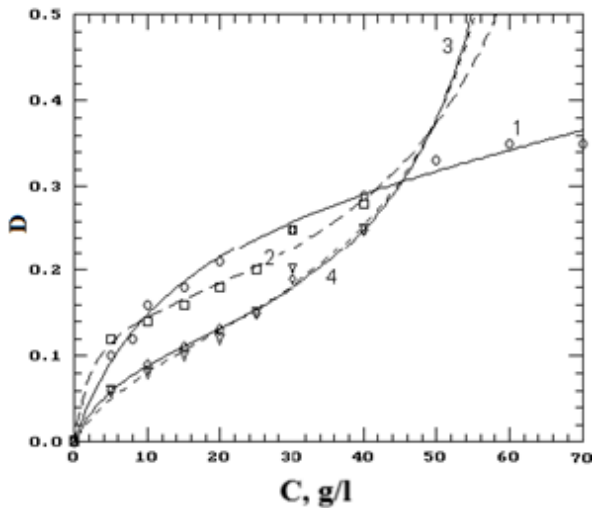


Figure 1. Dependence of optical density (D) of TM-treated solutions on aniline concentration (C): 1 – without surfactant; 2 – 0.5 g/l ASAS; 3 – 1 g/l ASAS and 4 – 2 g/l ASAS.

Table 1. Effect of aniline and surfactant concentration in the treatment bath on the synthesis of polyaniline on PA textile material.

C ASAS, [g/l]	Equation type*	Empirical equation parameters
0	$S = 0,01;$ $R = 0,997$ $y = \frac{a + bx}{1 + cx + dx^2}$	$a = 0.00268;$ $b = 0.02392$ $c = 0.066;$ $d = -0.0002$
0.5	$S = 0.013;$ $R = 0.99$ $y = \frac{a + bx}{1 + cx + dx^2}$	$a = 0.00028;$ $b = 0.0689$ $c = 0.429;$ $d = -0.0053$
1	$S = 0.012;$ $R = 0.993$ $y = \frac{a + bx}{1 + cx + dx^2}$	$a = 0.00318;$ $b = 0.01246$ $c = 0.0698;$ $d = -0.0011$
2	$S = 0.006;$ $R = 0.998$ $y = \frac{a + bx}{1 + cx + dx^2}$	$a = 0.00036;$ $b = 0.0189$ $c = 0.1349;$ $d = -0.0021$

* S – standard deviation; R – correlation coefficient; x – aniline concentration in the bath in g/l; y – optical density.

Polyamide fibers in acidic media (pH below the isoelectric point, pH < 5) exhibit a positive surface charge due to protonation of amino groups. When an anionic surfactant (sodium alkylbenzene sulfonate, ASAS) is used, PANI particles acquire a negative surface charge. This charge combination enables classical heterocoagulation, where negatively charged PANI particles are adsorbed onto the positively charged polyamide surface [19].

Surfactant molecules adsorbed on the surface of insoluble PANI particles suppress their uncontrolled growth and reduce particle–particle coagulation. As a result, the dispersion remains stable, and particle deposition onto fibers becomes more uniform.

Polyaniline content and sorption behavior

Figure 1 shows the dependence of the amount of synthesized PANI on the aniline concentration and surfactant content in the treatment bath. The experimental data are well described by empirical equations with correlation coefficients ranging from 0.99 to 0.998 (Table 1).

In the absence of surfactants (curve 1, Fig. 1), the dependence exhibits a typical sorption behavior with a tendency toward saturation. This behavior is characteristic of conventional dyeing processes, where dye uptake is limited by adsorption or absorption mechanisms [20] [21].

In contrast, in the presence of anionic surfactants, the shape of the curves changes qualitatively. A sharp increase in optical density is observed at aniline concentrations above 35–40 g/L, and no clear saturation is detected. Since PANI solutions in sulfuric acid obey the Bouguer–Lambert–Beer law, the optical density is directly proportional to the amount of deposited conductive polymer. Therefore, the observed behavior indicates a change in the deposition mechanism rather than simple sorption.

