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A New Approach for Preparation of Smart Conductive Textiles by Polyaniline through in-situ Polymerization Technique

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IN THIS work, cotton fabrics were treated by aniline (ANI) monomer. The treatment bath was consists of Aniline (ANI) at different concentrations (0.10.5- mole/l), oxidizing agent (0.2 mole/l) as Ferric Chloride and doping agent as Egyptol(0.2 g/l) at different pH(14-) which adjusted by hydrochloric acid (HCl). This treatment carried out in order to deposit polyaniline (PANI) through in-situ polymerization technique to achieve conductive cotton fabrics in one step. The resulted fabrics were monitored by different tests as electrical measurements (conductivity, dielectric constant), color depth (K/S), square meter weight, tensile strength and elongation. To verify the presence of polyaniline, the treated fabric sample was examined using energy dispersive X-ray spectrum (EDX) and scanning electron microscopy (SEM). The results of tests show that, the electrical measurements, color depth, square meter weight, tensile strength and elongation increase by increasing aniline concentration. This behavior arises from increasing the extent of the conductive polymer on the fabric surface. The formulation, $FeCl_3$ (0.2 M/L), Egyptol (0.2 g/L), aniline (0.2 M/L), temperature (25°C) and 4 hrs treatment at pH 2 is the best for polymerization of aniline on the surface of the fabric to produce conductive and antimicrobial fabrics.

Keywords: Aniline, in-situ polymerization, Conductive fabric, Electrical measurements.

Introduction

Polyaniline (PANI) is one of the conducting polymers that has many successful applications in routine life such as batteries[1], sensors [2], electro-chromic devices, capacitors [3], solar cells, corrosion inhibitors, light emitting diodes, metallization [4], covering metallic surfaces by coat, etc [5–9]. The widespread range of polyaniline applications is due to its simplicity of synthesis, cheap treating cost, chemical stability, and conductivity [10]. So many scientists interested in finding new requests such as its application in textile industries [11-13].PANI can be chemically synthesized in acidic media using different oxidizing agents, such as hydrogen peroxide, cerium nitrate, ammonium persulfate, potassium dichromate, etc [14].

The formation of PANI on the fabric surface carried out into two steps. First step, fabric treated with oxidant and dopant second step, the treated fabric in the first step treated with aniline. This method consumed time and energy [11-13].So In this work the paper authors thought for other method or a new approach to save more time and energy, the synthesis of polyaniline in its doped state, by in-situ polymerization on fabric surface in presence of Ferric chloride as oxidant and Egyptol as dopant at different pH values for 4 hours and 25°C in one step was studied. This technique can prime to a decline in energy and time, so it considers a new approach in the formation of PANI on the fabric surface. Fabric output will be examined using several tests such as electrical measurements (conductivity, dielectric constant), color depth (K/S), square meter weight, antimicrobial activity tensile strength and elongation. Energy dispersive X-ray spectrum (EDX) and scanning electron microscopy (SEM) were helped in identifying the elements on the fabric surface and also to reveal the presence of polyaniline on the surface of fabric.

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Experimental and Methods

Fabrics and Materials

White cotton fabrics and Egyptol (non-ionic surfactant, Ethoxylated p-nonyl phenol) were supplied by El-Nasr Company for Spinning, Weaving and Dyeing, El-Mahallah El-Kubra, Egypt, Aniline monomer from sigma-aldrish. Ferric chloride and Hydrochloric acid of analytical reagent grade from Aldrich Chemicals. All aqueous solutions were prepared with distilled water.

Methods

Preparation of fabric coated by Polyaniline

Aniline was coated on the cotton fabric by in-situ polymerization technique by the following steps.

Aqueous solution of $FeCl_3$(0.2 M/L) and Aniline (0.1-0.5M/L) was prepared with doping agent Egyptol,0.2g/l, then cotton fabric sample was added into the solution by keeping the liquor ratio 1:25, adding drops of conc. HCl to adjust the pH medium, polymerization process was carried out in glass pots for 4 hr with continuous shaking. After polymerization, the samples were rinsed with water to remove unreacted monomer, and polymer excess, the samples were dried at ambient conditions. The fabric was dark green (color hue directed to black) in color resembling polyaniline. Eight samples were prepared by taking different concentrations of aniline and pHs as shown in Table 1

TABLE 1. Recipes for the preparation of substrates

Sample no.	ANI Conc. (M/L)	pH	$FeCl_3$ Con. (M/L)	Egyptol Con. (M/L)
1	0.2	1	0.2	0.2
2	0.2	2	0.2	0.2
3	0.2	3	0.2	0.2
4	0.2	4	0.2	0.2
5	0.1	2	0.2	0.2
6	0.3	2	0.2	0.2
7	0.4	2	0.2	0.2
8	0.5	2	0.2	0.2

Testing and analysis

Scanning electron microscopy (SEM): SEM measurements Microscopic investigations of fabric samples were carried out using a Philips XL30 scanning electron microscope (SEM) equipped with a LaB6 electron gun and a Philips-EDAX/ DX4 energy-dispersive X-ray spectroscope (EDX). Images were taken at different magnifications (from 1509 to 3, 0009), using secondary electrons (SE) in accordance with the clarity of the images. Fabric samples were fixed with carbon glue and metalized by gold vapor deposition to record images.

Square Meter Weight: The amount of PANI deposited on the cotton fabrics was determined by weighing the cotton samples before and after treatment under standard conditions of temperature (20˚C) and relative humidity (65%). The percentage weight increase (W%) was calculated as follows:

$$W\% = \frac{w_f - w_i}{w_i} \times 100$$

where W_i and W_f are the initial and final weight, respectively.

Electrical Measurements: To determine the dielectric properties, the fiber of diameter 9.4 mm is supported on the cell and conductivity, dielectric constant will be determined at frequencies between 0.1 KHz and 5 MHz at temperature 298 K by means of a HI0KI3532 LCR Hi-Tester with a computer. The real part of the dielectric function (constant) ε` of the coated samples calculated from the measured capacitance (C_p) according to the equation:

$$\varepsilon^{`} = \frac{C_p d}{\varepsilon_o A}$$

Where d is the diameter of the disc, A is its cross section area and ε_o is the dielectric constant of the vacuum ($8.85x10^{-12}$).

Tensile Strength

Fabric tensile strength test was conducted according to ASTM method 1682 (1994) (using Testometric (M350- 5CT), which is a standard method for breaking force and elongation of tensile fabrics [12]. The width and the length of the fabric strip were 50 mm and 200 mm, respectively.

