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Antimicrobial and chemical detoxifying functions of cotton fabrics containing different benzophenone derivatives

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Abstract

Several benzophenone chromophoric groups were incorporated onto cotton fabrics by using 4-hydroxybenzophenone, 4,4′-dihydroxybenzophenone, 4-chloro-4′-hydroxybenzophenone, and 4-benzoylbenzoic acid as reagents. The fabric treatment was conducted by a pad-dry-cure method, and the benzophenone chromophoric group incorporated cotton fabrics were characterized and confirmed by FTIR. Tensile strengths of benzophenone chromophoric groups modified cotton fabrics were also measured. 4-Hydroxybenzophenone treated cotton fabric showed the most powerful antibacterial activity among all samples, and 4-benzoylbenzoic acid treated cotton fabric demonstrated pesticide degradation ability, under UV irradiation.

Keywords: Protective clothing material; Radical; Cotton; Anti-bacterial fabrics; Pesticide degradation; Benzophenone

1. Introduction

Various environmental hazards such as pathogenic bacteria, toxic pesticides, and pollutants, coupled with growing threats of biological and chemical attacks from terrorists, have inspired scientists to develop countermeasures (Singh, Lee, & Dressick, 2004). More importantly, protection of human body from invasion of toxic chemicals and pathogens is an urgent research subject, and development of biological and chemical protective clothing materials is one of the important solutions. Current protective garments are based upon: (1) removal of environmental toxins using efficient adsorbent material, (2) use of impermeable barriers to protect targets from toxins, and (3) inactivation of bacteria on the surface of the treated garment. These approaches are effective at certain level, but also bear some shortfalls, such as high cost, impaired user performance due to the weight and bulkiness, and environmental concerns (Horrocks & Anand, 2000; Schreuder-Gibson et al., 2003). Therefore, there is an urgent need to develop systems that are more energetic in neutralizing biological agents, chemical pollutants and toxins while environmentally friendly (Singh et al., 2004).

In fact, radicals could be a potential agent for self-decontamination applications. Radicals tend to undergo fast reactions upon creation, and they are highly reactive chemical species possessing at least one unpaired electron (Kochi, 1973). Many chemical processes involve the use of radical reactions, such as the biological reduction of O_2 by antioxidants, radical addition to unsaturated molecules, and radical polymerization processes. In particular, benzophenone chromophoric groups, known to undergo light-excitation from n to π^* triplet states, are commonly used as photosensitizers in photochemistry. The triplet can be readily quenched by oxygen, and can also react with a suitable hydrogen donor to form a ketyl radical. Therefore, the fabrics containing benzophenone chromophoric group could be easily excited to the very active radical states which can not only interact (kill) microorganisms but also react (decompose) toxic compounds, such as pesticides, when they are exposed to UV light.

In this study, different benzophenone chromophoric groups were incorporated onto cotton fabrics treated by

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4-hydroxybenzophenone, 4,4′-dihydroxybenzophenone, 4-chloro-4′-hydroxybenzophenone and 4-benzoylbenzoic acid, and via a pad-dry-cure method. The structures of the modified cotton fabrics were confirmed by using FTIR, and antimicrobial and chemical detoxifying properties of the fabrics were evaluated following different protocols.

2. Experimental

2.1. Materials

Cotton fabrics (no. 400) were purchased from Testfabrics, Inc. (West Pittston, PA). 4-Hydroxybenzophenone, 4,4′-dihydroxybenzophenone, 4-chloro-4′-hydroxybenzophenone, 4-benzoylbenzoic acid, 1,2,3,4,-butanetetra carboxylic acid (BTCA), sodium hypophosphite hydrate, and dicyclohexylcarbodiimide (DCC) were purchased from Aldrich Co. (MO, USA). And aldicarb ($C_7H_{14}N_2O_2S$) was purchased from Chem Service, Inc. (PA, USA). All other reagents were purchased from Fisher Science (Pittsburgh, PA) or Acros Co. (NJ, USA).