Applicability of the Langmuir model

Since polyaniline solutions in sulfuric acid obey the Bouguer-Lambert-Baer law, the optical density of solutions of dyed textile materials in sulfuric acid is proportional to the amount of electrically conductive dye (polyaniline) on the textile material. In the presence of anionic surfactants, the nature of the dependence undergoes a qualitative change: the curves do not show a tendency to saturation, but there is a rather sharp increase in optical density with an increase in the aniline concentration above 35 – 40 g / l. Thus, it can be stated that in the presence of anionic surfactants, the nature of the dye sorption changes qualitatively.

The curve of the dependence of the amount of synthesized dye on the aniline concentration in the absence of surfactants (curve 1 in Fig. 1) is a conventional dye sorption curve, which is characterized by a tendency to saturation. Knowledge of the process mechanism allows us to develop methods for intensification of dyeing processes. With a linear dependence of the dye concentration in the fiber on the dye concentration in the bath, the process of dye dissolution in the textile is usually taken as the mechanism, with a nonlinear isotherm, as a rule, the adsorption of the dye molecule on some internal surface of the fiber is taken as the mechanism [20] [21].

The nonlinear dye sorption isotherm can be described by the Langmuir model, which assumes adsorption

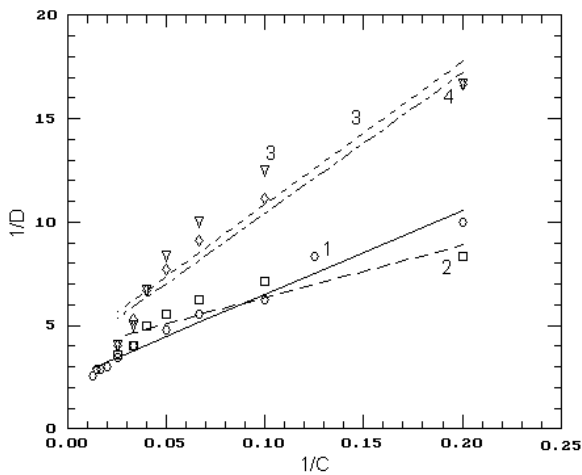


Figure 2. Dependence of the optical density of TM-treated solutions on the aniline concentration in the coordinates of the Langmuir equation. 1 – without surfactant; 2 – 0.5 g/l surfactant; 3 – 1 g/l surfactant and 4 – 2 g/l surfactant.

Table 2. Parameters of lines in Langmuir equation coordinates.

SAS Content [g/l]	$a \pm \sigma_a$	$b \pm \sigma_b$	R
0	40.7 ± 2.3	2.42 ± 0.18	0.99
0.5	25 ± 5	3.8 ± 0.5	0.91
1	70 ± 10	3.9 ± 0.9	0.95
2	68 ± 6	3.6 ± 0.5	0.98

with the formation of a monomolecular layer on the fiber surface. This approach has been successfully applied in previous studies on disperse dye sorption [22] demonstrating its adequacy for describing adsorption processes in textile materials. In the present study, the Langmuir equation is used in its linearized form [20] [21]:

$$\frac{1}{C_f} = \frac{1}{C_{f,\infty}} \frac{1}{K C_{f,\infty}} \cdot \frac{1}{C_s} \quad (1)$$

$C_{F,\infty}$ – saturation value corresponding to the filling of the monomolecular layer; K – sorption-desorption equilibrium constant, C_f i C_s – concentration of dye in the fiber and in the external environment at equilibrium.

The analysis of the dye dissolution process in the fiber under the assumption that the solvate theory of solutions can be applied was carried out in [20]. As active centers, fragments of macromolecules with an energy of intermolecular interaction sufficient for the formation of solvates were taken. The interaction of active centers with plasticizer molecules dissolved in an amorphous polymer takes into account the rule of molar particles [21]. When a low-molecular compound is sorbed by a polymer due to adsorption or absorption (with the formation of solvates) of intermolecular interactions, which provide the “lifetime” of the sorbate molecule in the adsorption layer or in the solvate similarly. For the absorption process with the formation of solvates, the expression [19] was obtained [20]:

$$\frac{1}{C_f} = B + A \frac{1}{C_s} \quad (2)$$

were $A = 1/(K_D + K C_{FMC,\infty})$ and $B = K/(K_D + K C_{FMC,\infty})$, K_D – dye distribution coefficient, $K = K_{MC} K_D$, K_{MC} – equilibrium constant in the reverse process of dye solvate formation, $C_{FMM,\infty}$ – maximum possible concentration of active centers.