Color Measurement

The color intensity expressed as K/S value, of the stained samples before and after treatment was, as a function for polymerization efficiency, determined spectrophotometrically using Data color spectrophotometer. K/S was calculated by applying the Kubelka-Munk equation [12]. All the determinations in this work were done in triplicate and the results present mean values.

Results and Discussion

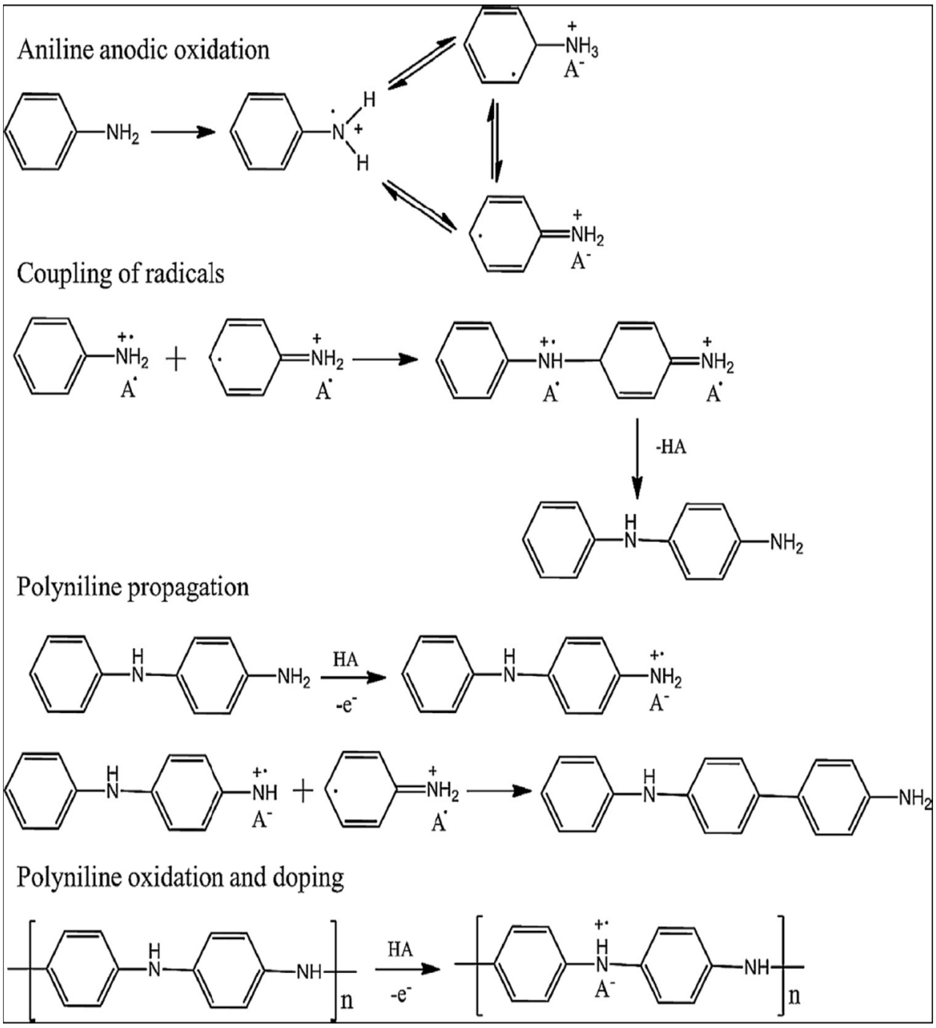
Tentative Mechanism of Aniline Polymerization

The reaction is initiated by the oxidation of monomer into radical cations, which combine to form dimers. The insoluble oligomers deposited on the surface and in the crevices of the textile fibers and fabrics by the continuation of the process. Due to low oxidation potential of aniline, a wide range of oxidizing agents can be used to initiate polymerization. Oxidant salts such as ferric chloride ($FeCl_3$) function as both the oxidant and the dopant agent, and hence the polymer is obtained in the conducting form. The transition metal ion is an electron acceptor. Therefore it oxidizes the ð-electron system of the aniline ring at the initiation step. The intermediate steps of the polymerization of aniline initiated by oxidizing agent. The polymerization mechanism of aniline as shown in Scheme 1 as follows [15]:

Reaction mechanism of Aniline polymerization

Aniline Concentration Effect

The effect of Aniline concentration (0.1-0.5 M/L) on the fabric properties, expressed as square meter weight, color strength (K/S), tensile strength and elongation when the treatment was carried out with Egyptol as a doping agent (0.2 M/L) and $FeCl_3$ (0.2 M/L) as an oxidant, and pH 2 at 25°C for 4 hours are described in Fig. 1-4. It can be seen in Fig. 1 that, the use 0.2 M/L gave colour strength 29.79, above this concentration no significant increasing of K/S values. This behavior arises from increasing the amount of polymer deposited on the surface of the fabric.



Scheme 1. Reaction mechanism of Aniline polymerization

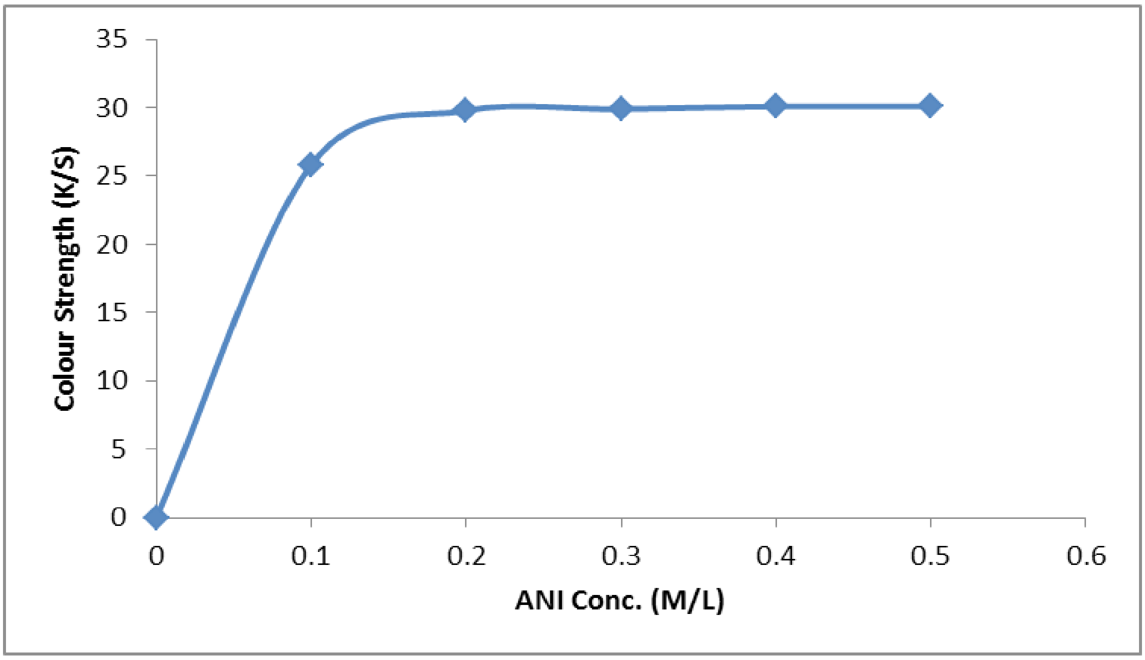
This is a result the increase of radical cations number which attached with each other to form polyaniline polymer by oxidant influence on the fabric surface and hence quantity of the polymer on the fabric increased [1].

