

## Microwave and Nanotechnology Advanced Solutions to Improve Eco-friendly Cotton's Coloration and Performance Properties

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**T**HIS RESEARCH aims to utilize microwave energy and nanotechnology to improve the printability and performance of cotton prints via screen printing technique. Each cotton sample was individually pre-treated by microwave power ranged from 300 to 700 watts and for a period ranged from 1 to 9 min. Afterwards the optimum sample was printed by printing paste containing Remazol™ reactive dye and silver nanoparticles (Ag-NPs) with different concentrations. The printed samples were fixed using microwave energy then were subjected to steaming or thermo-fixation. The obtained results clarified that, the prints obtained using microwave and Ag-NPs were found to have better color strength, fastness properties, antibacterial behavior and surface morphology when they were compared to the conventional techniques using thermo-fixation and steaming.

**Keywords:** Microwave, Nanotechnology, Printing, Cotton, Silver nanoparticles, Antimicrobial textiles.

### Introduction

Cotton is the most widely used of the textile fibers because of its intrinsic properties of biodegradability, breathability, hydrophilicity, comfortability and versatility, etc. Therefore, it is an excellent substrate for dyeing and printing as well as for imparting high performance with additional functionalities. Typical examples of such functionalities are antimicrobial activity, softness, easy-care, flame retardancy, and water & oil repellency [1-3]. To achieve this task, traditional wet methods were used for applying coloration and finishes, however they require large amounts of chemicals, water and energy. On the contrary, microwave (MW) is a dry processing technique that provide a solution to reduce the use of the above mentioned three resources [4].

The mechanism of microwave heating can be explained as follows: Many materials with large dipole moments, such as water, are rotated or vibrated vigorously in the presence of a magnetic

field caused by the frequency of the alternating current of microwave as (the 2,450 MHz microwave energy), then they are heated by quick energy conversion from the above kinetic energy. As a result, microwaves can usually heat various materials more quickly and homogeneously than conventional methods such as heat conduction or heat convection[5, 6].

(MW) energy applications have been used for many years in industrial technology. According to the previous literatures, (MW) irradiation has been successfully applied to a number of classical reactions such as dyeing, finishing and drying [7-12]. Microwave can provide various fabrics [13-17] with high coloration efficiency and color fastness properties in short heating time and low energy. Also, it is easy to be controlled and it is pollution free heating process. So, it is convenient for continuous production in textile industry[18, 19].

On the other hand, the importance of textile's functionalization with nanoparticles (NPs)

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materials such as (Ag), (Au), (Pt) and (Cu) have been increased in the recent years because of their electronic, optical, biological, chemical and physical properties. Various methodologies have been created using metal NPs to improve textiles functions as flame retardant, hydrophobic, antistatic, UV-protective and antimicrobial agents, etc. [20, 21]. Based on literature, nano silver particles (Ag-NPs) are widely used due to their antimicrobial behaviors [22-28]. It provides biological effect due to their large surface area in addition to the associated potential to release silver ions and it has been applied on different textile materials [28].

To the best of our knowledge, microwave has been utilized in textile dyeing applications and proved to be efficient. However, using MW in textile printing has not been explored as much. Therefore, the aim of this study is to evaluate microwave irradiation as a source of heat for pre-treatment of cotton fabrics and subsequent drying and fixing of print supplemented Ag-NPs in one-step. So, this work gives an environmentally eco-friendly solution for printing and finishing processes.

## Experimental

### Materials and instrumentation

In this study, 100% scoured and bleached plain weave cotton fabrics (200g/m<sup>2</sup>) were used, commercial reactive dye namely (Remazol blue RR gran) which based on methylsulphonylchloromethylpyrimidine, C.I. RB and Ludigol namely (Seracon M-lu) were received from (Dystar), sodium alginate (low viscosity), cream coloured powder, manufactured by Loba Chemie Chemical Company, were used. All other chemicals used during work such as urea, sodium bicarbonate were of laboratory grade chemicals.

Microwave treatment: Daewoo electronic - Korea, Model KqG-1N4A of maximum power 1000W.

