



Antimicrobial Finishes for TENCEL/Cotton blended Fabric

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ABSTRACT

This research aims to prepare TENCEL/Cotton textile (50/50) against microbes and to use the best processing components to obtain the best specifications. Different concentrations of monochloroacetic acid and neutralized carbonate was used to treated samples were then in aqueous solution of sodium hydroxide to replace (OH) groups with (COONa) groups to prepare the textiles for binding with (CuSO₄) or (AgNO₃). The purpose this study is to examine the antibacterial activity of cooper or silver nitrate containing TENCEL/Cotton blended fabric against (*S.entridis*), (*B.cereus*), (*Penicillium*) and (*Aspergillus*) which are the microbes commonly exposed to house hold and hospital environments. The antibacterial effect was evaluated by a zone of inhibition test. The result showed that the highest microbial resistance and the least microbial growth is the concentration (2mol/l) in (*B.cereus*), (*Penicillum*) microbes, while the (*S.entridis*) and (*Aspergillus*) microbes were equal microbial resistance at concentration (1, 1.5, 2 mol/l).

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Introduction

Recently, diseases and epidemics have spread as a result of the spread of microbes that are transmitted to humans through infection, whether direct or indirect infection, through the presence of microbes on textiles and clothing. Hence, the aim of the study was to equip textiles against microbes to reduce the spread of infection. In order to increase the protection against microbes, new types of textiles have been researched that are more comfortable on the body and provide microbial protection. TENCEL/Cotton blended fabric were used in this research because of enhancement their anti-microbial properties and characteristics superior to cotton in terms of absorbency, softness, tensile strength and durability.

Cellulose

Cellulose is natural materials in all planets but ranges deferent, it exists for example in cotton where the cellulose content can be up to 94% and in other plant sources. In wood from trees, it exists in lower content due to the presence of lignin however the cellulose content is over 50 %. Cellulose can also be made by bacteria and are then called microbial or bacterial cellulose [1].

Types of cellulose

Cellulose-I is found in native cellulose, like wood, linen or cotton. The chains are parallel and bonded inter molecularly by H-bonds, laterally to sheaths, which are in their turn stacked. In the stacking direction Van-der-Waals bonding is what holds the crystal together. The upper limit for the stiffness of an ideal sample of this structure [1,2].

Cellulose-II is the result when cellulose is dissolved and precipitated to produce regenerated fiber chains are antiparallel and inter chain H-bonds connect in both directions perpendicular to the c-axis as shown in Fig.1 [1,2].

Cellulose-III is formed when cellulose is exposed to amines or liquid ammonia [1].

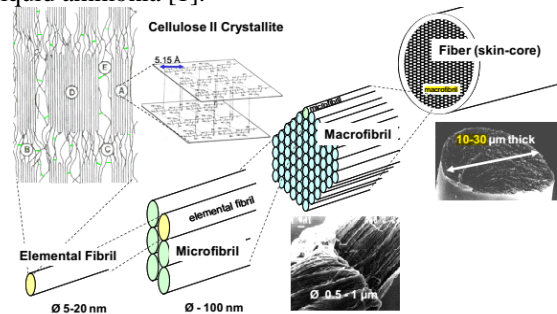


Figure.1 Cellulose II crystallite.

Cellulose-IV is formed when cellulose is treated with glycerol at high temperatures [1].

Regenerated fibers

Regenerated fibers are kinds of filaments, which are produced using normally happening polymers yet that are not in normally in type of strands but rather are prepared to be made into fiber structure; rayon, lyocell and acetic acid derivation are a few instances of recovered man-made strands comprising of cellulose polymer chains [3].

TENCEL (Lyocell) [3,4,5,6,7]

The lyocell is a Greek inferred word came as Lyocell: lyo (Greek word) which is really gotten from the word 'lyein' which implies break up and the word 'cell' infers as a short condensing from cellulose [8]. It is named TENCEL fiber after perceived by the International Bureau of Artificial and Synthetic Fiber Standards in 1989 [9]. The greatest maker of lyocell strands is Lenzing which moves their filaments and textures under the exchange name TENCEL. Since the strands are created with the lyocell procedure, it is better than numerous other cellulose based filaments with regards to ecological effect.