Comparison of equations (1) and (2) shows that for the description of the absorption of a dye in a textile material, an equation similar in form to the Langmuir equation for monomolecular adsorption of a dye can be obtained. As can be seen from Fig. 2, the Langmuir equation coordinates describe the synthesis of polyaniline on a textile material in the absence of surfactant additives quite well. An objective assessment of the degree of adequacy of the description of the experimental data by a straight line in the coordinates of the Langmuir equation is possible using the least squares method.

For samples of polyamide textile material, in the absence of anionic surfactant in the dye bath, the dependence of the optical density (D) of the solution of the fabric dyed with a polymer dye on the concentration of aniline in the dye bath (C) is described by a straight line with a correlation coefficient of 0.986 (3):

$$1/D = (2.42 \pm 0.18) + (40.7 \pm 2.3)(1/C) \quad (3)$$

When deriving the Langmuir equation, it was assumed that dye molecules are adsorbed from the solution in the form of a monomolecular layer [20, 21]. During processing, aniline in the solution is in three states: a saturated solution in water, a solution in micelles due to solubilization, and dispersed aniline. When studying the real processing process, the total concentration of aniline in the processing bath was used, which does not correspond to the initial assumptions of the Langmuir equation. In this case, linearity in the coordinates of the Langmuir equation is the linearity of a parabola given in the reciprocals of the argument and the function. A similar type of functional dependence is characteristic of reversible processes with saturation [20]. Thus, the nature of the dye absorption isotherm curve by the fiber cannot be used as a criterion for the mechanism of dye-fiber interaction (dissolution or adsorption). To establish the mechanism of sorption of aniline or its oxidation products, it is necessary to use methods that are not related to the Langmuir equation.

Comparison of the standard deviations of the parameters of the straight lines in the coordinates of the Langmuir equation (Fig. 2) shows (Table 2) that the nature of the synthesis process, reflected in its result, is different for processing baths without and with the content of surfactants. It should be noted that the main changes occur at a surfactant concentration of up to 1 g/l, the dependence of the optical density on the concentration of aniline in the processing bath

are practically identical (Fig. 1, Table 2) at surfactant concentrations in the range of 1–2 g/l. Thus, comparison of the standard deviations of the parameters of the straight lines shows that the nature of the sorption of polyaniline particles is different for processing baths without and with the content of surfactants (Table 2). Therefore, the nature of the sorption isotherm curve cannot be a criterion for the interaction of polyaniline with PA TM (dissolution or adsorption of molecules from the solution in the form of a monomolecular layer).

A possible reason for the qualitative difference in the nature of the process of synthesis and sorption of polyaniline by fiber may be the process of heterocoagulation of negatively charged colloidal particles of polyaniline (due to the sorption of surfactant anions on their surface) on the positively charged surface of the polyamide fabric (at a pH greater than the isoelectric point, the surface of the polyamide textile material is positively charged due to the ionization of amino groups).

Electrical conductivity and percolation behavior

Polyaniline belongs to the class of electronically conductive polymers [12] [23]. They can be considered as derivatives of a polymer whose base form has the structure (a) and consists of reduced (b) and oxidized (c) repeating units (Fig. 3).

Complete protonation of the nitrogen atom in the imino group in the emeraldine base by an aqueous solution of hydrochloric acid (doping) is accompanied by a sharp increase in electrical conductivity [12] [23].

The first percolation threshold is the concentration of the electrically conductive component (in this case, the emeraldine salt), above (or at) which a continuous chain of particles of the conductive component appears. The sample loses its insulator properties and becomes a conductor, although the electrical conductivity may be significantly lower than that of metals.

Due to the structural heterogeneity of textile materials, electrical resistance was measured at several locations on each sample, and the reported conductivity values represent the average of repeated measurements. The standard deviation of the measurements did not exceed 8–12%, confirming acceptable reproducibility of the obtained data.

Figure 4 shows the dependence of electrical conductivity on aniline concentration for polyamide knitted fabrics and cotton nonwoven materials. For both substrates, the use of anionic surfactants leads to a significant increase in conductivity, with differences reaching up to two orders of magnitude compared to systems synthesized without surfactants.

