

Preparation and Characteristics of a New Antibacterial Cotton Fiber Bonding a Gemini-type Quaternary Ammonium Salt

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Abstract: Two kinds of antibacterial cotton fiber (called as C₁₄ and C_t) were prepared by bonding a gemini-type quaternary ammonium compound with a hydroxyl group in their spacer chain [1,5-bis(tetradecyl dimethyl ammonium iodide)-3-pentanol (called as DAP₁₄)] and a mono-type quaternary ammonium compound [*N*-tetradecyldimethyl (3-trimethoxysilylpropyl) ammonium chloride (called as TMMP)], respectively, to the surface of cotton fiber using a coupling agent, tetraethoxysilane. Antibacterial characteristic of C₁₄ was investigated comparatively with C_t. The experimental results indicated that C₁₄ has higher antimicrobial activity than C_t against a broad range of microorganism, and average net bactericidal ratio against 15 strains of gram-negative, gram-positive bacteria and yeasts is 98.0% at 30°C and pH 6.0, which are 25.2 and 17.2 percentage point higher than those of C_t at treated time of 30min and 60min, respectively. Antibacterial activity of C₁₄ was hardly influenced by environmental pH and temperature, and could not be removed from the surfaces of cotton fiber by repeated washing with water.

Keywords: quaternary ammonium salt, coupling, cotton fibers, anti-bacterial activity, washability, gemini

1. Introduction

In spite of advances in control technique to harmful microorganisms, those resulting from methicillin-resistant *S. aureus* (MRSA) are still clinically significant problem [1,2,3]. Microbes growing on the fibre surface may be harmful to skin and fibre, which not only damage these fibre materials but also result in skin inflammatory reaction and infections by destroying ecological balance of indigenous flora on the skin surface. Recently, fiber products treated for providing the antimicrobial property has been actively developed and accepted for use in clinical setting and clothing materials to block the transmission of pathogens [4,5,6]. A product which exhibits a broad antimicrobial spectrum and can still remain the antimicrobial property under severe washing conditions is desired.

Quaternary ammonium compounds (QACs), which were adsorbed on the surface of fiber, are widely applied in antimicrobial finishing for textiles to endow them with additional surface-active and

antifouling properties [7,8]. Those early products show marked antibacterial function, but can be desorbed during further washing.

Cotton fiber consist mainly of cellulose, which is linear chain of several hundreds to over nine thousand $\beta(1\rightarrow4)$ linked D-glucose units [9]. The hydroxyl groups of glucose in cellulose molecular can be partially or fully reacted with various reagents to afford derivatives with useful properties. Since Isquith et al. linked a quaternary ammonium salt, 3-(trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride, to cotton cloth [10], immobilizing sterilization technology was developed vigorously [11]. However, immobilized mono-type QACs have not a high performance because of its defects such as low sensitivity to gram-negative bacteria, instability to temperature and pH, and so on.

It is known that most of the mono-type QACs have a lower antifungal activity, although they

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are effective as bactericide, but they do not have a excellent balance between against gram-negative and gram-positive bacteria [12,13,14,15]. Due to long-term and broad-range to be used, microbial resistance phenomena against QACs were reported about conventional mono-type QACs [16]. On the basis of our previous studies[17,21], gemini-type QACs, whether immobilized on carries or free molecular, has a higher antibacterial activity than mono-type QACs, so it is necessary that new antimicrobial finishing processes are developed with more effective compounds, such as gemini-type QACs.

The present paper reports the immobilization of a new genimi-type QAC, 1,5-bis(tetradecyl dimethyl ammonium iodide)-3- pentanol (called as DAP₁₄) on cotton fiber and its antimicrobial characteristics.

2. Materials and Methods

2.1 Chemicals and Materials

Bromophenol blue (BPB) was purchased from Kanto Chemicals Co., Inc. Tetraethoxysilane was from Tokyo Kasei Kogyo Co., Ltd. *N*-Tetradecyldimethyl(3-trimethoxysilylpropyl) ammonium chloride(TMMP) was supplied by Shin-Etsu Chemical Co., Ltd. DAP₁₄ were synthesized at our laboratory according to the previous report [17]. For simplification of experiments, degreased cotton, obtained commercially from Xu Kang Sanitation Material Co., Ltd., was used instead of general cotton.

2.2 Coupling of DAP₁₄ and TMMP to Cotton Fiber

Immobilization of DAP₁₄ on cotton fiber is shown in Figure 1

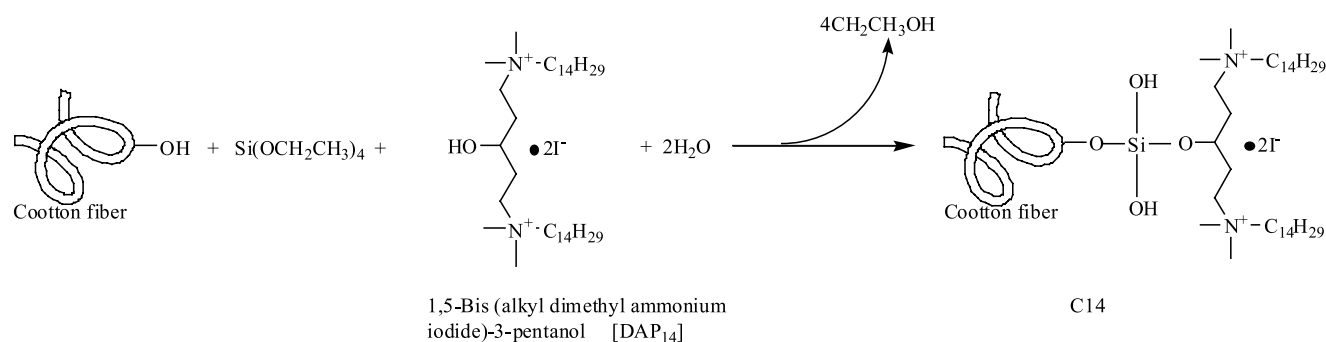


Figure 1 Immobilization of DAP₁₄

0.5g Cotton fiber was washed for 3h at 40°C with 100 ml ether to remove the lipid on its surface, and then dried for 1h under reduced pressure. Degreased cotton fiber was soaked in 50 ml 2% tetraethoxysilane in ethanol/water mixture [ethanol : water = 95 : 5 (v/v), pH 4.5~5.5 adjusted with acetic acid]. After heating for 2h at 70 °C and cooling down to the room temperature, the cotton fiber was washed three times with methanol and then dried in vacuum.

A mixture of 50 ml ethanol, 0.2 mmol DAP₁₄ and 0.5 g silylated cotton fiber was heated at 60°C for 10 min, and 10 ml distilled water was added. After the mixture was kept at 60°C for 3h with stirring,

then solvent was removed by air pump filtration. The treated cotton fiber above was heated at 70°C for 1 h, and then washed with distilled water until the supernatant turned colorless and transparent, and finally dried at room temperature for 24 h under reduced pressure.

50.0 ml Ethanol and 1.0 ml TMMP were added into 0.1g degreased cotton fiber successively, and then concentrated to 30.0 ml. 5.0 ml of distilled water was added to perform a coupling reaction for 1h at 60°C. Then solvent was filtered and cotton fiber was heated at 80°C for 1 h. Finally, the cotton fiber was washed with methanol until supernatant looks