It is naturally benevolent generation process it is better than other cellulosic strands [10]. TENCEL is normal material originating from wood mash, midway position among common and concoction filaments, Natural wearing properties of regular strands joined with the upsides of manufactured filaments, for example, virtue and reliable quality [11,12]. Lyocell (TENCEL) is a cellulosic fiber recovered from eucalyptus wood [13], beech, spruce [14], and Bamboo [15]. The requirement for such a fiber was felt because of the negative natural impacts of the thick process [3], because the Viscose fiber process [16]. Once in a while causes eco sensible issues since it utilizes dangerous substance to break down as (NaOH) which it is toxic [17,18]. The procedure by which this fiber is made utilizations an amine oxide recovery shower and the cellulose polymer is broken down in amine oxide. As amine oxide has low poisonous quality along these lines, it is increasingly great and the cellulose in wood mash breaks up more effectively and without harming the cellulose as shown in Fig. 2 [3,17]. Lyocell is said to be nearer to cotton than some other fiber [3]. So, it is viewed as the third era of recovered cellulose fiber. As a result of its interesting properties, it had been connected into an assortment of merchandise in numerous fields [9].

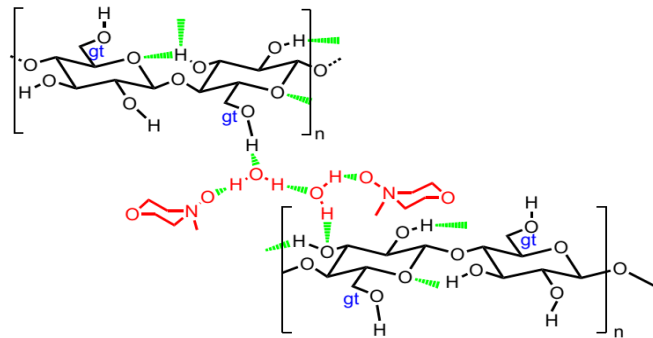
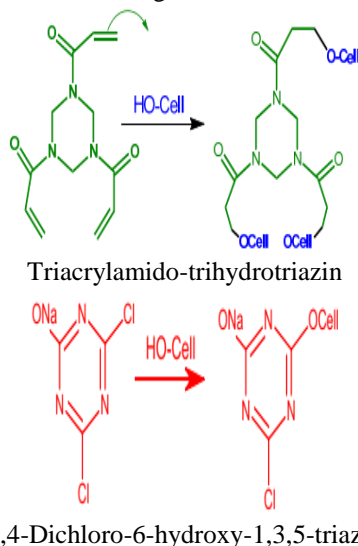


Figure 2 Molecular structure of TENCEL [19,20].

Types of TENCEL

TENCEL STD (A100): is standard TENCEL which produced from cellulose wood pulp.

TENCEL LF: is TENCEL STD after treatment against cross-linking as shown in Fig. 3.



2,4-Dichloro-6-hydroxy-1,3,5-triazin

Figure 3. TENCEL STD after treatment against cross-linking (TENCEL LF) [19].

Preparing of TENCEL

TENCEL coming from Wood pulp which contains of 40% -50% Cellulose [14]. In the manufacture process Wood

pulp mixed with NMMO (N-methylmorpholine-N-oxide) hydrate ($C_5H_{11}N_2O$) (3) 76 - 78% and water in temperature {70-90} as shown in Fig. 4 [8, 21].

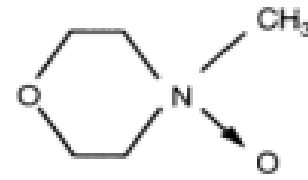


Figure 4. Molecular NMMO [8].