Figures 2 and 3 show that, tensile strength and elongation of the fabric under investigation increase by increasing ANI concentration in treatment formulation. The increase in tensile strength and elongation of the coated fabric with PANI was attributed to the reinforcing effect of the high strength and modulus conductive polymer coating on the fiber [12].

Square meter weight affected by ANI concentration increasing in polymerization of ANI into fabric surface process as shown in Figure 4. To achieve appropriate square meter weight 0.2 M/L aniline can be used. This arises out of the same reason as mentioned in the preceding paragraph.

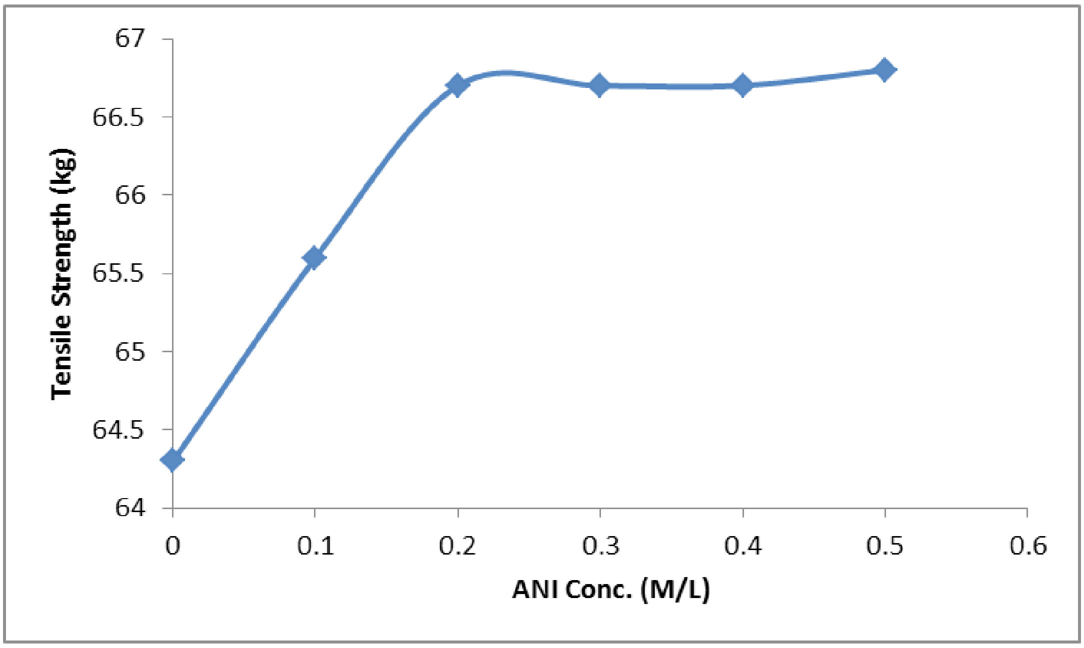
Effect of pH

Figure 5 depicts that effect of the pH values (1-4) of polymerization process medium on colour strength of the treated fabric. The treatment process carried out under the following conditions: Egyptol (0.2 M/L) and $FeCl_3$ (0.2 M/L), Aniline (0.2M/L) and pH 2 at 25°C for 4 hours. Figure 5 appears that, the change in pH value has an effect on the color depth value of the processed fabrics. On the other hand, the more acidic pH value results in depth of color; the most appropriate pH of the polymerization process of Aniline on the fabric surface is pH 2.This behavior is due to the fact that the acidic pH value leads to an increase in the protonated hydrogen, which, in turn, ionizes the aniline molecule (protonated ANI) and thus increases the bonding of the ionized aniline molecules with each other and with the cellulose molecule in the presence of the oxidizing agent (protonated chain) as described in Scheme 2 [15]. All of the above increased the amount of aniline polymerized on the surface of the fabric and thus increased the color depth of the treated fabrics.



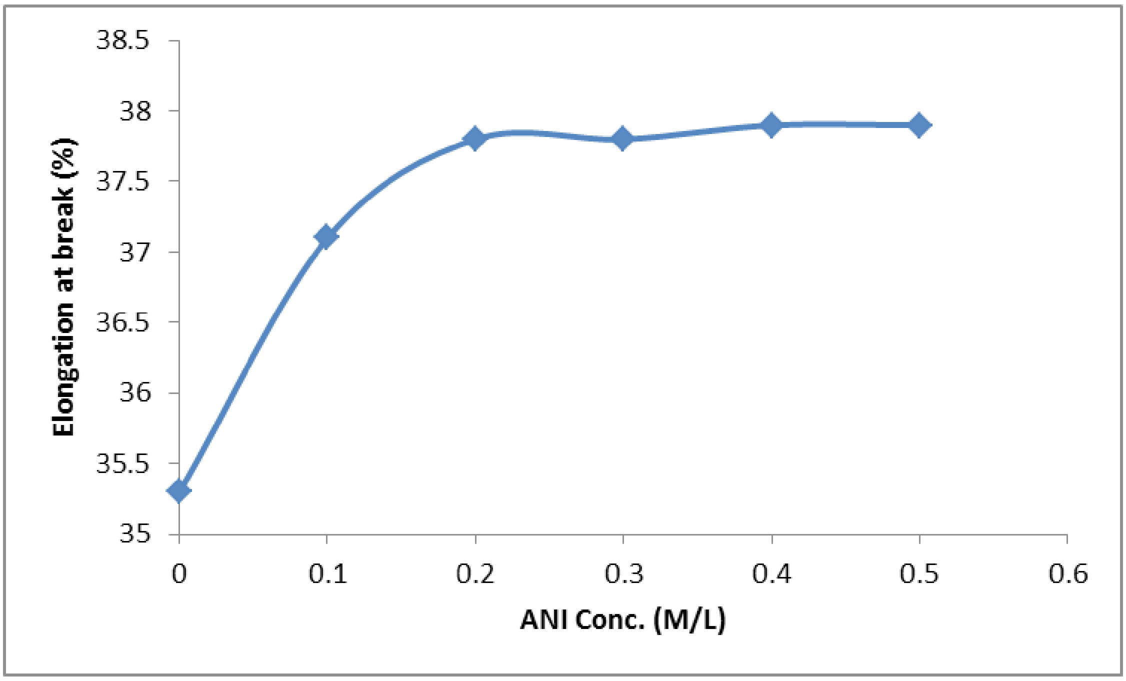
Constant parameters: [FeCl3]: 0.2 M/L; polymerization time: 4 h; polymerization temperature: 25°C.

