

# بسم الله الرحمن الرحيم



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شبكة المعلومات الجامعية التوثيق الالكتروني والميكرونيلم





# جامعة عين شمس

التوثيق الإلكتروني والميكروفيلم

# قسم

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# PRODUCTION OF SOME BIO-ACTIVE FIBERS

M. Sc. Thesis Submitted to

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# **ABSTRACT**

The present work aims at: (a) Producing of antibacterial and antialgae cotton and polyester/cotton blend fabrics carrying different percentages of copper or lead compounds, and (b) studying the antibacterial and antialgae capacity of the treated fabrics and the stability of such produced bioactive agents. These fabrics are looking forward to be used in production of water filters and antibacterial clothing. Water filters can be applied in the companies that used sea-water in cooling systems to inhibit the growth of bacteria and algae.

A bio-active cotton and polyester/cotton (50/50) blend fabrics were produced by impregnation in a reaction mixture containing Carboxy Methyl Starch (CMS) and Melamine Formaldehyde (MF). After this the samples squeezed, dried, washed, dried, immersed in water solutions of copper sulfate or lead acetate, dried, washed in water containing detergent, washed with fresh water and finally dried.

Factors affecting the amount of Cu<sup>2+</sup> or Pb<sup>2+</sup> on the treated fabrics were studied. The bactericidal and algicidal activities of treated fabrics were tested and the stability of both copper and lead on the fabrics was determined.

It was found that the percentage of both copper and lead ions increases with increasing the amount of MF/CMS reacted with the fabrics. On the other hand it was noticed that the higher increase in

weight due to reaction of (MF/CMS) mixture with the fabrics can be obtained when the percentage of MF was increased up till 30 % in the reaction mixture and the reaction of the fabrics with the MF/CMS was carried out at PH 5, reaction time of 60 min, and reaction temperature of 100 °C. The amount of Cu<sup>2+</sup> was also found to increase with increasing the concentration of copper sulfate solution up to 0.8 mole/l. Fabrics containing from 0.3 to 1.4 % Cu<sup>2+</sup> and from 1.2 to 4 % Pb<sup>2+</sup> were obtained.

The bio-activity of cotton fabrics contained Cu<sup>2+</sup> was excellent against many types of bacteria, where the inhibition zones were ranged between 7 and 25 mm. The fabrics contained Pb<sup>2+</sup> do not show any response towards the tested types of bacteria. On the other hand it was found that the algicidal of the fabrics contained Pb<sup>2+</sup> higher than that of the fabrics carrying Cu<sup>2+</sup>.

The fabrics contained either Cu<sup>2+</sup> or Pb<sup>2+</sup> show a high degree of stability of the metal ions, where the loss in both Cu<sup>2+</sup> and Pb<sup>2+</sup> that released in water was 1.6 % and 5.5 %, respectively, after 90 days.

# **SUMMARY**

In the last years a large amount of textile materials, produced from natural and man-made fibers, is used in many industrial areas as ion-exchange and antibacterial substances. These materials are obtained by chemical modification of textile fabrics to impart them such properties.

The use of modified textile materials, in the production of water filters and antibacterial clothing, is due to the following advantages:

- (a) the low cost of production of these fabrics, and
- (b) the slow release of active sites, i.e. the low pollution effect of these materials.

The present work aims at: (a) producing of antibacterial and antialgae cotton and polyester-cotton blend fabrics carrying different percentages of copper or lead compounds, and (b) studying the antibacterial and antialgae capacity of the treated fabrics, and the stability of such produced bio-active agents.

These fabrics are looking forward to be used in production of water filters and antibacterial clothing. Water filters can be applied in the companies that used sea water in cooling systems to inhibit the growth of bacteria and algae and to filtrate cooling water from any solid materials.

The thesis is subdivided into two main parts; namely general part and special part. A general part deals with literature survey on the production of bio-active fibers and fabrics by treatments with:

- i- Biocidal reactive dyes.
- ii- Water insoluble metal compounds (tin, lead, copper, zinc, mercury, silver ...... etc.).

iii- Chitosan compounds.

iv- Sulfur and phosphorus compounds.

v- Phenols and phenol derivatives.

vi- Quaternary ammonium salts.

vii- Other compounds.

A special part deals with the experimental work, results and discussion and conclusion.

A bio-active cotton and polyester/cotton (50/50 %) blend fabrics were produced by impregnation in a reaction mixture containing Carboxy Methyl Starch (CMS) and Melamine Formaldehyde (MF). Then the samples were squeezed, dried, washed, dried, immersed into water solutions of copper sulfate or lead acetate, dried, washed in water containing detergent, washed with fresh water and finally dried.

Factors affecting the amount of Cu<sup>2+</sup> or Pb<sup>2+</sup> on the treated fabrics were studied. The bactericidal and algicidal activities of treated fabrics were tested and the stability of both copper and lead on the fabrics was determined.

# FACTORS AFFECTING THE AMOUNT OF Cu<sup>2+</sup> OR Pb<sup>2+</sup> ON THE TREATED FABRICS

## 1. Effect of MF Concentration

Cotton fabrics were introduced into an aqueous solution of CMS containing different amounts of MF, thermal treated at  $100\,^{\circ}\text{C}$  for  $60\,\text{min}$ , immersed in aqueous solutions of  $\text{CuSO}_4$ .  $5\text{H}_2\text{O}$  or  $\text{Pb}(\text{CH}_3\text{COO})_2$ .  $3\text{H}_2\text{O}$ , washed and dried.