2.2. Fabric finishing process

A laboratory procedure was used to incorporate different benzophenone chromophoric groups onto cotton fabrics, as shown in Fig. 1. The cotton fabrics, cut in sizes of around 30 cm × 30 cm, were immersed in a finishing solution for 30 min at room temperature. The finishing solutions were prepared as displayed in Table 1. After immersing, the fabrics were padded through a laboratory padder to control the fabrics to have a wet pick-up rate around 100%. Then the treated fabrics were pre-dried at 85 °C for 30 min, and cured at 160 °C for 3 min. After then the fabrics were washed with distilled water, and air-dried in a conditioning room (25 °C, 65%RH) for 24 h. The ultra-violet (UV) lamp (BLE-8 T365, Ultraviolet Spectonics Co., NY, USA) was used for photoactivation of the treated cotton fabrics.

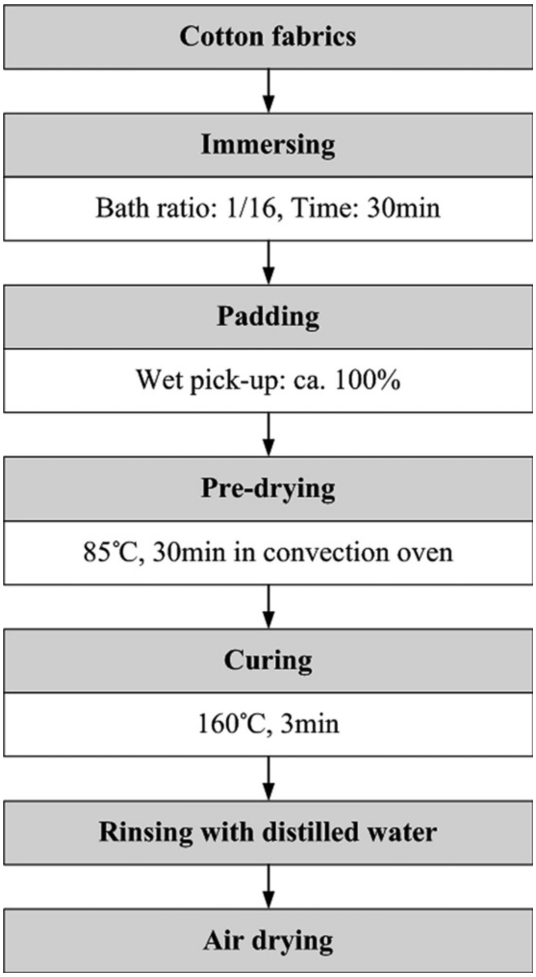


Fig. 1. Preparation process of benzophenone chromophoric group incorporated cotton fabrics.

2.3. Characterization

Fourier transform infrared (FTIR) spectroscopy was performed with a Nicolet 6700 FTIR spectrometer (Thermo Electron Co., USA) with a resolution of 4 cm^{-1}, and the measurements were carried out by using KBr pellets. The tensile strengths of the fabrics were measured by the cut strip method (KS K 0520-1995) with Instron Tensile tester (model 4465). To investigate the pesticide degradation ability of the benzophenone derivative treated cotton fabrics, a high performance liquid chromatography (HPLC) and mass spectrometry (MS) were performed. Aldicarb aqueous solution was employed as a standard pesticide in this study. The standard and the reacted aldicarb aqueous solutions were chromatographed on an Eclipse XDB-C18 column fitted in a Hewlett Packard series 1100 HPLC system containing a diode array detector. The mobile phase was methyl alcohol:water in a ratio of 40:60, the flow rate was 0.4 mL/min, and the absorbance wavelength was measured at 254 nm. And MS analyses were carried out on a standard Q TRAP™ (AB/MDS sciex, Concord, Canada) tandem mass spectrometer. On the other hand, the antimicrobial activities of the fabrics were tested against *Staphylococcus aureus* (*S. aureus*, ATCC 12600, a gram-positive bacterium) and *Escherichia coli* (*E. coli* K-12, a gram-negative bacterium) according to modified testing method for antibacterial activity of textiles (AATCC 100). During the antimicrobial test, pristine and benzophenone derivative treated cotton fabrics were inoculated by bacteria broth and then illuminated under UV light (wavelength: 365 nm; intensity: 1850 $\mu W/cm^2$) in a container for 1 h at room temperature. After the contact time (1 h), 0.1 mL of microbial suspension was taken from the container and then the suspension was diluted to 10^1, 10^2, 10^3, and 10^4 in series. Hundred microliter of the dilution was placed onto agar plates and incubated at 37 °C for 18 h. The reductions of bacteria were calculated according to the following equation:

Table 1
Prescriptions of finishing solutions

1	0.15 M 4-hydroxybenzophenone/acetone	+	0.1 M BTCA + 0.1 M sodium hypophosphite hydrate/distilled water
2	0.15 M 4,4′-dihydroxybenzophenone/acetone	+	0.1 M BTCA + 0.1 M sodium hypophosphite hydrate/distilled water
3	0.15 M 4-chloro-4′-hydroxybenzophenone/acetone	+	0.1 M BTCA + 0.1 M sodium hypophosphite hydrate/distilled water
4	0.15 M 4-benzoylbenzoic acid	+	0.1 M DCC/tetrahydrofuran (THF)

$$\text{Bacteria reduction } (\%) = \frac{(B-A)}{B} \times 100, \qquad (1)$$

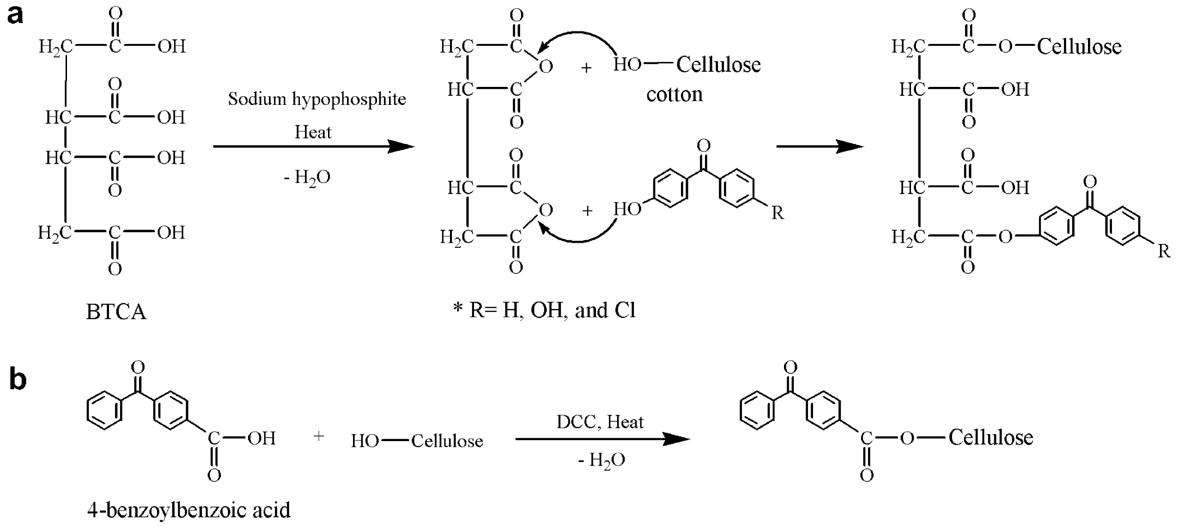
where A and B are the surviving bacterial cells (colony forming unit × mL^{-1}) for the plates containing test samples (treated cotton fabrics) and the control (pristine cotton fabric), respectively.

3. Results and discussion

3.1. Preparation and mechanical properties of benzophenone chromophoric group incorporated cotton fabrics

The crosslinking mechanism between cotton cellulose and polycarboxylic acids, such as butane tetracarboxylic acid (BTCA) is well known (El-tahlawy, El-bendary, Elhendawy, & Hudson, 2005; Sricharussin, Ryo-Aree, Intasen, & Poungraksakirt, 2004). The reaction of polycarboxylic acid with cellulosic polymers occurs through esterification of hydroxyl groups. The polycarboxylic acids contain three or more carboxylic groups, and those groups are capable of forming five- or six-member cyclic anhydride rings. Researchers have suggested that polycarboxylic acids first form cyclic anhydrides as intermediates and then the anhydrides react with cellulosic hydroxyl groups to form ester bonds (El-tahlawy et al., 2005; Sricharussin et al., 2004). Therefore, we expected that the hydroxyl group in benzophenone derivatives, such as 4-hydroxybenzophenone, 4,4′-dihydroxybenzophenone, and 4-chloro-4′-hydroxybenzophenone, could directly react with anhydrides of BTCA while it simultaneously was reacting with cotton cellulose. Thus, BTCA could serve as a crosslinking unit to connect the benzophenone derivatives to cotton fabrics through esterification, as shown in Scheme 1(a). On the other hand, BTCA treated cellulosic fabrics generally suffer a major loss of such mechanical properties, because the cellulosic fibers are decomposed by the acidity of the polycarboxylic acid at high temperature, and the crosslinked sites between cellulosic polymers induce the restriction of stress distribution within the fibers (Kang, Yang, Wei, & Lickfield, 1998; Sricharussin et al., 2004). Therefore, to avoid such a problem another compound containing benzophenone chromophoric group, 4-benzoylbenzoic acid was employed to directly react with cellulose with dicyclohexylcarbodiimide (DCC) as a catalyst, with the reaction shown in Scheme 1(b).