Fig. 1. Effect of ANI concentration on the color strength of treated fabrics.



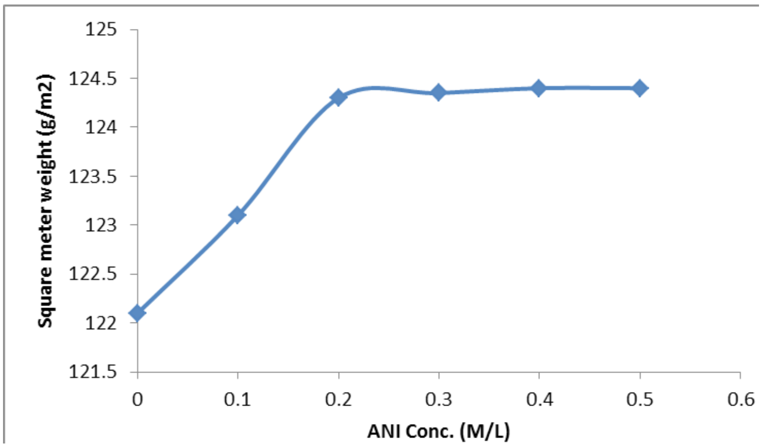
Constant parameters: [FeCl3]: 0.2 M/L; polymerization time: 4 h; polymerization temperature: 25°C.

Fig. 2. Coated fabric elongation change under $FeCl_3$ concentration effect.



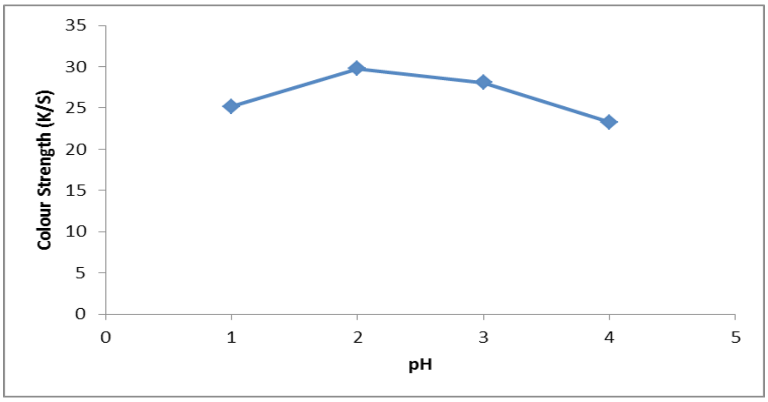
Constant parameters: [FeCl3]: 0.2 M/L; polymerization time: 4 h; polymerization temperature: 25°C.

Fig. 3. Effect of ANI concentration on the strength of treated fabrics.



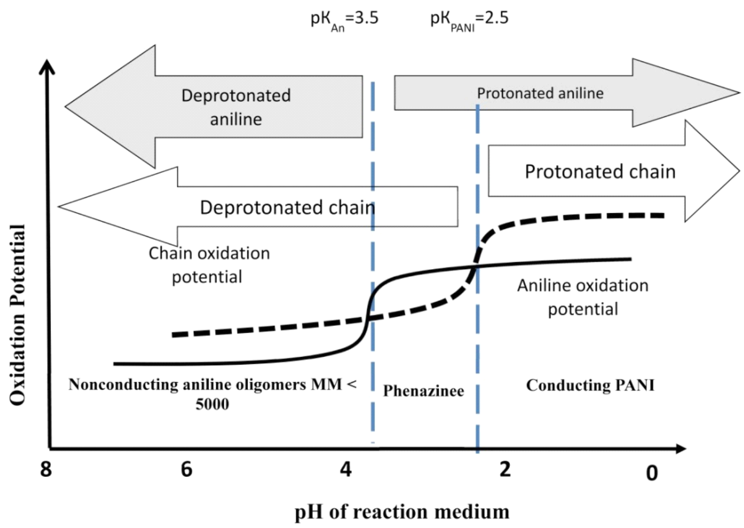
Constant parameters: [FeCl3]: 0.2 M/L; polymerization time: 4 h; polymerization temperature: 25°C.

Fig. 4. Square meter weight of treated cotton fabrics with the increase in ANI concentration.



Constant parameters: [ANI]: 0.2 M/L; [$FeCl_3$]: 0.2 M/L; polymerization time: 4 h; polymerization temperature: 25°C.

Fig. 5. Color strength of treated cotton fabrics with the increase in pH of polymerization process.



Scheme 2.

Constant parameters: [$FeCl_3$]: 0.2 M/L; polymerization time: 4 h; polymerization temperature: 25°C.

Fig. 2. Coated fabric elongation change under $FeCl_3$ concentration effect.

Oxidation of aniline depending on protonation constant of monomer pKAn and that of chain imino groups pKPANI [16].

Electrical properties of PANI coated fabric

The frequency dependence of the dielectric constant (Ý) for coated fabric in different concentration of the ANI monomer is shown in Fig. 6. The dependence of dielectric constant on the frequency of the alternating electric field or the rate of change of the time-varying field appears clear where there is a decrease in the dielectric constant with increasing frequency. At lower frequencies, the dielectric constant values are high which indicates polarity of the material. When the frequency begins to increase, the dielectric constant starts to fall indicating the independent of electric dipoles on the field variations [17]. The polarization resulted from charge accumulation decreases at higher frequencies, which leads to the decrease in the value of the dielectric constant till reaching a constant value [18–20]. Greater change in the values of dielectric constant at low frequencies with the concentration of the monomer is observed where the dielectric constant decreases with increasing the monomer concentration.

