

# A Study of Plasma-Assisted Rechargeable Antimicrobial Finishing for Cotton

C. W. Kan <sup>1+</sup>, C. E. Zhou <sup>1</sup>, A. Luximon <sup>1</sup>, K. W. Lau <sup>1</sup> and K. S. Chen <sup>2</sup>

<sup>1</sup> Institute of Textiles and Clothing, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong

<sup>2</sup> Department of Materials Engineering, Tatung University, 40 Zhongshan North Road, 3rd Section Taipei 104, Taiwan

**Abstract.** In this paper, oxygen plasma treatment was applied in different application stages of finishing process of cotton fabrics of coating with 5,5-dimethylhydantoin (DMH). The aim of plasma treatment is to improve and assist the DMH coating. Chlorine was then introduced on the DMH coated cotton fabrics in order to make it with antimicrobial property by chlorination with sodium hypochlorite. The antimicrobial property of the coated cotton fabrics, its rechargeability and stability were investigated. The results showed that oxygen-plasma could assist the adhesion of DMH on fabric and then the successful introduction of chlorine on the coated fabric could inhibit growth of bacteria and the antimicrobial property is regenerable. Therefore, the coating of cotton fabrics with DMH with the assistance of oxygen-plasma treatment, plus chlorination with sodium hypochlorite achieves good effect in enhancing the antimicrobial property.

**Keywords:** Plasma, rechargeable, antimicrobial, cotton

## 1. Introduction

Cotton is a commonly used natural cellulose fiber consisting of  $\beta$ -1, 4-D-glucose which has contain large amount of hydroxyl ( $-OH$ ) groups and a small number of carboxylic acid groups. Therefore, the good moisture absorption ability leads to moist cotton easier to be attacked by bacteria. A number of chemicals were used to impart antimicrobial activity to cotton fabrics. However, antimicrobial property of cotton fabrics finished with those chemicals is not regenerable. N-halamines are the antimicrobial agents which make the antimicrobial property of fabrics regenerable. The bactericidal process consumes halogens, and the lost halogens can be easily recharged by chlorination. 5,5-dimethylhydantoin (DMH) is a basic finishing agent which can be transferred into antimicrobial agent with N-halamine structure [1]. After finishing and chlorination, the antimicrobial materials could kill a broad range spectrum of microbial [2]-[5].

Plasma techniques are a suitable tool to assist the generation of the demanded modification, which can activate the surface of fabric and improve the adhesion of chemicals on fabrics. In this study, DMH was coated onto cotton fabrics with the aid of plasma treatment. After chlorination, N-halamine structure was introduced into the coated cotton fabrics. In order to investigate the effect of plasma treatment on the antimicrobial activity of coated fabrics, plasma treatment was applied to the finishing process in different stage.

## 2. Experimental

### 2.1. Materials

100% well pretreated woven cotton fabric was supplied by Lai Tak Enterprises Ltd. Oxygen and helium

---

<sup>+</sup> Corresponding author. Tel.: +852 27666531; fax: +852 27731432.  
E-mail address: tccwk@polyu.edu.hk.

gases were supplied by Linde HKO Ltd. 5,5-Dimethyl hydantoin (DMH) (97%), sodium hypochlorite (5%, activated chloride), potassium iodide, and starch indicator (1% in H<sub>2</sub>O) were purchased from Sigma-aldrich.

## 2.2. Plasma Treatment

Plasma treatment was carried out with an atmospheric pressure plasma jet system (APPJ, Surfx Technologies LLC, CA) with a rectangular nozzle. The plasma was generated by radio frequency of 13.56MHz. The carrier gas was helium, while the reactive gas was oxygen. The discharge power of plasma treatment is 80W, the flow rate of oxygen is 0.1L/min, the flow rate of helium is 10.0L/min, jet distance is 5mm and the moving speed of fabric is 0.2m/s. The experimental set-up of plasma treatment was shown in Fig. 1.