### Methods

#### Conventional printing paste

Guide formulation for aqueous printing using flat screen printing technique follows:

Printing paste components	g/kg paste
Reactive dyes	40
Urea	100
Sodium alginate (12%)	500
Sodium bicarbonate	30
Ludigol	10
Water	X
Total	1000

#### Fabric treatment

Cotton fabrics were treated by microwave irradiation using microwave oven (IEC 705) that has the continuously adjustable power of 250-1000 Watt, the microwave frequency 2,450 MHz.

#### Procedure for studying the effect of microwave treatment power

Cotton fabric samples were placed in the microwave oven and exposed to microwave irradiation at various power settings, i.e. 300, 400, 500, 600 and 700 w for different durations 1,3,5,7 and 9 min, respectively.

Thereafter, the cotton fabrics were removed from the microwave oven and allowed to cool slowly at the room environmental condition (20°C ±2 and relative humidity 65% ±2), followed by printing and subsequent fixation according to the conventional technique by steaming at 103°C for 10min.

#### Procedure for studying the effect of microwave treatment time

Cotton fabric samples were placed in the microwave oven and exposed to microwave irradiation at power settings 600 W and for different durations 1,3,5,7 and 9 min, respectively.

After the irradiation treatment, the cotton fabrics were removed from the microwave oven and allowed to cool slowly at the room environmental condition (20°C ± 2 and relative humidity 65% ± 2%). Followed by printing and subsequent fixation according to the conventional technique by steaming at 103°C for 10min.

#### Fixation methods

After printing the optimum treated fabric samples were subjected directly, i.e. before drying to microwave irradiation for various periods of time ranging between 1 to 9 min at different microwave powers (400, 500, 600, 700, 800, 900 and 1000W). For the sake of comparison,

control samples of cotton fabrics were printed with the same paste, dried and subjected to fixation according to the conventional technique, i.e. either for steaming at 103°C for 10 min. or thermo-fixation for 5 min at 180°C according to (Dystar) technical data sheet for Remazol™ dye.

After fixation, the fabric samples were washed according to the aforementioned procedure in the experimental section followed by drying at ambient conditions and assessed for color strength (K/S) as well as overall fastness properties.

#### *Green synthesis of silver nanoparticles (Ag-NPs)*

The silver nanoparticles in this study were prepared according to the method described in Literature [29] with an improved modification. Ag-NPs were synthesized using *Plucheadioscoridis* Leaves extract (Pd) with different volumes. Six milliliters of Pd were mixed with 45 ml AgNO<sub>3</sub> (5 mM) in 100 mL Erlenmeyer flask. While, 20 ml of Pd were added to 45 ml AgNO<sub>3</sub> (20 mM) in other flask. The change in color from colorless to colloidal reddish brown indicated complete reduction of AgNO<sub>3</sub> to Ag-NPs.

#### *Procedure for studying the effect of silver nanoparticles on cotton fabrics*

Cotton fabrics were placed in the microwave oven and then treated with microwave irradiation at 600 W for 5 min. After the irradiation treatment, the cotton fabrics were removed from the microwave oven and allowed to cool slowly at the room temperature followed by flat screen printing and fixation in microwave at 700 W for 3 min. Ag-NPs were added to printing paste with different concentrations and volumes (10-40 ml). Ag-NPs synthesized using 6 ml Pd were used at various volumes [10 (T<sub>1</sub>), 20 (T<sub>2</sub>), 30 ml (T<sub>3</sub>) and 40 ml (T<sub>4</sub>)]. Therefore, Ag-NPs formed by 20 ml Pd were added with the same volumes [10 (T<sub>5</sub>), 20 (T<sub>6</sub>), 30 (T<sub>7</sub>) and 40 ml (T<sub>8</sub>)]. Also, untreated paste with Ag-NPs (T<sub>0</sub>) was used in printing to compare the Ag-NPs effect.