The effect of frequency on the ac-conductivity of the polyaniline (PANI) coated fabrics at different concentration of the aniline are carried out at 25°C and presented in Fig. 7and 8. It is clear that, the conductivity obeys the empirical law of frequency dependence given by the power law of the form [21]

$$\sigma(\omega) = \sigma_{dc} + A\omega^{s}$$

Where σ_{dc} is the Dc conductivity (frequency independent at the low frequency region), A is a constant depends on the temperature, s is the frequency 1 exponent (0.5 < s < 1) and the Ac conductivity is represented by $A\omega^{s}$.

The logarithm of the extracted ac conductivity values of the polymer coated fabric at different polymer concentrations are in linear relationship

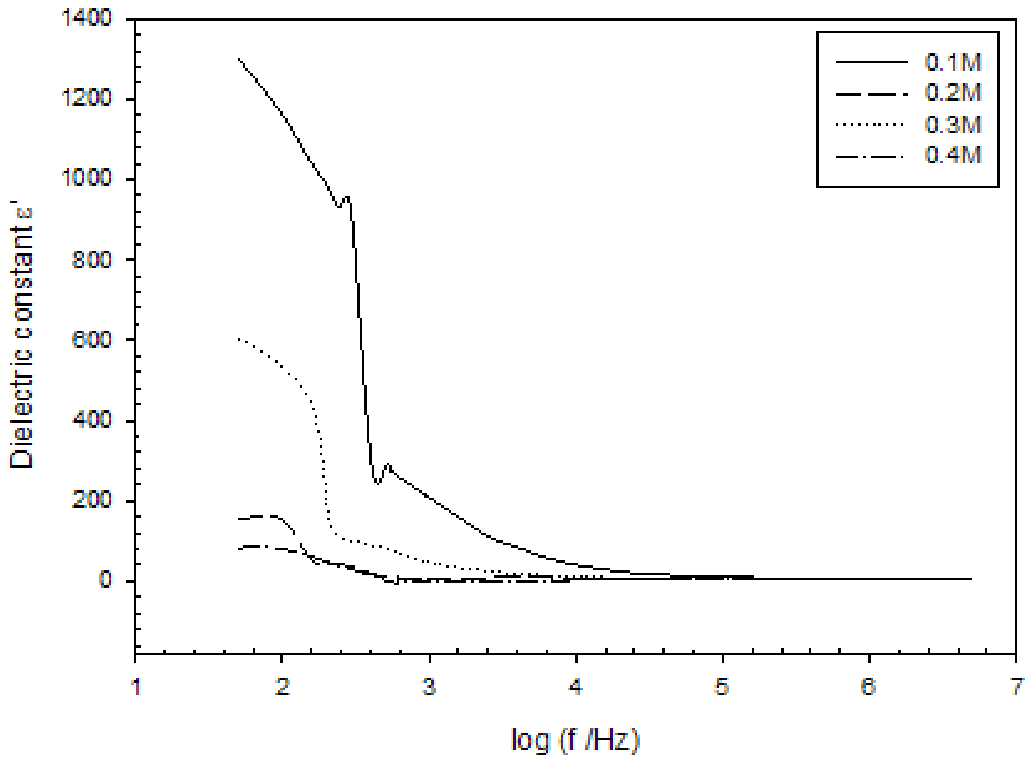


Fig. 6. Variation of dielectric constant as a function of frequency and monomer concentration at 25°C and pH 2 for PANi coated fabric

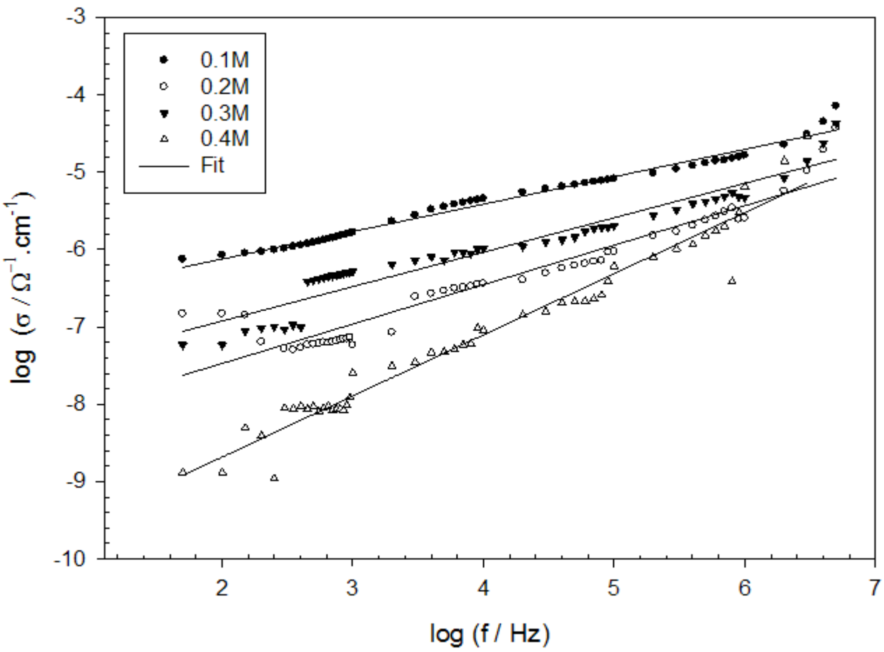
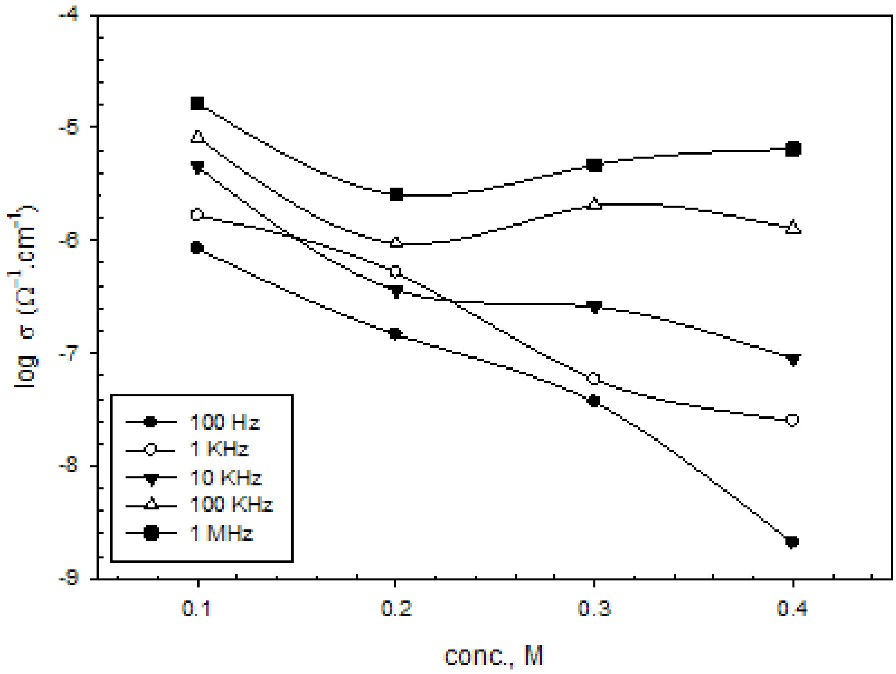