Fig. 2 shows FTIR spectra of pristine cotton and the benzophenone derivatives treated cotton fabrics. All the cotton fabrics show a 1315 cm^{-1} band associated with a bending vibration mode of hydrocarbon structures as well as 1370 and 1430 cm^{-1} bands associated with symmetric stretching of carboxylate groups, which are originated from cellulosic molecules. On the other hand, the hydroxybenzophenone derivatives/BTCA treated cotton fabrics (Fig. 2(c)–(e)) showed some evident peaks of benzophenone chromophoric group. The weak peaks from 1500 to 1602 cm^{-1} are attributed to aromatic C=C stretching, and the peak around 1650 cm^{-1} is carbonyl stretching peak originated from benzophenone group. Also from 800 to 850 cm^{-1}, out of plan bending peaks of *para*-substituted



Scheme 1. Incorporation of benzophenone derivatives to cotton fabrics.

phenyl groups are observed, which is a proof of the linkage between BTCA and the hydroxybenzophenone derivatives. On the other hand, carbonyl peak around 1650 cm^{-1} was overlapped with the cellulose characteristic peak at 1640 cm^{-1}, which is due to the absorbed water molecules in cellulose fiber (Chung, Lee, & Choe, 2004). And also it was observed that the peak in the 4-chloro-4′-hydroxybenzophenone treated cotton fabrics slightly shift to higher field than those in other treated cotton fabrics. Because of the existence of electron-withdrawing group (Cl), the bonding force of the C=O could be enhanced and thus the peak position shifted to higher field. On the other hand, a peak at 1725 cm^{-1} attributed to the ester groups in linkage between cellulose and BTCA (Yang, 1991), which appeared in all hydroxybenzophenone derivatives/BTCA treated cotton fabrics. The relative intensity of this peak in 4,4′-dihydroxybenzophenone treated cotton fabric was stronger than those of other hydroxybenzophenone derivatives treated cotton fabrics. More –OH groups in 4,4′-dihydroxybenzophenone structure could increase reactions between it with BTCA, resulting increased grafting yield of this compound. On the other hand, there are also some traces of benzophenone chromophoric group in 4-benzoylbenzoic acid/DCC treated cotton fabric (Fig. 2(b)) at around 1500–1602 cm^{-1} attributed to aromatic C=C stretching and at around 1650 cm^{-1} attributed to carbonyl stretching. However, these peaks are relatively weak, possibly due to lower incorporation yield of benzophenone chromophoric group on the 4-benzoylbenzoic acid/DCC treated cotton fabric.

Fig. 3 shows the tensile strengths of bezophenone derivatives treated cotton fabrics. The tensile strengths of 4-hydroxybenzophenone, 4,4-dihydroxybenzophenone, 4-chloro-4′-hydroxybenzophenone treated cotton fabrics decreased compared to that of pristine cotton fabric. However, 4-benzoylbenzoic acid treated cotton fabric did not show any significant loss in strength compared to pristine cotton fabric. Therefore, it was presumed that the declining strengths of the hydroxybenzophenone derivatives treated cotton fabrics were caused by the crosslinker, BTCA.