Guide formulation for aqueous printing using flat screen technique follows :

Printing paste components	g/kg paste
Reactive dyes	40
Urea	100
Sodium alginate (12%)	500
Sodium bicarbonate	30
Ludigol	10
Ag-NPs	(0_40)ml
Water	X
Total	1000

#### *Washing*

After printing and fixation via microwave irradiation, steaming and thermo-fixation, the printed samples were subjected to washing through 3 stages as follows:

- Rinsing with warm water.
- Soaping with solution containing 2 ml/l soap 60°C for 15min.
- Rinsing with cold water.

#### *Testing*

##### *Color Strength (K/S) measurement*

The color strength (K/S) of the printed cotton fabrics were measured using the (Konica Minolta spectrophotometer CM-3600 d) spectrophotometer (Minolta, Tokyo, Japan).

##### *Mechanical testing*

The tensile strength and percentage elongation at break of untreated and treated samples for different periods were carried out according to the standard method using a Shimadzu universal tester typeS-500 (C.R.T) (Japan) [30].

##### *SEM and EDX analysis*

Scanning electron microscope (SEM) images of conventional and microwave treated cotton fabrics were obtained using JEOL JXL-840A electron probe micro-analyzer equipped with energy disperse X-ray spectroscopy (EDX) for composition analysis.

##### *Assessment of color fastness*

Fastness to washing was performed using AATCC-61 Test Method [31]. Fastness to light was performed using AATCC-16 Test Method [32]. Fastness to rubbing was evaluated according to AATCC-8A Test Method [33].

##### *Antibacterial test*

Antibacterial activity assessment against G+ve bacteria (*Staphylococcus aureus* & *Bacillus cereus*), G-ve bacteria (*Escherichia coli*) and *Candida utilis* was evaluated qualitatively according to AATCC-147 Test Method [34] and expressed as zone of growth inhibition (mm<sup>2</sup>).

## **Results and Discussion**

This work exhibits a novel green procedure for printing cotton fabrics using microwave irradiation and silver nanoparticles to attain multifunctional functionality. Results obtained along with their appropriate discussion follow.

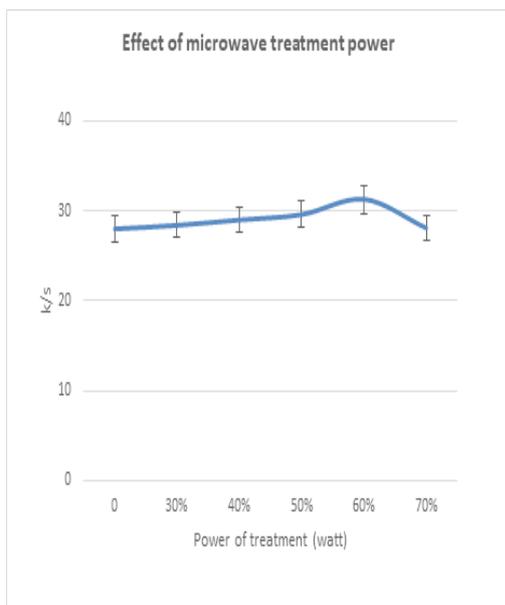
#### *Microwave irradiation treatment of cotton fabrics*

##### *Effect of microwave power on K/S*

As is evident in Fig.1, increasing the

microwave treatment powers from 300 to 600 W for 5 min brings about a noticeable increase in the K/S value until reached a maximum value at ( $k/s=31.3$ ), then decreased with a further increment. The main reason for this phenomenon is that, increasing the power of microwave up to 600 w, led to more rapid, uniform, efficient, and easy penetration of the dyestuff into the fabric i.e. higher absorbency.