It was found that increasing the MF percentage up till 30 wt. % of CMS concentration increases the weight of the cotton fabric. The further increase of MF concentration leads to a decrease in the fabric weight. The same holds true for the content of Cu<sup>2+</sup> and Pb<sup>2+</sup> ions on the fabrics.

It was also found that the reaction of Cu<sup>2+</sup> ions with the treated fabric proceeds to a much greater extent than that which we calculated for the reaction between Cu<sup>2+</sup> and CMS. These results implies that beside the reaction of Cu<sup>2+</sup> with CMS, cupric ions seems to trap also in polymeric network of melamine formaldehyde leading to complex formation.

### 2. Effect of PH of the Reaction Medium

The acidity of the medium was controlled by ortho-phosphoric acid (10 %) and was studied in the range of PH value from 4.0 to 9.0, keeping other conditions constant. As it is evident the weight of the fabric increases as the PH of the reaction medium increases up till 5.0. Above PH 5.5 the weight of the fabric decreases. Stated in words, PH 5.0 constitutes the optimal PH for the reaction between MF/CMS mixture and cotton fabric.

The percentages of copper attached on the fabrics follow the same trend of the increase in fabric weight as indicated above.

### 3. Effect of Squeezing Degree

The degree of squeeze was changed by applying different inter-roll distances (S = 0.03, 0.25, and 0.50 mm). It was found that when the inter-roll distance increases from 0.03 to 0.50 mm the percentage of the cotton fabric weight increase due to reaction of the MF/CMS mixture with the fabric

changes from 0.86 % to 4.2 %. The maximum value of the fabric weight increase was attained for the unsqueezed cotton fabric specimen which was 8.6 %. The percentages of copper and lead on the fabric follows the increase of the fabric weight. When the increase in weight of the cotton fabric changes from 0.86 % to 8.6 %, the percentage of Cu<sup>2+</sup> increases from 0.54 to 1.31 % and lead increases from 1.8 to 3.7 %.

The effect of the degree of squeeze (inter-roll distance) on the fabric weight increase and the percentages of copper and lead on the polyester/cotton (50/50 %) blend fabric, was also studied.

### 4. Effect of the Reaction Temperature

The reaction of MF/CMS mixture with the cotton fabric was carried out at five temperatures in the range 60 - 100 °C, keeping all other conditions constant. Obviously, the fabric weight and the content of Cu and Pb increase as the reaction temperature increases from 60 to 100 °C. Nevertheless, at 60 °C, 70 °C and 80 °C the reaction of MF/CMS mixture with cotton fabric proceeds initially (during first 15 min.) very slowly then becomes faster during the later stages of the reaction. At 90 °C, on the other hand, through the initial rate of reaction is higher than that at 80 °C, yet the maximum increase in weight obtained at 100 °C is much higher than at 90 °C.

## 5- Effect of Reaction Time

It was found that both the fabric weight and the content of copper and lead on the fabric increase as the reaction time increases. Reaction time of 60 min. constitutes the optimal duration to attain the higher value of the fabric weight increase.

#### 6- Effect of Copper Sulfate Concentration

The effect of copper sulfate concentration on the percentage of copper reacted with the cotton fabric treated with MF/CMS mixture was studied. It was found that the increase in copper sulfate concentration from 0.08 to 0.79 mole/l is accompanied by a substantial increase in the percentage of copper reacted with cotton fabric. The further increase of copper sulfate concentration up till 1.10 mole/l did not cause any significant increase in the copper content on the fabric.

# BIO-ACTIVITY AND STABILITY OF FABRICS <u>CARRYING COPPER OR LEAD</u>

### I. Bio-activity of Fabrics Carrying Copper or Lead

#### I. 1. Bactericidal Activity

Cotton fabrics containing different percentages of copper (Cu % = 0.0,0.48, 0.67, 0.79, 0.85 and 1.38) and lead (Pb % = 0.0, 1.5, 2.2, 2.36, 2.76 and 3.9) were used for the bactericidal tests. These fabrics were tested against different types of bacteria such as *Escherichia coli*, *Pseudomonas aeruginosa*, *Bacillus subtilis*, *Staphylococcus aureus*, *Candida albicans* and *Escherichia coli* 0157.

It was found that there is no effect for cotton fabric which contains lead even at maximum value (3.9 %) of lead on the fabric.

Cotton fabrics treated with copper showed significant effect against many types of bacteria. The highest effect was observed against *Bacillus* 

subtilis and Candida albicans which affected even by the fabric containing the minimum value (0.48 %) of copper which produced inhibition zones of 15 mm for Bacillus subtilis and 7 mm for Candida albicans. The diameter of inhibition zone increased with increasing the copper concentration on the fabric. It measured 25 mm at copper value of 1.38 %. Significant effect for Escherichia coli and Escherichia coli 0157 was observed at copper concentrations higher than 0.67 %. Pseudomonas aeruginosa was not affected at all by the copper treated fabric even at the maximum value of copper (1.38 %).

#### I. 2. Algicidal Activity

Algicidal test (diffusion test) of the cotton fabric containing different percentages of copper or lead using sea- water, which already contains algae, were carried out. A standard membrane of pore size 0.45 µm (through which any algae could not pass) was used as a test specimen for sea-water to show the algicidal efficiency of the treated fabric related to this membrane. It was found that there is a little increase in the algicidal efficiency of the fabric (from 37.08 % to 41.05 %) with increasing the copper content (from 0.48 % to 1.38 %).

Cotton fabrics containing lead showed a higher algicidal efficiency than that containing copper. The cotton fabrics containing 1.50 % Pb showed algicidal efficiency of 33.0 %. This value increases gradually to reach the highest inhibitory action (47.30 %) at the maximum lead content on the fabric (3.90 %).