The water boil and turn to evaporate When the amount of water has reached a certain level the cellulose is dissolved [1, 21] in temperature {90 -120 c} and high pressure [8,21,22]. This solution is then filtered and extruded through a spinneret, tiny holes on a plate, with high pressure and high viscosity into a spinning bath which contains an aqueous solution of NMMO [1, 21]. Must be processed in similar high-pressure equipment to that used in melt polymer systems. The fibers are formed by spinning into an air gap and then coagulating in a water/ amine oxide bath. They are then washed, dried and cut. The wash liquors are recovered, purified, concentrated then recycled as shown in Fig. 5 [21,22].

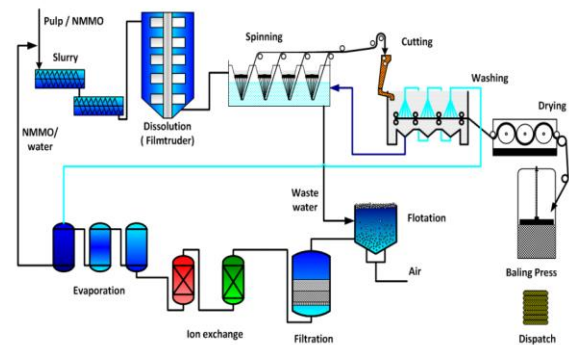


Figure 5. Preparation of TENCEL.

Nano-finishes

With the rapid development of modern social culture and economy, there is a great need to extend the functionality and performance of textiles to meet different demands for different people. The maintenance and improvement of current properties and the creation of new material properties are the most important reasons for the fictionalization of textiles. Recently, the advent of smart Nano-textiles has revolutionized clothing, home furnishings, and the materials used in industry [23].

These can be defined as methods of changing properties of textile goods by application of very small particles of finishing agents or by modification of very thin layers on the surfaces of textiles. The particles consist of many metal oxides whose chemical and physical characteristics make them useful for special finishes for the textile industry. Nanoparticles (10^{-9} m) are obtained by sol-gel synthesis in water or organic solvents. Nanoparticles possess a high surface area to weight ratio and this gives them some considerably enhanced properties [24].

1- Materials and Methods

1-1 Fabric

Desized, scoured and bleached TENCEL/cotton fabric was used. the fabric was supplied by Beshier company for engineering and commerce, , Tenth city of Ramadan in Egypt. TENCEL fabric imported from (Lenzing group) Austria.

1-2 Reagents

Monochloroacetic acid, sodium hydroxide, sodium carbonate, copper sulphate, silver nitrate, were laboratory grade chemicals. From Chem Tec in Kafer El shaik.

1-3 Procedures

Preparation of partially carboxy methylated TENCEL/Cotton (PCMTC).

Partial carboxy methylation of TENCEL/Cotton fabric was performed by using monochloroacetic acid solution which was prepared and neutralized with the equivalent amount of sodium carbonate samples of the TENCEL/Cotton were padded in this solution to a wet pickup of a 100% followed by air drying.

The samples were then padded in aqueous solution of sodium hydroxide (25%) to a wet pickup of ca 130% rolled and batched for 4 hours in polyethylene cover. The treated samples were then unrolled, washed with water, neutralized with dilute hydrochloric acid (0.1 N), washed with water again and finally dried under ambient condition. Samples containing different carboxyl content were obtained by varying the monochloroacetic acid concentration from 0.5 to 3 N.

Treatment of partially carboxy methylated TENCEL/cotton (PCMTC) as follows.

- The treatments were carried out by padding the fabric sample through two dips and two nips in solution containing CuSO_4 (10 g/l) to a wet pickup of ca.70%. the fabric was dried at 70°C for 5 minutes.

- The treatments were carried out by padding the fabric sample through two dips and two nips in solution containing AgNO_3 (10 g/l) to a wet pickup of ca.70%. the fabric was dried at 70°C for 5 minutes.

-washed in bath containing 5m/L soap for 15 min . The samples were then thoroughly washed and dried the samples were then conditioned at 65% relative humidity and 25°C for 48h before testing and analysis. [25].