On the contrary, the increasing in powers above 600 w changed the thermal stability and the crystallinity of the treated fabrics so its absorption became lower than before. [19]

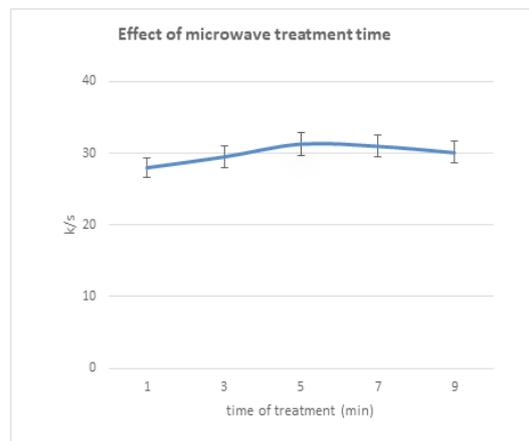


**Fig.1. Effect of microwave powers on (K/S) of cotton fabrics at 5 min.**

#### *Effect of microwave treatment time*

As depicted in Fig. 2, increasing the microwave treatment times from 1 to 9 min at 600 w brings about a noticeable increase in the K/S value until reached a maximum value at ( $k/s=31.3$ ) at 6 min, then decreased with a further increment. This may be attributed to the fact that, the increase of microwave time and power enhanced the fabric roughness which was favorable to improve the dye ability of cotton fabric.

On the contrary, the increasing in time above 6 min changed the crystallinity and the orientation of the treated cotton cellulose lead to lower absorbency. Accordingly, the optimum treatment time at power 600 w was found to be 6 min ( $k/s=31.6$ ). [10, 19].

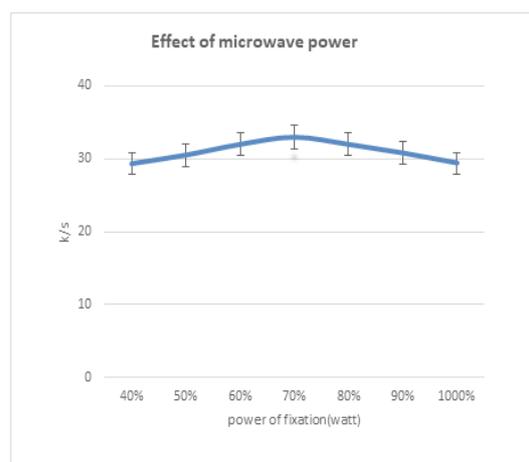


**Fig.2. Effect of microwave time on (K/S) of cotton fabrics.**

#### *Fixation of cotton fabrics by microwave irradiation*

##### *Effect of microwave fixation power*

It is clear from the data represented in Fig.3 that, increasing the magnitude of microwave fixation powers from 400 to 1000W for 3 min led to increase in K/S value. It increased up to 700 W, However, it decreased with further increase in power. The current data is expected since increase in the power at constant time is accompanied by an increase in the temperature. Accordingly, the rate of reaction between the reactive dye molecules and the hydroxyl groups of cotton increased. At relatively high power, i.e. 1000 W, the opposite holds true where the K/S decreased from 33 to 29.4 because of the decomposition and hydrolysis of some dye molecules as a result of over exposure to irradiation.



**Fig.3. Effect of fixation microwave powers on (K/S) of cotton fabrics at 3 min.**

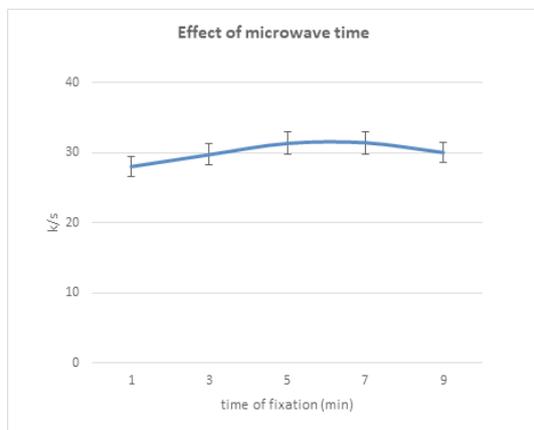
#### Effect of microwave fixation time

Figure 4 comprises the results obtained for the samples printed and fixed *via* microwave irradiation at various durations (1, 2, 3, 5, 6, 7 and 9 min) at optimum microwave power 700 W obtained from the power's study.