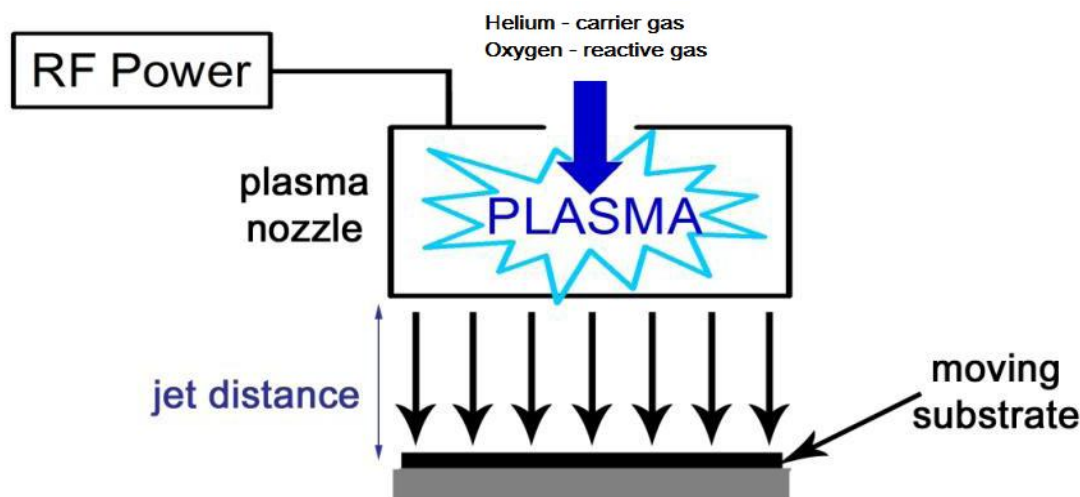


Fig. 1: Schematic diagram of APP treatment

## 2.3. Applying DMH to Cotton Fabric

4% of finishing bath (DMH) were used to finish cotton fabrics. There were four processes for coating cotton fabric with DMH, i.e. (i) plasma treatment-pad-dry-cure (CPC), (ii) pad-plasma treatment-dry-cure (CWPC), (iii) pad-dry-plasma treatment-cure (CDPC), and (iv) pad-dry-cure (CC). The fabrics were dried at 80 °C for about 5 minutes and cured at 160 °C for minutes.

## 2.4. Chlorination

The DMH coated fabrics were chlorinated at room temperature with 0.8% of sodium hypochlorite solution to transform some of the amino groups in DMH into N-halamines.

## 2.5. Antimicrobial Activity

Antimicrobial activity of samples was tested according to AATCC Test Method 147-2011 with using *S. aureus* (ATCC 6538) and *K. pneumoniae* (ATCC 4352).

## 2.6. Rechargeability

Chlorinated cotton fabrics were washed to test their rechargeability according to AATCC Test Method 61-1A. Active chlorine content of chlorinated fabrics before washing (BW), after washing (AW), and after re-chlorination(AW+CH) was tested and compared. The active chlorine content of the chlorinated cotton fabric coated with DMH was determined by colorimetric method.

# 3. Results and Discussion

## 3.1. Antimicrobial Activity

The antimicrobial efficacy test for chlorinated cotton fabrics coated with DMH against *S. aureus* (ATCC 6538) and *K. pneumoniae* (ATCC 4352) is illustrated in Fig. 2. The growth of microorganism underneath the tested specimen was observed and the width of the clear zone was measured. The antimicrobial efficacy data are presented in Table I.

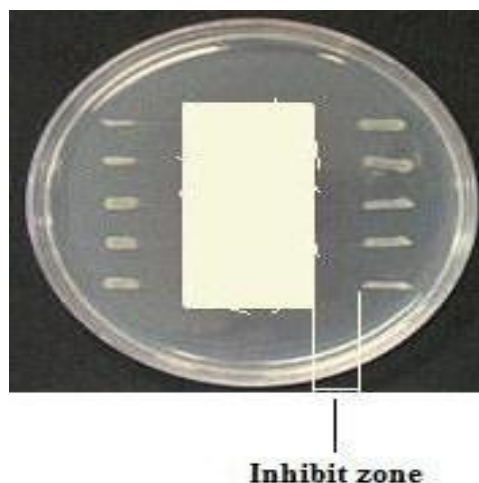


Fig. 2: Example of the test of antimicrobial activity

Table 1: The Mean Clearance Distance of the Bacteria Obtained from the Specimens

Treatment process	Mean Clear Width against <i>S. aureus</i> (cm)	Mean Clear Width against <i>K. pneumonia</i> (cm)
Untreated	0	0
CPC	1.2	1.3
CDPC	1.2	1.2
CWPC	1.3	1.4
CC	0.9	0.9