A pronounced decrease in conductivity is observed in the aniline concentration range of 5–10 g/L. In

composite systems containing a conductive phase dispersed in an insulating matrix, such behavior is typically associated with disruption or incomplete formation of percolation networks [14]. Once the first percolation threshold is exceeded, a continuous conductive pathway is formed, and the material transitions from an insulating to a conductive state.

Influence of substrate structure and surfactant on percolation network formation

The textile structure strongly affects percolation network formation. Polyamide knitted fabrics exhibit lower percolation thresholds and higher conductivity due to their ordered fiber arrangement and stable inter-fiber contacts. These features facilitate the formation of interconnected conductive pathways.

It should be noted that the selected substrates differ not only in fiber chemistry but also in structural organization. Polyamide knitted fabrics represent a structurally ordered textile system with relatively stable inter-fiber contacts, whereas cotton nonwoven materials are characterized by a more disordered fiber arrangement and higher structural heterogeneity. Therefore, the observed differences in electrical behavior reflect the combined influence of fiber surface properties and textile morphology on the formation of conductive percolation networks.

In contrast, cotton nonwoven materials show higher percolation thresholds and lower conductivity. Their random fiber orientation and looser structure hinder the formation of continuous networks, requiring higher amounts of deposited PANI to achieve electrical connectivity.

Polyamide knitted fabrics, with their ordered fiber structure, formed well-connected PANI networks, resulting in higher conductivity, while cotton nonwoven substrates, with disordered fibers, exhibited less regular networks and lower conductivity. Comparisons across surfactant concentrations demonstrated that the formation and connectivity of percolation networks, rather than the total polyaniline content, dictate the electrical performance.

Electrical conductivity measurements further confirmed this: samples synthesized with anionic surfactants reached conductivities up to two orders of magnitude higher than those without, even when the total PANI amount was similar. This highlights the critical role of network structure in controlling macroscopic electrical properties.

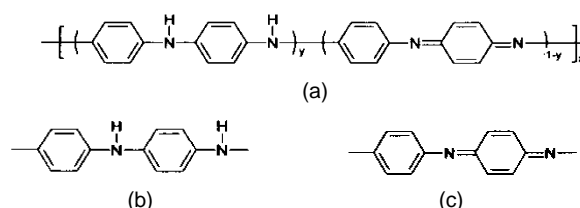


Figure 3. Structure of polyaniline and its forms.