3.2. Antibacterial activity of benzophenone chromophoric group incorporated cotton fabrics

Antibacterial test of the benzophenone derivative treated cotton fabrics were performed against *S. aureus* and *E. coli*, and the results are shown in Tables 2 and 3. 4-Hydroxybenzophenone treated cotton fabric showed the most effective antibacterial ability and then in decreasing order were 4,4′-dihydroxybenzophenne, 4-chloro-4′-hydroxybenzophenone, and 4-benzoylbenzoic acid treated cotton fabrics. Since the Cl group in the 4-chloro-4′-hydroxybenzophenone treated cotton fabric enhanced the stability of the carbonyl group in benzophenone group, the radicals resulted in the carbonyl position of the sample could be reduced, resulting in low antibacterial activities. And, in the case of 4,4′-dihydroxybenzophenone treated cotton fabric, both of the –OH in the structure might be bonded to BTCA, then the benzophenone would be adjacent to ester groups on both side of the benzophenone group, thus the electrons around the carbonyl group in benzophenone would be de-localized, therefore, the radical reactivity would be decreased compared to that of 4-hydroxybenzophenone treated cotton fabric. On the other hand, the low antibacterial ability of the 4-benzoylbenzoic acid treated cotton fabric could be caused by the low grafting yield, which is consisted with the FTIR result. On the other side, slight hydrophobicity of 4-benzoylbenzoic acid treated cotton may also contribute to even lower antimicrobial ability. During the inoculation step in antibacterial test, it was observed that bacterial broth had poor contact with and could not be absorbed by the 4-benzoylbenzoic acid treated cotton fabric easily. The poor contact between bacteria and benzophenone chromo-

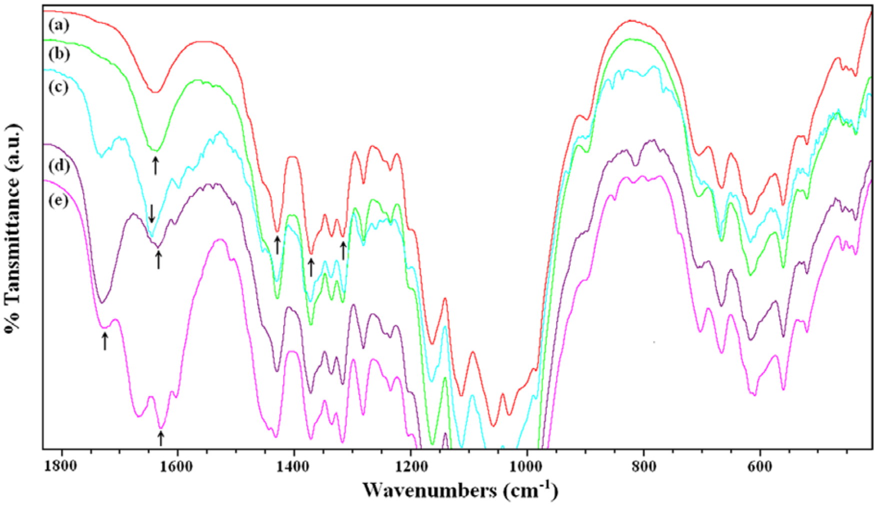


Fig. 2. FTIR spectra of cotton fabrics: (a) pristine cotton fabric, (b) 4-benzoylbenzoic acid/DCC treated cotton fabric, (c) 4-chloro-4′-hydroxybenzophenone/BTCA treated cotton fabric, (d) 4,4′-dihydroxybenzophenone/BTCA treated cotton fabric, and (e) 4-hydroxybenzophenone/BTCA treated cotton fabric.

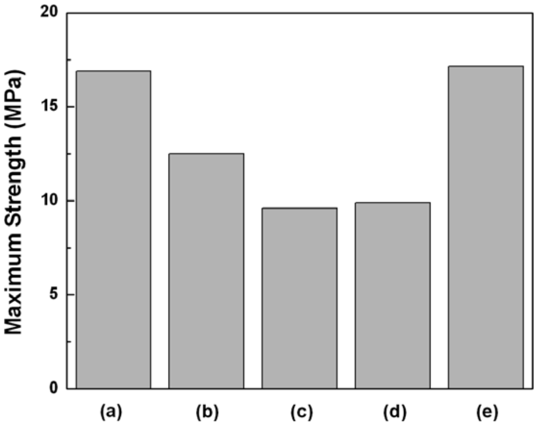


Fig. 3. Tensile strengths of cotton fabrics after 3 h UV illumination; (a) pristine cotton fabric, (b) 4-hydroxybenzophenone/BTCA treated cotton fabric, (c) 4,4′-dihydroxybenzophenone/BTCA treated cotton fabric, (d) 4-chloro-4′-hydroxybenzophenone/BTCA treated cotton fabric, (e) 4-benzoylbenzoic acid/DCC treated cotton fabric.