Microbiological examination

Antimicrobial activity

A Zone of Inhibition Test, also called a Kirby-Bauer Test, is a qualitative method. A glass petri dish (90 mm diameter) containing the appropriate solidified medium was inoculated with 100 ul of a physiological solution containing 10^5 colony forming units (CFU/ml) of the microorganisms under study. Different concentration of both monochloroacetic acid and sodium carbonate (from 0.5 to 3 mol/l) and added 5 mm sterile column disc, placed on top of the cultural media. After incubation at optimal condition (temperature and time), the average diameter of free zone (where no growth of microorganisms was measured. All analyses were carried out in triplicate).

The tested microorganisms causing undesirable change on textile samples. The selected microorganisms were (*S.entridis*, *B. cereus*, *Staph.aureus*, *penicillium*, *Aspergillus*)

Measuring anti bacteria and antifungal

The resistance of samples against bacteria and fungi was measured after being allowed against microbes in the Microbiology Laboratory of the Faculty of Specific Education, Fayoum The resistance of samples against bacteria

and fungi was measured after being allowed against microbes in the Microbiology Laboratory of the Faculty of Specific Education, Fayoum University

Result and discussion

.The following tables 1,2 showing different concentrate and antimicrobial activity after clear zone test.

Table 1. Concentrate both Monochloroacetic acid and Sodium carbonate.

Concentrate mol/liter (N)	Weight monochloroacetic acid	Weight Sodium carbonate (Na_2CO_3)
0.5 mol/l	47g/l	53g/l
1 mol/l	94g/l	106g/l
1.5 mol/l	141g/l	159g/l
2 mol/l	188g/l	212g/l
2.5 mol/l	235g/l	265g/l
3 mol/l	282g/l	318g/l

The samples padded in aqueous solution of sodium hydroxide(25%) wet pickup 130%, batched for 4 h, neutralized with HCL.

Total inhibition or clear zone means no growth of microorganism. An inhibition of 90mm represents total inhibition (Petri dish diameter). Data followed by different letters in the same row are statistical significantly different.

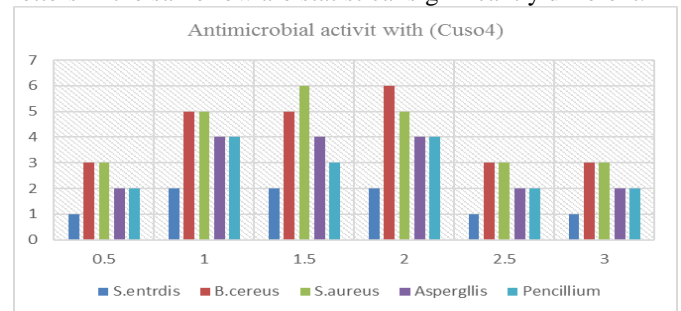


Chart (1) antimicrobial activity with (CuSO4)

From table (1): Tencil/cotton textile was treated with different concentrations of monochloroacetic acid and sodium carbonate from (0.5 to 3 mol/l) to replace (OH) groups with (COOH) groups in monochloroacetic acid for the preparation of samples by binding to antimicrobial substances (copper sulfate).

From Table (2) and chart (1): the clear zone shows different results for resistance to bacteria and fungi.

The control sample scored (0d ul / 100 ml). It showed a significant decrease in the results compared to the other samples where contamination occurred. Moreover,. Gram-positive bacteria gradual increase in the clear zone at concentrations from (0.5.1 mol / liter), to (3 mol / l).

The best results were recorded in this study Gram-negative bacteria *S.entridis*, showed a significant increase in the clear zone at concentrations (2 mol / liter), Gram-positive bacteria (*B. cereus*) showed (3b,5a and 5a ul/100 ml) a gradual increase in the clear region at concentrations of (0.5,1and 1.5 mol / L), respectively. A rise occurred again at a concentration of (2 mol / liter) showed that (6a ul/100 ml) with an increase Concentration, but then there was a sudden and steady decrease in the resistance of bacteria at high concentrations (2.5, 3 mol / L) (3b and 3b ul/100 ml)

Table 2): Partially carboxy methylated antimicrobial activities of TENCEL/Cotton fabrics containing CUSO4 against microorganisms.