It is clear from the data of Fig. 4 that, as the time of exposure to microwave irradiation increased from 1 to 3 min, the K/S of the printed cotton fabrics increased regularly from 29.8 to 33. But it decreased with a further increase. The current data is expected since increase in the power at constant time is accompanied by increase in the temperature. It reached the maximum value at 3 min, afterwards, the K/S decreased with further increase. It seemed that, under these severe conditions, the produced heat affected the rate of reaction between cellulosic cotton and the reactive dye via either hydrolysis of some dye molecules or thermal decomposition [9].

Since the conversion of microwave radiation to heat energy [7] depends on the presence of polar water molecules, a trial was made to conserve some water molecules and decrease their evaporation and escaping from the printed film, this was done via covering the printed goods with a plastic sheet [35].

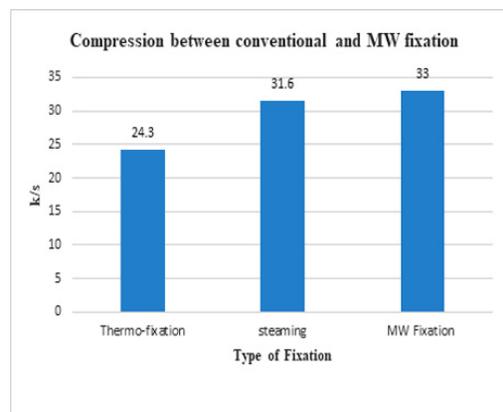
Accordingly, the optimum values of fixation time and irradiation power were found to be 3 min and 700 W.



**Fig.4. Effect of fixation microwave time on (K/S) of cotton fabrics**

A comparison was made to show the efficiency of the proposed technique shown in Fig. 5 and the results revealed that: K/S value= 33 of treated sample fixed with microwave (700 W at 3 min) was better than that treated and fixed *via* conventional thermo-fixation for 5 min at 180° °

C with K/S = 24.3, or that fixed by steaming for 10 min at 103° ° C (31.6).



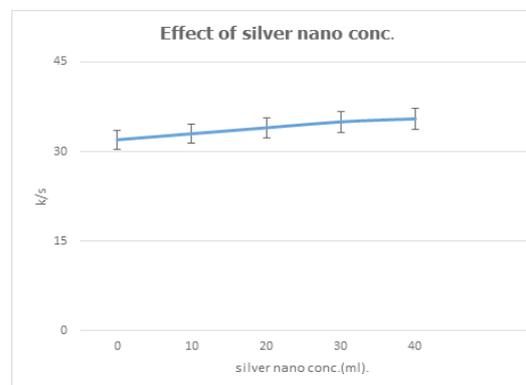
**Fig.5. Compression between conventional fixation and microwave fixation.**

It is inferred from Fig. 4 that microwave fixation ensured similar color yields in shorter setting time compared to conventional methods. So the new system represents an alternative method with the advantages of time savings and lower energy consumption.

#### Effect of silver nanoparticles concentration

Figure 6 shows that increasing the concentration of synthesized Ag-NPs resulted in a significant improvement in the color depth of the obtained prints, expressed as K/S values.

The observed data indicates that the increasing is achieved by using higher concentrations of Ag-NPs. This can be explained by the fact that, high number of spherical Ag-NPs were formed on the surface of the fabric by using more concentrated Ag-NPs colloidal solutions (cf. SEM results, Fig. 9, 10). According to that, more absorbency of the dyestuff occurred leading to higher k/s values.



**Fig.6 Effect of the concentration of Ag-NPs on K/S value of printed cotton fabrics.**

### Fastness properties

Table 1 represents the different color fastness properties (washing, rubbing and light) of all printed cotton fabrics that were measured according to standard methods AATCC Test Methods (61), (8) and (16A), respectively. The results obtained showed that all printed fabrics that were fixed via microwave irradiation

exhibited excellent fastness levels to washing and rubbing as well as light. The results are equivalent or even better than the samples that were fixed via conventional methods. These results reflect the efficiency of the proposed technique in producing printed cotton fabric with excellent fastness properties as well as a noticeable reduction in the powers and time that are used for fixation.