phoric radical in the treated cotton fabric could reduce antimicrobial efficacy. In addition, the reusability of the benzophenone chromophoric radical in 4-hydroxybenzophenone treated cotton fabric was investigated, and results (shown in Tables 2 and 3 as 4-hydroxybenzophenone/BTCA treated cotton fabric (Re)) were very encouraging, the sample maintained the same antibacterial power after repeated exposure to UV and bacteria.

3.3. Pesticide degradation ability and UV–VIS transmittance of benzophenone chromophoric group incorporated cotton fabrics

The benzophenone chromophoric group treated cotton fabrics were also tested against some pesticides as an effort to develop protective clothing material. Pesticide degradation ability and UV–VIS transmittance of 4-hydroxybenzophenone and 4-benzoylbenzoic acid treated cotton fabrics were examined. For analyzing the pesticide degradation ability, 1 g of pristine cotton fabric, 4-hydroxybenzophenone treated cotton fabric, and 4-benzoylbenzoic acid treated cotton fabric were put in containers containing 0.25 mmol aldicarb aqueous solution (4 g), respectively. Then the fabrics immersed aldicarb aqueous solutions were irradiated by UV light for 3 h and analyzed by HPLC and MS spectroscopy. Aldicarb is a carbamate insecticide with structural formula: 2-methyl-2-(methylthio) propionaldehyde *O*-methylcarbamoyloxime, and it is the active substance in Temik pesticide, which is effective against thrips, aphids, spider mites, lygus, fleahoppers, and leafminers but is primarily used as a nematicide. And there have been several documented cases of human poisoning with aldicarb indicating cholinergic overload and acute peripheral nervous system dysfunction when consumed in excessive concentrations (Schlenk, Cashman, Teung, Zhang, & Rettie, 2002). Fig. 4 shows the HPLC chromatogram of pure aldicarb aqueous solution, pristine cotton fabric immersed aldicarb aqueous solution, 4-hydroxybenzophenone treated cotton fabric immersed aldicarb aqueous solution, and 4-benzoylbenzoic acid treated cotton fabric immersed aldicarb aqueous solution, respectively. An aldicarb characteristic peak appears at around 6.6–6.9 min, and the peak was observed in all chromatograms. However, the average areas (mAU* s) of the aldicarb characteristic peak in the fabric immersed aldicarb solutions decreased compared to that of pure aldicarb solution as following order; 4-benzoylbenzoic acid treated cotton fabric (69.31%) ≫ 4-hydroxybenzophenone

Table 2
Colony numbers of *S. aureus* after injection of 0.1 mL bacteria suspension on agar plates and incubation at 37 °C for 18 h

Against to *S.aureus*	Dilution ratio of the bacteria solution after contact time				Reduction of bacteria (%)
	×10	$\times10^2$	$\times10^3$	$\times10^4$	
Pristine cotton fabrics	∞	∞	∞	230	–
4-Hydroxybenzophenone/BTCA treated cotton fabrics	0	0	0	0	>99.999
4-Hydroxybenzophenone/BTCA treated cotton fabrics (Re)	0	0	0	0	>99.999
4,4′-Dihydroxybenzophenone/BTCA treated cotton fabrics	4	0	0	0	99.998
4-Chloro-4′-hydroxybenzophenone/BTCA treated cotton fabrics	184	21	0	0	99.909
4-Benzoylbenzoic acid/DCC treated cotton fabrics	∞	∞	∞	26	88.696

Table 3
Colony numbers of *E. coli* after injection of 0.1 mL bacteria suspension on agar plates and incubation at 37 °C for 18 h