Fig. 7. Variation of conductivity as a function of frequency and monomer concentration at 25°C and pH 2 for PANi coated fabric



with logarithm high frequency values as shown in Fig.7 and the slope of this line represents the s value. It is obvious from these plots that the ac conductivity is dependent on frequency and as the frequency increases the conductivity of the polymer coated fabric increases for the four monomer concentration, this increase indicates that there may be hopping charge carries which can be transported through the polyaniline chain in the defect sites [22]. Figure 8 represents the effect of the change in monomer (aniline) concentration on the values of conductivity at selected frequency values. It shows reduction in the conductivity of the polyaniline coated fabric with increasing the aniline concentration. When the monomer concentration increased the polymerization at the bulk of solution increased than that at the surface of the substrate as a result of that insoluble dendritic polymer was formed in solution. Non adherent insoluble dendritic polymers were deposited on the substrate which can be dissolved by washing leading to a decrease in the conductivity [23].

It is obvious that the conductivity of the PANI coated fiber ranging from 10^{-7}- 10^{-5} $Ù^{-1}.cm^{-1}$which is greater than the conductivity of the fiber without coating ($4.5x10^{-14}Ù^{-1}$. cm^{-1}).

The effect of changing the pH value of the polymerization bath on the dielectric constant and the conductivity of the produced polyaniline coated was presented in Fig. 9,10 respectively.

The behavior of the dielectric constant of polyaniline coated fabric at different pH values are looked the same as in case of changing the concentration of aniline which is high at low frequencies and reduced to low value at high frequencies. Also, greater change in the values of dielectric constant at low frequencies with the change in pH value of the bath is observed, however the dielectric constant increases with increasing the pH of the bath.

The trend of increasing the conductivity with raising the pH of the bath from 1 to 2 is observed at nearly all the selected frequencies then decreasing the conductivity or having nearly a constant value is observed for pH>2. Acidic media is required for polymerization of aniline, so increasing the pH value of the bath results in formation of different nature of short conjugation oligomeric material [24], also the protonation of emeraldine base in acidic media results in raising the conductivity [25, 26].

From the effect of monomer concentration and the pH of the medium on the conductivity of the polyaniline coated fiber, it is clear that 0.1 M aniline in a medium of pH 2 results in the most conductive coated fiber in the studied series with conductivity reaches to $2x10^{-5}$ $Ù^{-1}.cm^{-1}$ at high frequency (5 MHz).

SEM morphology of treated cotton fabric

A suitable way to donate information on

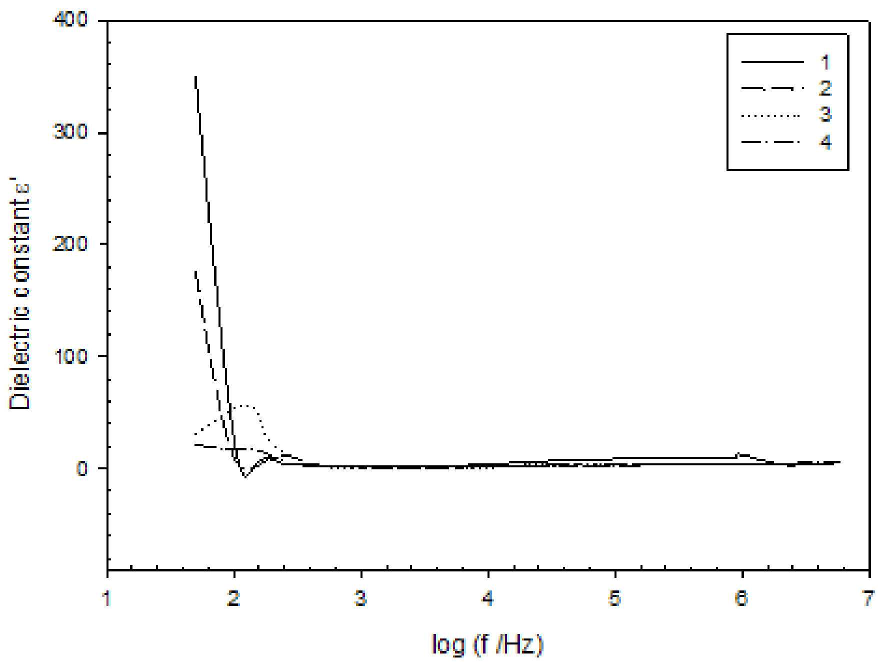


Fig. 9. Variation of dielectric constant as a function of frequency and pH of the polymerization bath at 25°C for PANI coated fabric (monomer conc. is 0.1M)

treated fabrics with PANI comes from SEM-EDX analysis. An elemental analysis of the particles was realized by SEM equipped with an energy dispersive X-ray spectrum (EDX), which can provide a rapid qualitative and quantitative analysis of the elemental composition (Fig. 11a). The EDX quantitative analysis (Fig. 11a) confirmed that the treated fabric about 44.86wt% carbon, 48.66wt% oxygen, 3.13wt% Fe, 3.35 wt% N and reminder wt% was traces of another element such as K. The SEM image (Fig. 11b) discloses the surface morphology of treated cotton fabric with PANI and the PANI deposition on fabric surface is clear. The tiny bright dots in SEM feature may be for PANI oligomers.

Antibacterial activity

Antimicrobial activity of fabric containing PANI as shown in Fig. 12. To investigate the activity of treated fabric with PANI towards different kinds of microorganisms such as *Staphylococcus aureus (Gm+ve), Candida albicans (Yeast) and Aspergillusniger (Fungus)* Fig.12 shows the cotton fabric containing PANI after being subjected to different microorganisms using diffusion method (zone inhibition method). It is clear that, treated fabric by PANI has antimicrobial activity for *Staphylococcus aureus* (S), *Candida albicans* (can) and *Pseudomonas aeruginosa* (A).This due to the presence of charge on aniline molecule which reacts with the cellular enzyme microbes, so killing microbes.