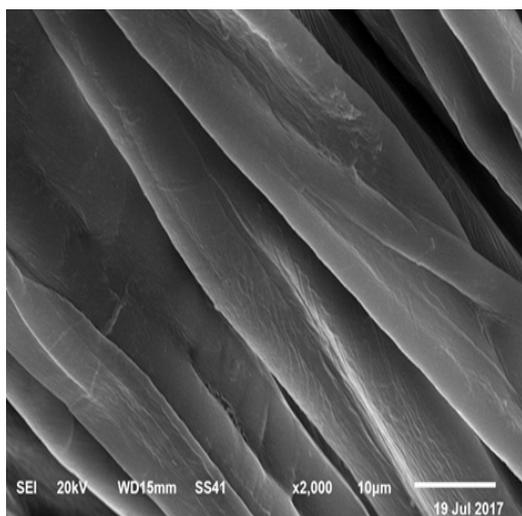
**TABLE 1. comparison between the printed samples using MW & Ag-NPs and the conventional printing techniques.**

Light	Fastness properties				Reactive dye used & optimum condition of each fixation type
	Rubbing		Washing		
	Dry	Wet	Alt. <sup>b</sup>	St. <sup>a</sup>	
4-5	5	5	5	5	microwave at 700w for 3min.
4-5	5	5	5	5	steaming (103°C for 10 min)
4	5	4-5	5	5	thermo-fixation (180°C for 5 min)

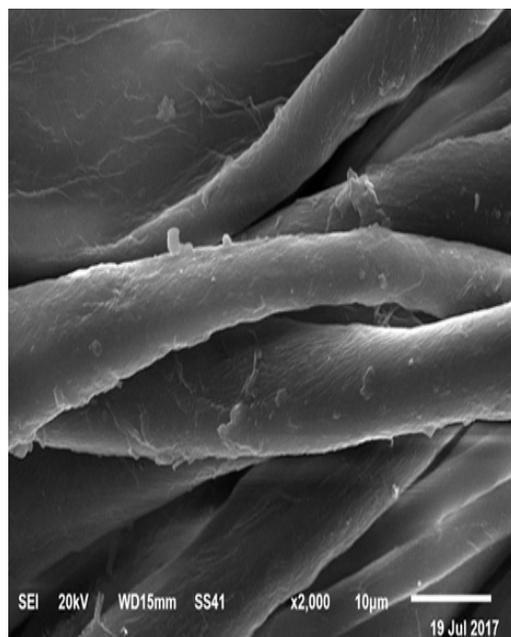
(a: staining b:alteration) on cotton fabric.

### SEM and EDx analysis

SEM observation of the cotton fibers reveals that the untreated fabric in Fig. 7 has fairly smooth surface, but the surface morphology of the treated fabric in Fig. 8 has been changed dramatically and there are different features on the surface of the fibers, such as grooves and voids and surface roughness. The obtained results were found in compliance with previous studies [10, 19]. As a result of this treatment, the morphological structure of the cotton fiber is partially destroyed, which promotes the adsorption and permeation of the dye molecules into the cotton fibers, resulting in improving the extent of reaction between the reactive dye and the cotton fibers.



**Fig.7 SEM image of cotton fabric without treatment.**



**Fig.8 SEM image of treated cotton fabric with microwave (Condition 600 Wfor 5 min before printing)**

### SEM and EDx for samples printed with Ag-NPs

SEM images in Fig. 9, 10 showed the nano silver particles on the fiber surface that incorporated into cellulosic fibers in cotton fabric. Ag-NPs were successfully distributed on the surface of all examined printed fabrics. Nano silver particles were observed in EDEX results in Fig. 11 with, 0.56% weight and 0.08% atomic

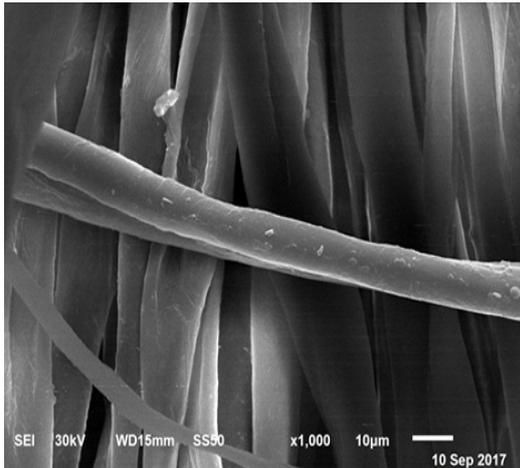


Fig. 9 SEM image of treated cotton fabric with Ag-NPs.