Bacteria		control	Antimicrobial activity of CU_2SO_4 (10g/l water)					
			0.5 mol/l	1 mol/l	1.5 mol/l	2 mol/l	2.5 mol/l	3 mol/l
Gram negative	<i>S.entridis</i>	0 ^d	1 ^c	2 ^c	2 ^c	2 ^c	1 ^c	1 ^c
	<i>B.cereus</i>	0 ^d	3 ^b	5 ^a	5 ^a	6 ^a	3 ^b	3 ^b
Gram positive	<i>S.aureus</i>	0 ^d	3 ^b	5 ^a	6 ^a	5 ^a	3 ^b	3 ^b
	<i>Aspergillus</i>	0 ^d	2 ^c	4 ^b	4 ^b	4 ^b	2 ^c	2 ^c
Fungi	<i>Aspergillus</i>	0 ^d	2 ^c	4 ^b	4 ^b	4 ^b	2 ^c	2 ^c
	<i>Penicillium</i>	0 ^d	2 ^c	4 ^b	3 ^b	4 ^b	2 ^c	2 ^c

respectively. Gram-positive bacteria (*S.aureus*) showed the highest recorded (6a ul/100 ml) at concentration of (1.5 mol / L) It showed a significant decrease in results compared to the (2.5 and 3 mol / L) concentration, Where registered (3b ul/100 ml) respectively.

On the other hand it was recorded Gram-negative bacteria (*S.entridis*) showed a gradual increase then a stabilization occurred in the clear zone at concentrations (1, 1.5 and 2 mol / l), but then there was a sudden and steady decrease in the resistance of bacteria at high concentrations (2.5, 3 mol / l) Where registered (2c,2c,2c,1c and 1c ul/100 ml) respectively.

Penicillium showed a significant decrease in the different concentrations which recorded (2c, 3b, 4b, 5b, 3b and 3b ul/100 ml) than Aspergill which recorded (2c, 4b, 4b, 6a, 3b and 3b ul/100 ml).

Aspergill fungus the table did not show an increase in the resistance of samples with a different concentrations. Moreover, it showed significant stabilization of the resistance of samples to the fungus at concentrations (1, 1.5 and 2.5,3 ml/l) which recorded (4b,4b, 3b, and 3b ul/100 ml) respectively.

- Penicillium fungus the results showed a fluctuation in the resistance of the samples to the fungus, as the resistance increased at a concentration of (0.5,1 mol/l), then it decreased at a concentration (1.5 mol / l), then the resistance increased again at a concentration (2 mol/l), then the resistance decreased with a rise Concentration and stability occurred at (2.5,3 mol/l) concentrations.

Total inhibition or clear zone means no growth of microorganism. An inhibition of 90 mm represents total inhibition (Petri dish diameter). Data followed by different letters in the same row are statistical significantly different.

From table (1): Tencel / cotton textile was treated with different concentrations of monochloroacetic acid and sodium carbonate from (0.5 to 3 mol / l) to replace (OH) groups with(COOH) groups in monochloroacetic acid for the preparation of samples by binding to antimicrobial substances (copper sulphate). From Table (2) and chart (1): the clear zone shows different results for resistance to bacteria and fungi.

From Table (3) and chart (2): The resistance of samples to Gram-negative bacteria (*S.entridis*) showed a gradual increase in the clear zone at concentrations (0.5 and 1 mol / l) that recorded (2c and 2c ul/100 ml), then a stabilization occurred to a concentration of (2 mol / l) that recorded (3b ul/100 ml), but then there was a sudden and steady decrease