Against to *E. Coli*	Dilution ratio of the bacteria solution after contact time				Reduction of bacteria (%)
	×10	$\times10^2$	$\times10^3$	$\times10^4$	
Pristine cotton fabrics	∞	∞	∞	155	–
4-Hydroxybenzophenone/BTCA treated cotton fabrics	5	1	0	0	99.997
4-Hydroxybenzophenone/BTCA treated cotton fabrics (Re)	7	0	0	0	99.995
4,4′-Dihydroxybenzophenone/BTCA treated cotton fabrics	∞	52	2	0	99.665
4-Chloro-4′-hydroxybenzophenone/BTCA treated cotton fabrics	∞	175	17	0	98.903
4-Benzoylbenzoic acid/DCC treated cotton fabrics	∞	∞	∞	49	68.387

treated cotton fabric (26.95%) > pristine cotton fabric (4.65%). Therefore, 4-benzoylbenzoic acid treated cotton fabric showed the most prominent aldicarb degradation ability, and the result was confirmed by MS analysis as shown in Fig. 5. In positive ion mode of MS, 89 (*m*/*z*) $[M–CH{=}NOCONHCH_3]^+$, 116 (*m*/*z*) $[M–OCONHCH_3]^+$ and, 213 (*m*/*z*) $[MNa]^+$ are natural fragment ions of aldicarb (Corcia, Nazzari, Rao, Samperi, & Sebastiani, 2000; Totti et al., 2006), and also 403 (*m*/*z*) was known as dimmer type ion of aldicarb molecule $[MMNa]^+$. These aldicarb characteristic ion peaks were observed in all MS spectra. On the other hand, in the spectrum of 4-ben-