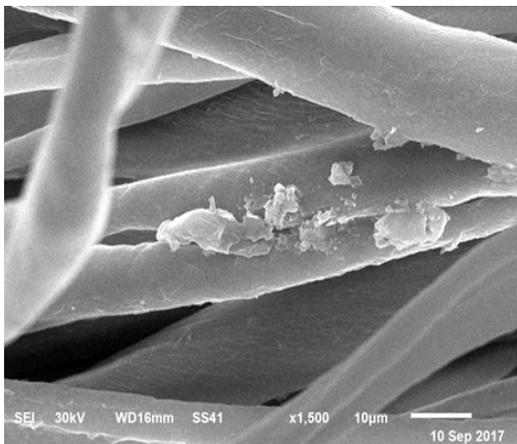


Fig. 10. SEM image of treated cotton fabric with Ag-NPs.

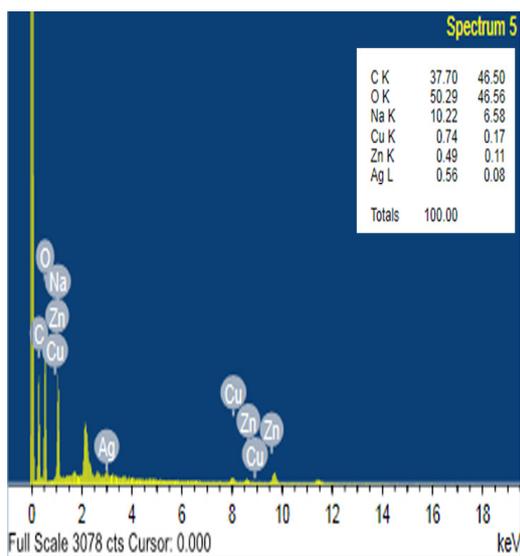


Fig. 11. EDX of treated cotton fabric with Ag-NPs.

absorption of the analyzed spot in the fiber surface.

#### Mechanical testing

The changes in the mechanical properties (tensile strength and percentage Elongation) of untreated cotton fabrics and those exposed to microwave treatments are assessed and the attained results are symbolized in Table 2.

The analysis proved that the microwave power and time of the treatment have significant effect on fabric breaking load. Elongation decreasing trend was detected assuring that as both variables levels

TABLE 2. Effects of microwave treatment on mechanical properties.

Treated sample with microwave	Untreated sample	Samples
357.6	412.0	Force(N)
7.88	8.48	Elongation (%)

Rate :100mm/min.

increased the fabric breaking load and elongation decreased [10].

#### Antimicrobial activity

Antimicrobial properties for fabrics printed with Ag-NPs are shown in Table 3. The results showed that the untreated printed samples ( $T_0$ ) were not affected and there were no inhibition areas, while the treated printed cotton samples using Ag-NPs have an excellent antimicrobial activity against *S. aureus*, *E. coli* and *C. utilis* in  $T_8$ , but they have negative effect in other concentrations ( $T_1$ - $T_7$ ).

*Bacillus subtilis* Gram positive and endospore forming bacterium which have a negative effect by Ag-NPs. The lowest value ( $82 \text{ mm}^2$ ) of antibacterial activity was in the case of the *Staphylococcus aureus* which is Gram positive bacterium and non-spore former. Antibacterial activity increased ( $155 \text{ mm}^2$ ) in the case of the Gram negative and non-spore forming bacterium (*E.coli*). On the other hand, the yeast *C. utilis* achieved the highest value of antimicrobial activity being  $220 \text{ mm}^2$ .