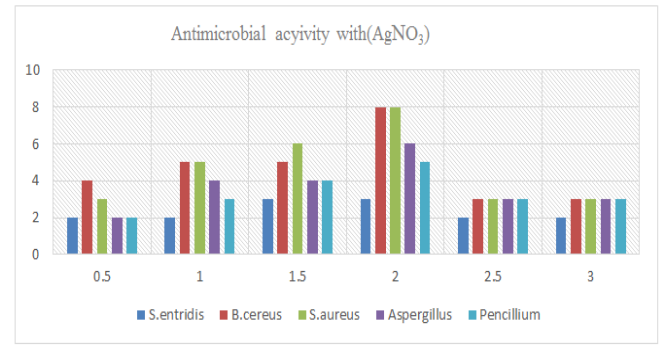


Chart 2. antimicrobial activity with (AgNO₃).

in the resistance of bacteria at high concentrations (2.5, 3 mol / l). On the other hand Gram-positive bacteria (*B.cereus*), a gradual increase in the resistance of bacteria was observed at a concentration of (0.5,1 and 1.5 mol / liter) that recorded (4b,5b,5b ul/100 ml) and a gradual decrease in the resistance of bacteria was observed at concentration (8a ul/100 ml) From (2 mol / l). As for the bacteria (*S.aureus*), there was a gradual increase in the resistance of bacteria at a concentration of (0.5,1 and 1.5 mol / l) that recorded (3b, 5b and 6a ul/100 ml) respectively, and a gradual decrease in the resistance of the bacteria was observed at a concentration of (2 mol / l) that recorded (8a ul/100 ml), then showed (3b and 3b ul/100 ml) where there was a stabilization of the resistance at a concentration of (2.5,3 mol / l).

Aspergillus fungus the table did not show resistance of samples with a different concentration. Moreover, it showed significant stabilization of the resistance of samples to the fungus at concentrations (1, 1.5 and 2.5,3 ml / l) which recorded (4b,4b, 3b, and 3b ul/100 ml) respectively. The results of the Penicillium fungus showed a fluctuation in the resistance to different concentrations, and a change in the results was recorded as the concentrations increased. - Penicillium showed a significant decrease in the different concentrations which recorded (2c, 3b, 4b, 5b, 3b and 3b ul/100 ml) than Aspergillus which recorded (2c, 4b, 4b, 6a, 3b and 3b ul/100 ml).

From table (4, 5) showed an increase in microbial growth on all samples as the concentration decreased, so it was noticed that the largest microbial growth was on the control sample, then the microbial growth decreased with an increase in concentration from (0.5, 1, 1.5, 2 mol/l), but the microbial growth increased again with the increase in concentration at concentration (2.5, 3 mol/l).

Table 3. Partially carboxy methylated antimicrobial activities of TENCEL/Cotton fabrics containing AgNO₃ against microorganisms.

Bacteria		Control	Antimicrobial activity of AgNO ₃ (10g/l water)					
			0.5	1	1.5	2	2.5	3
Gram negative	<i>S.entridis</i>	0 ^d	2 ^c	2 ^c	3 ^b	3 ^b	2 ^c	2 ^c
Gram positive	<i>B.cereus</i>	0 ^d	4 ^b	5 ^b	5 ^b	8 ^a	3 ^b	3 ^b
	<i>S.aureus</i>	0 ^d	3 ^b	5 ^b	6 ^a	8 ^a	3 ^b	3 ^b
Fungi	<i>Aspergillus</i>	0 ^d	2 ^c	4 ^b	4 ^b	6 ^a	3 ^b	3 ^b
	<i>Penicillium</i>	0 ^d	2 ^c	3 ^b	4 ^b	5 ^b	3 ^b	3 ^b

Table 4. Total microbiological counts of textile TENCEL/Cotton samples treatment by (Cu₂SO₄).