Conclusion

In this study, a new method was developed for the treatment of cotton fabrics using polyaniline. This method provided a great deal of time and energy. This treatment resulted in fabrics with distinct properties in terms of electrical current conduction and microbial growth resistance. Increased weight, color depth, and tensile strength of the sample after treatment are evidence of the success of this treatment and the achievement of the research objective. The resulted fabrics can be used for medical, biosensor and electromagnetic shielding applications.

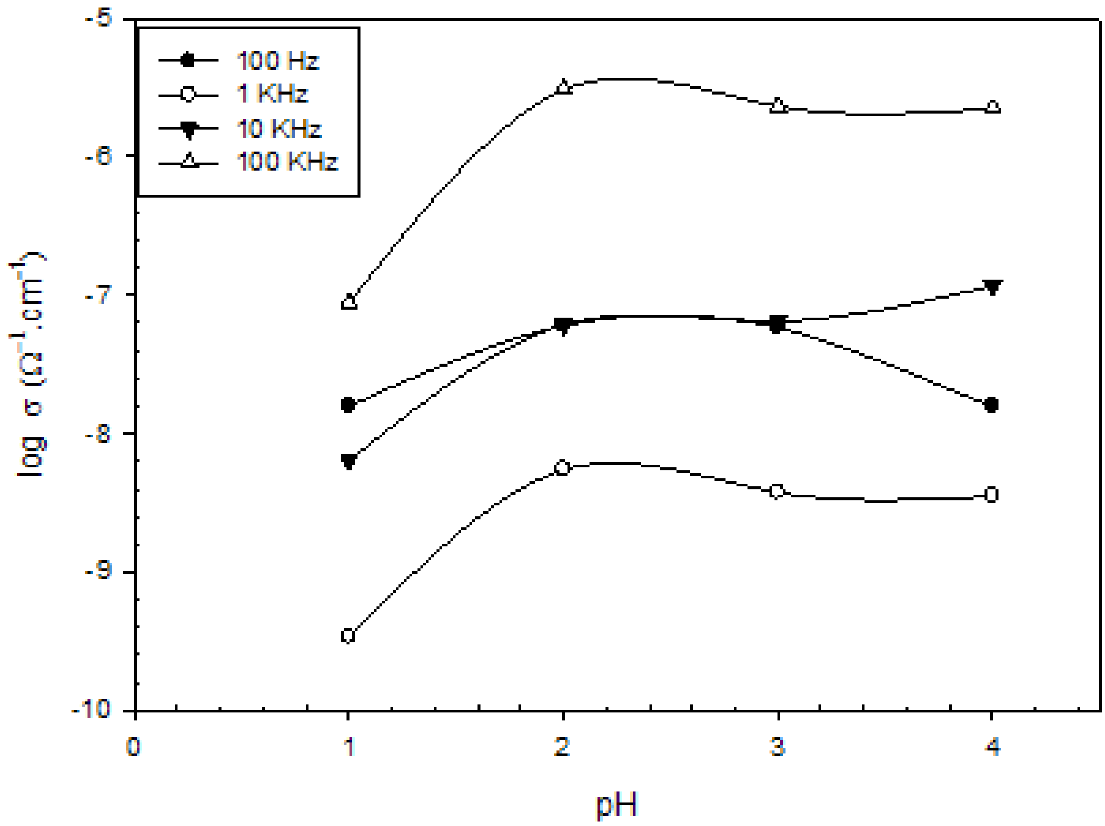
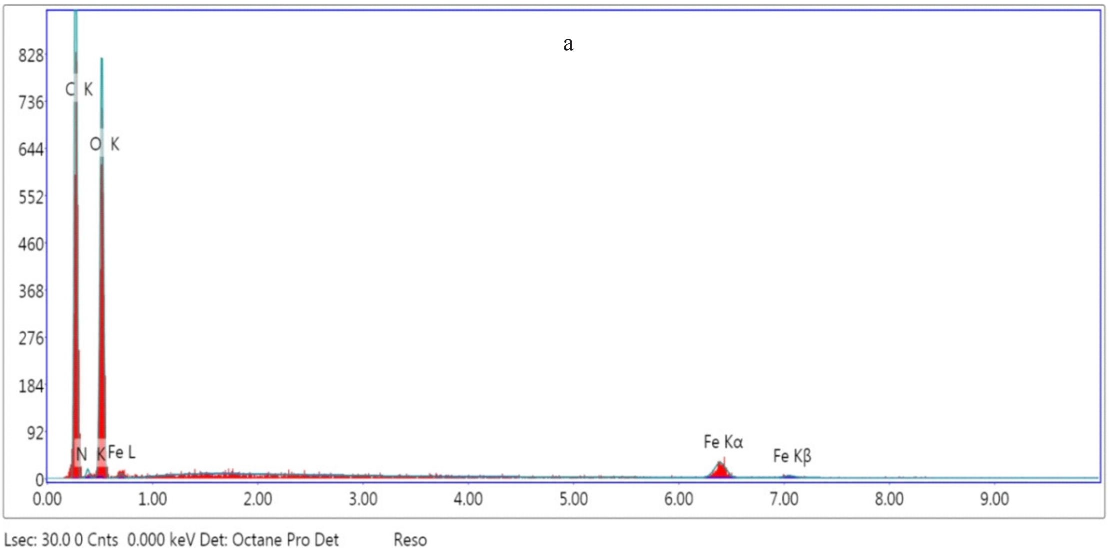


Fig. 10. Variation of conductivity of PANI coated fabric as a function of pH and frequency 25^{o}C for monomer conc. is 0.1M