The finding of Keuk-Jun et al. [36] could explain these results, where he reported that, Ag-NPs have proven antibacterial activity and became promising in relation to fungi. Antifungal activities have become more common in recent

years. Moreover, the properties of Ag-NPs vary according to their size and shape [37]. The reason for improvement in the antimicrobial functionality is referred to the interaction of the negatively charged cell walls of the pathogens with the positively charged cationic sites of the antimicrobial agent, which in turn change its physical and chemical properties causing the interruption of the cell membrane functions and protein activity, as well as the inability to multiply. Silver nanoparticles release the silver ions producing higher biocidal effect on the microorganisms [3].

The sizes of Ag-NPs play an important role in their antimicrobial activity. A higher antimicrobial activity was observed only when Ag-NPs between 20 to 25 nm but not in 80 to 90

nm range [38]. Ag-NPs with a diameter  $13.5 \pm 2.6$  nm are effective against yeast [39]. Ag-NPs have a bioactivity role because of their large surface area and high reactivity. Ag-NPs metal particles exhibit remarkable physical, chemical, and biological properties [37]. The reason for fungal death could be explained by the finding of Long *et al.* [40] where Ag-NPs may accumulate in the cytoplasmic membrane, causing increase in permeability of the fungal cell and leading to the death.

It is known that nano-materials exhibit strong inhibiting effects towards a broadened spectrum of bacterial strains, especially Ag-NPs which displayed good activity against all the indicator pathogens, showing a potential broad spectrum antimicrobial activity [41]. Consequently, adding

**TABLE 3. Effect of Ag-NPs on antimicrobial properties of printed cotton samples.**

Sample	Inhibition zone area (mm <sup>2</sup> )*			
	<i>Bacillus cereus</i>	<i>Staphylococcus aureus</i>	<i>Escherichia coli</i>	<i>Candida utilis</i>
Printed (T8)	0	82	155	220
Printed (T0)	0	0	0	0

\* Area of antimicrobial clear zone (mm<sup>2</sup>) after incubation period (24h)

Ag-NPs to the printing paste brings a significant improvement in the antimicrobial activity of prints against pathogens.

### Conclusion

It can be concluded that the present work aims to green processing of cotton fabrics using recent innovative technologies, viz., microwave irradiation and nanotechnology for functionalization of fabrics which refers to improvement in printability, interaction with silver nanoparticles to impart antimicrobial activity to the fabric. These recent technologies are economically feasible, secure and acquire superiority over other conventional methods. The influence of microwave treatment on cotton fabrics was investigated. It is observed that the microwave irradiation eliminated the use of heat energy and time of fixation. Also, the color strength expressed as K/S of microwave treated cotton was higher than untreated ones. The result of SEM and EDX measurements confirmed the uniform distribution of Ag-NPs on treated cotton fabrics. The nano-printed fabrics have excellent color fastness properties and highly durable antimicrobial activity.

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### حلول متقدمه صديقه للبيئه باستخدام الميكرووييف وجزيئات الفضة النانومترية لتحسين خواص اللون والأداء الوظيفي للقطن

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يهدف هذا البحث إلى استخدام طاقة الميكرووييف وتقنية النانو لتحسين إمكانية الطباعة وأداء الأقمشة القطنية المطبوعة عبر تقنية الطباعة بالشاشة الحريرية. كل عينة من القطن تم معالجتها بشكل فردي بواسطة طاقة الميكرووييف التي تراوحت بين (٣٠٠) إلى (٧٠٠) واط لفته تراوحت ما بين ١ إلى ٩ دقائق بعد ذلك طبعت العينة بواسطة عجيبة الطباعة التي تحتوي على صبغة نشطة وجزيئات النانو فضه بتركيزات مختلفة. بعض من العينات المطبوعة تمت تثبيتها بواسطة طاقة الميكرووييف والبعض الأخرى بالطرق التقليدية من التبخير والتثبيت الحراري.

أوضحت النتائج أن العينات المطبوعة التي تم معالجتها وتثبيتها بواسطة الميكرووييف وبها جزيئات النانو فضة لها قوة لون أفضل وخصائص ثبات وخصائص مضادة للبكتيريا وذلك عند مقارنتها بالطرق التقليدية من التبخير والتثبيت الحراري.