The growth of *S. aureus* (ATCC 6538) and *K. pneumoniae* (ATCC 4352) underneath the tested specimen was not observed except in the sample without DMH coating. That is to say, cotton fabrics coated with DMH can inhibit the growth of *S. aureus* (ATCC 6538) and *K. pneumoniae* (ATCC 4352) effectively. From the comparison of the distance between the clear zones, it can find that the biocidal efficacy of cotton fabrics coated with DMH with assistance of plasma treatment is better than samples coated with DMH without plasma treatment, especially when fabrics are treated with plasma after padding. Therefore, it can be concluded that plasma treatment following padding is the best process for cotton fabrics coated with DMH among all of these three processes.

### 3.2. Rechargeability

Rechargeability and is very important for antimicrobial textiles. Table II shows the rechargeability property of cotton fabrics coated with DMH with the aid of plasma treatment.

It can be seen in Table II that almost all of samples coated with DMH with the aid of plasma treatment can be recharged after washing. However, the active chlorine concentration on cotton fabrics after they were re-chlorinated is a little lower than they were on cotton fabrics before washing. That is because the adhesion of DMH on cotton fabrics may not good, DMH on cotton fabrics can be easily removed in water medium such as washing and even in bleaching solution. Compared the cotton fabrics treated with different processes, it also can find that the recharging effect of cotton fabrics treated with plasma is better than others, especially fabrics finished with plasma treatment after padding. That may be caused by the existence of water in the working environment reacting with the unstable particles generated by plasma. Therefore, it can be concluded that plasma treatment is helpful for improving the rechargeability of cotton fabrics coated with DMH and plasma treatment after padding can enhance the adhesion of DMH on cotton fabrics.

Table 2: The Rechargeability of Cotton Fabric Coated with DMH with Plasma Treatment

Treatment process	Concentration of active chlorine ( $\times 10^{-7}$ ) mol/L		
	BW	AW	AW+CH
CPC	6.0	5.6	5.5
CDPC	5.7	5.5	5.3
CWPC	6.7	5.7	5.6
CC	5.4	4.9	4.8

## 4. Conclusion

Rechargeable antibacterial functions can be chemically imparted onto cotton fabrics by coating with DMH, followed by chlorine bleaching. The rechargeable antibacterial properties can be improved by oxygen plasma treatment, especially plasma treatment following padding. The new system provides rechargeable antimicrobial functions against both Gram-positive bacteria, *S. aureus* (ATCC 6538) and gram-negative bacteria *K. pneumoniae* (ATCC 4352).

## 5. Acknowledgements

Authors would like to thank the financial support from the Research Grants Council of The Hong Kong Special Administrative Region, China (Project No. PolyU 5173/11E) and The Hong Kong Polytechnic University for this work.

## 6. References

- [1] L. Qian, and G. Sun. Durable and regenerable antimicrobial textiles: Improving efficacy and durability of biocidal functions. *Journal of Applied Polymer Science*. 2004, **91**(4): 2588-2593.
- [2] N. Abidi and E. Hequet. Cotton fabric graft copolymerization using microwave plasma. II. Physical properties. *Journal of Applied Polymer Science*. 2005, **98**(2): 896-902.
- [3] L. K. Huang and G. Sun. Durable and oxygen bleach rechargeable antimicrobial cellulose: Sodium perborate as an activating and recharging agent. *Industrial & Engineering Chemistry Research*. 2003, **42**(22): 5417-5422.
- [4] N. L. Lala, R. Ramaseshan, L. Bojun, S. Sundarrajan, R. Barhate, Y. J. Liu, and S. Ramakrishna. Fabrication of nanofibers with antimicrobial functionality used as filters: protection against bacterial contaminants. *Biotechnology and bioengineering*. 2007, **97**(6): 1357-1365.
- [5] X. Ren, L. Kou, H. B. Kocker, C. Zhu, S. Worley, R. Broughton, T. Huang. Antimicrobial coating of an N-halamine biocidal monomer on cotton fibers via admicellar polymerization. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*. 2008, 317(1): 711-716.