Atomic %	Weight %	
52.81	44.86	C K
2.10	3.35	N K
43.01	48.66	O K
0.79	3.13	Fe K

b

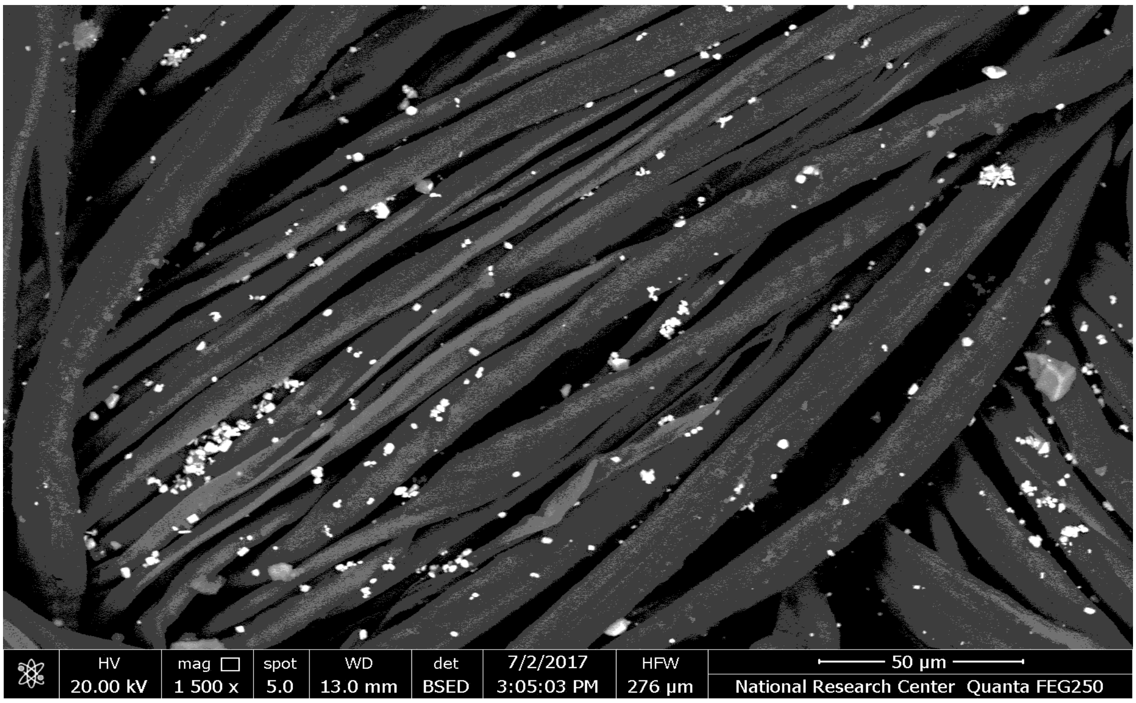


Fig. 11. (a) EDX of treated fabric with PANI. (b) SEM images of the surface morphology of treated cotton fabric with PANI.

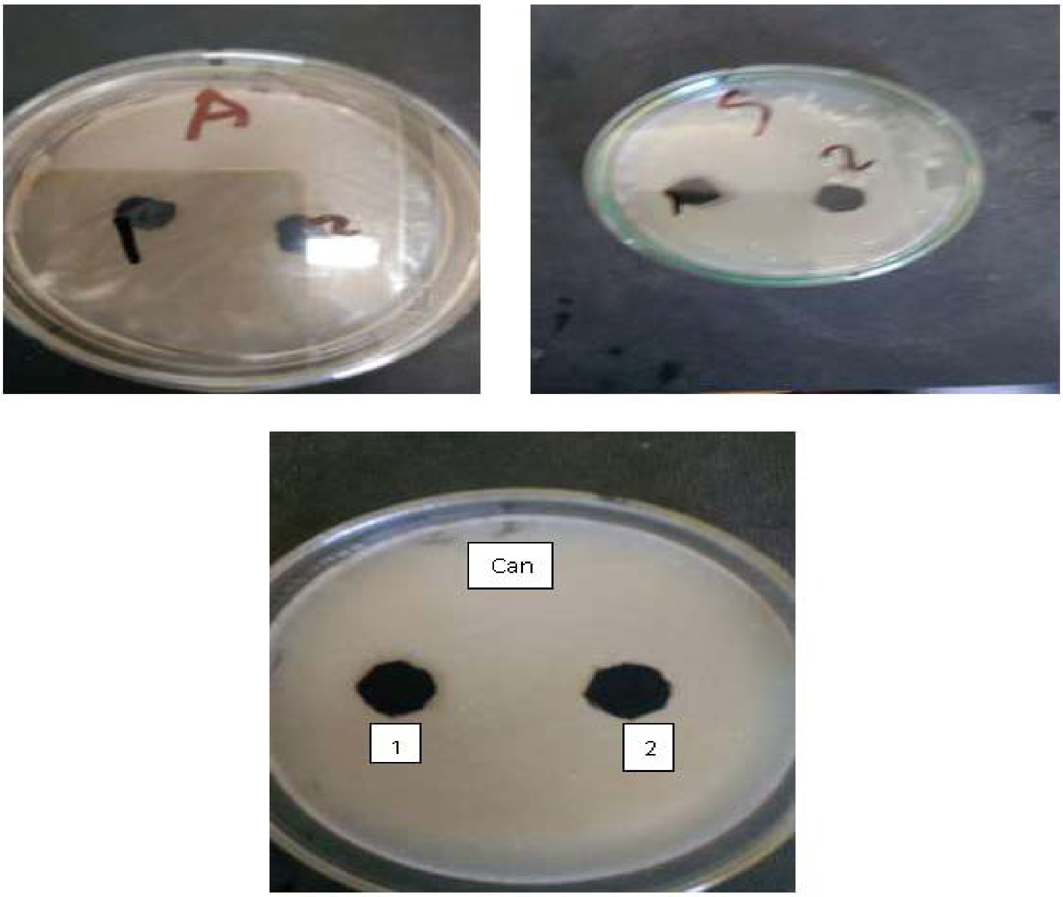


Fig. 12. Antimicrobial activity of the treated fabric with PANI against different types of microorganisms, 1- at pH 2 2- at pH 4

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اتجاه حديث لتحضير أقمشة قطنية ذات خصائص موصلة للكهرباء

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[١]شعبة بحوث الصناعات النسجية - المركز القومي للبحوث - الجيزة - مصر.
[٢]قسم الكيمياء الفيزيقية - المركز القومي للبحوث - الجيزة - مصر.

يهدف هذا البحث لتحضير أقمشة قطنية لها خصائص توصيل الكهرباء وذلك بمعالجتها بالا نلين بتركيزات من ١و٠ حتى ٥و٠ مول /لتر في وجود كلوريد الحديديك كمادة مؤكسدة بتركيز ٢و٠ مول/لتر عند أس هيدوجيني من ١-٤ وذلك باستخدام حمض الهيدروكلوريك عند درجة حرارة ٢٥°م وتتم هذه المعالجة في خطوة واحدة والأقمشة المعالجة تم اختبارها من حيث توصيل الكهربا, عمق اللون, قوة الشد والاستطالة ومقاومة البكتريا وكذلك تم عمل الأشعة السينية وماسح اللألكتروني.

وتوصل البحث أن أفضل النتائج كانت عند:
تركيز الأنيلين ٢و مول /لتر, كلويد الحديديك ٢و مول /لتر والأس الهيدوجيني٢