Microorganism	Control	Monochloroacetic acid concentration and sodium carbonate (mol/l water)					
		0.5	1	1.5	2	2.5	3
<i>S.entridis</i>	7.5x10 ^{3A}	5.5x10 ^{3A}	3.4x10 ^{3A}	3.2x10 ^{3A}	2.6x10 ^{3A}	3.0x10 ^{3A}	3.0x10 ^{3A}
<i>B.cereus</i>	6.3x10 ^{3A}	5.5x10 ^{3A}	3.2x10 ^{3A}	3.1x10 ^{3A}	2.3x10 ^{3A}	2.9x10 ^{3A}	2.9x10 ^{3A}
<i>S.aureus</i>	6.7x10 ^{3A}	3.3x10 ^{3a}	3.1x10 ^{3A}	2.9x10 ^{3A}	2.4x10 ^{3A}	2.8x10 ^{3A}	2.8x10 ^{3A}
<i>Aspergillus</i>	8.3x10 ^{3A}	3.1x10 ^{3A}	3.0x10 ^{3A}	2.4x10 ^{3A}	2.2x10 ^{3A}	2.6x10 ^{3A}	2.6x10 ^{3A}
<i>Penicillium</i>	7.3x10 ^{3A}	3.1x10 ^{3A}	2.9x10 ^{3A}	2.7x10 ^{3A}	2.1x10 ^{3A}	2.9x10 ^{3A}	2.9x10 ^{3A}

Table 5. Total microbiological counts of textile TENCEL/Cotton samples treatment by AgNO₃

Microorganism	Control	Monochloroacetic acid concentration and sodium carbonate (mol/l water)					
		0.5	1	1.5	2	2.5	3
S.entridis	7.5x10 ^{3A}	5.5x10 ^{3A}	3.1x10 ^{3A}	3.2x10 ^{3A}	2.4x10 ^{3A}	2.9x10 ^{3A}	3.0x10 ^{3A}
B.cereus	6.3x10 ^{3A}	5.5x10 ^{3A}	3.2x10 ^{3A}	3.0x10 ^{3A}	2.3x10 ^{3A}	2.6x10 ^{3A}	2.9x10 ^{3A}
S.aureus	6.7x10 ^{3A}	3.3x10 ^{3a}	3.0x10 ^{3A}	2.9x10 ^{3A}	2.2x10 ^{3A}	2.8x10 ^{3A}	2.8x10 ^{3A}
Aspergillus	8.3x10 ^{3A}	3.1x10 ^{3A}	2.9x10 ^{3A}	2.2x10 ^{3A}	1.8x10 ^{3A}	2.2x10 ^{3A}	2.4x10 ^{3A}
Penicillium	7.3x10 ^{3A}	3.1x10 ^{3A}	2.9x10 ^{3A}	2.2x10 ^{3A}	1.7x10 ^{3A}	2.1x10 ^{3A}	2.6x10 ^{3A}

Data followed by different letters in the row are statistical significantly different.

Conclusion

From the previous results, the study concluded that treating the samples with either copper sulphate or silver nitrate had an effect on tissue resistance against microbes in general, as the samples were given with Gram-negative bacteria (*S.entridis*) at a concentration of (2 mol/l) the highest resistance against bacteria the less bacterial growth, the higher the concentration, the more stable the resistance of bacteria occurs, and this indicates that the samples under study will not give higher resistance against bacteria, regardless of the concentration compared with The control sample scored (0d ul / 100 ml). , It showed a significant decrease in the results compared to the other samples where contamination occurred. The best results were recorded in this study Gram-negative bacteria And it showed a significant increase in the clear zone at concentrations (2 mol / l), Gram-positive bacteria (*B. cereus*) showed (3b,5a and 5a ul/100 ml) a gradual increase in the clear region at concentrations of (0.5,1and 1.5 mol / L), respectively. then a stabilization of bacterial resistance, and this indicates that no matter the concentration of the preparation materials on the samples under study, it will not give a higher resistance against bacteria and that this concentration is the best concentration of resistance. And this repeated in the samples with Gram-positive bacteria (*B.cereus*) beside the samples with *S.aureus*.

The samples infected with *Aspergillus* fungus gave the highest resistance at a concentration of (2 mol / l), then there was a decrease in the resistance of bacteria with the increase of the treatment materials and the stability of the resistance, and this indicates that it is preferable to use a concentration (2 mol / l) as it recorded the best results. In terms of Clear Zone and the microbial count test.

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