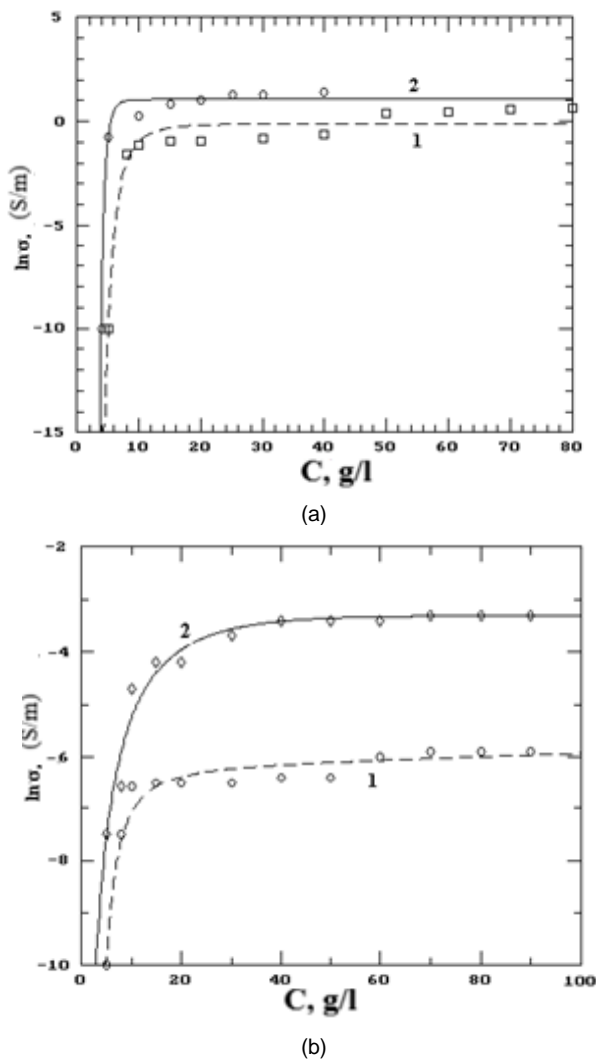


Figure 4. Dependence of the electrical conductivity of polyamide TM (a) and cotton nonwovens (b) (σ) on the concentration of aniline (C) in the treatment bath. Curve 1 – synthesis without surfactant, curve 2 – synthesis of PANi with 0.5 g/l of surfactant. The presented conductivity values correspond to mean values obtained from repeated measurements on different areas of the textile samples (\pm SD).

Table 3. Functional properties of the developed electrically conductive of polyamide knitted fabric.

Property	Value
Electrical conductivity σ (S/m)	$(1.0 \pm 0.2) \times 10^{-2}$ – $(1.0 \pm 0.1) \times 10^2$
Washing fastness (ISO 105-S06)	5
Rubbing fastness – dry (ISO 105-X12)	4–5
Rubbing fastness – wet (ISO 105-X12)	4–5

* Electrical conductivity values represent mean results obtained from repeated measurements at several locations on the textile samples (\pm SD).

Overall, these findings show a clear structure–property–percolation relationship: by controlling deposition conditions and fiber interactions, it is possible to optimize conductive network formation and achieve high conductivity at lower polymer content, while preserving textile handle and durability.

The presence of anionic surfactants changed the nature of polyaniline deposition. Optical density measurements showed a sharp increase in polyaniline uptake above 35–40 g/l aniline in the bath, indicating that surfactants not only stabilize particles but also promote network connectivity. The Langmuir model confirmed that sorption alone cannot explain the differences; instead, controlled particle assembly and percolation network formation govern conductivity.

Role of controlled heterocoagulation

Controlled heterocoagulation plays a key role in determining the morphology and connectivity of the conductive phase. Electrostatic attraction between positively charged fibers and negatively charged PANi particles prevents uncontrolled aggregation and promotes uniform particle distribution.

This method allowed precise control of the formation of continuous percolation networks, linking substrate morphology, particle stabilization, and network connectivity to macroscopic conductivity.

Macroscopic conductivity is governed primarily by the formation of continuous percolation networks rather than by the total PANi content. Anionic surfactants perform a dual function: they stabilize PANi particles in the dispersion and direct their adsorption onto fiber surfaces. This leads to improved reproducibility, enhanced conductivity, and better mechanical stability of the conductive layers. The experimental results confirm the initial hypothesis regarding the influence of particle surface charge on PANi deposition during semi-continuous textile treatment. Since polyamide knitted fabric demonstrated significantly higher electrical conductivity after polyaniline deposition compared to the cotton nonwoven substrate, further evaluation of functional durability properties, such as abrasion and washing resistance, was performed only for this textile material (Table 3).

The proposed controlled heterocoagulation deposition mechanism enables the production of electrically conductive textile materials with high washing fastness and abrasion resistance (Table 3), making them promising for smart textiles, sensors, antistatic fabrics, and flexible electronics.

CONCLUSIONS

This study demonstrates that the electrical performance of polyaniline-based textile composites is governed primarily by the formation and connectivity of percolation networks, rather than by the total polymer content. Heterocoagulation-assisted deposition, mediated by anionic surfactants, enables selective assembly of negatively charged PANi particles onto positively charged fiber surfaces, promoting uniform coverage and network connectivity. Comparative analysis of polyamide knitted fabrics and cotton nonwoven materials shows that substrate

structure and surface charge critically influence network formation, percolation onset, and achievable conductivity. Surfactant-assisted heterocoagulation significantly reduces the percolation threshold, allowing high conductivity at lower monomer concentrations while maintaining textile flexibility and handle. These results establish a clear structure–property–percolation relationship, providing practical guidelines for designing electrically conductive textiles with controlled microstructure and optimized functional performance. This framework can support the development of wearable electronics, sensors, smart textiles, and other applications requiring efficient conductive networks.

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