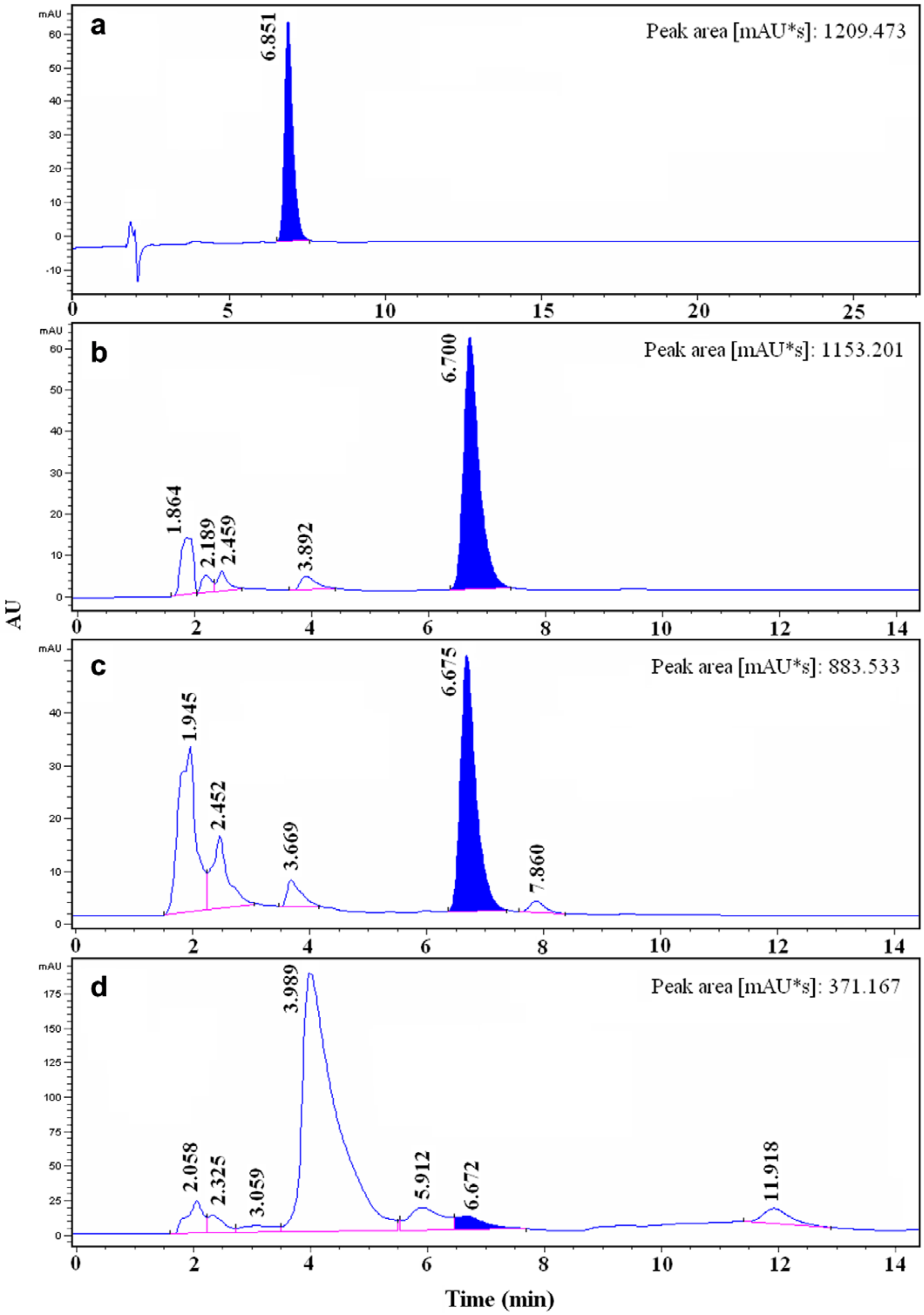


Fig. 4. HPLC chromatograms of (a) pure aldicarb aqueous solution, (b) pristine cotton fabric immersed aldicarb aqueous solution, (c) 4-hydroxybenzophenone/BTCA treated cotton fabric immersed aldicarb aqueous solution, and (d) 4-benzoylbenzoic acid/DCC treated cotton fabric immersed aldicarb aqueous solution.

zoylbenzoic acid treated cotton fabric immersed aldicarb aqueous solution, a peak at 183 (*m*/*z*) is newly observed, and it was tentatively assigned as a decomposed fragment ion of aldicarb $[CH_3SC(CH_3)_2CH{=}NOCONa]^+$. This result is opposite to what we observed from antibacterial tests. The 4-benzoylbenzoic acid treated cotton fabric is more hydrophobic than other treated cotton samples, which may be more interactive with hydrophobic aldicarb and then has increased contact and decomposed aldicarb.

UV–VIS transmittance (%) of pristine cotton fabric, 4-hydroxybenzophenone treated cotton fabric, 4-benzoylbenzoic acid treated cotton fabric were measured as shown in Fig. 6, because UV blocking ability is also an important value for protective clothing. All cotton fabrics did not transmit the 345–390 nm of light, but the benzophenone derivative treated cotton fabrics blocked a broader range of light extended to lower wavelength. However, in 200–340 nm, 4-hydroxybenzophenone treated cotton fabric transmitted more light than pristine and 4-benzoylbenzoic acid treated cotton fabric did. This result is unable to illustrate the difference of the degradation power of the fabrics. Therefore, to achieve the optimal functionalities of the finish, it would be necessary to further study on structures and their antibacterial and chemical detoxifying properties of benzophenone groups in the future.

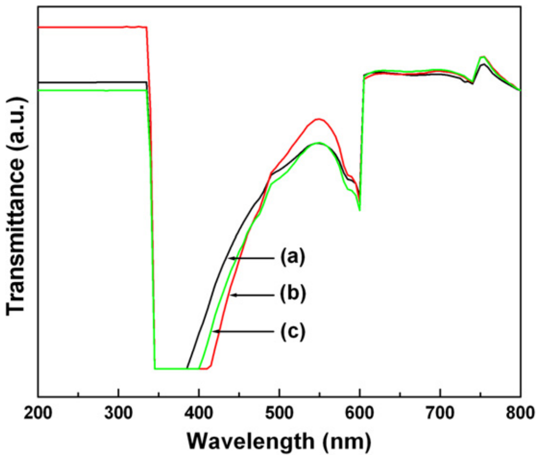


Fig. 6. UV–VIS spectra of (a) pristine cotton fabric, (b) 4-hydroxybenzophenone/BTCA treated cotton fabric, and (c) 4-benzoylbenzoic acid/DCC treated cotton fabric.

4. Conclusion

Various benzophenone derivatives incorporated cotton fabrics were prepared and analyzed. The results indicated that the reaction yield of 4-benzoylbenzoic acid/DCC with cotton fabric was lower than those of hydroxybenzophenone derivatives/BTCA with cotton fabrics. However, the mechanical strength of the 4-benzoylbenzoic acid/DCC

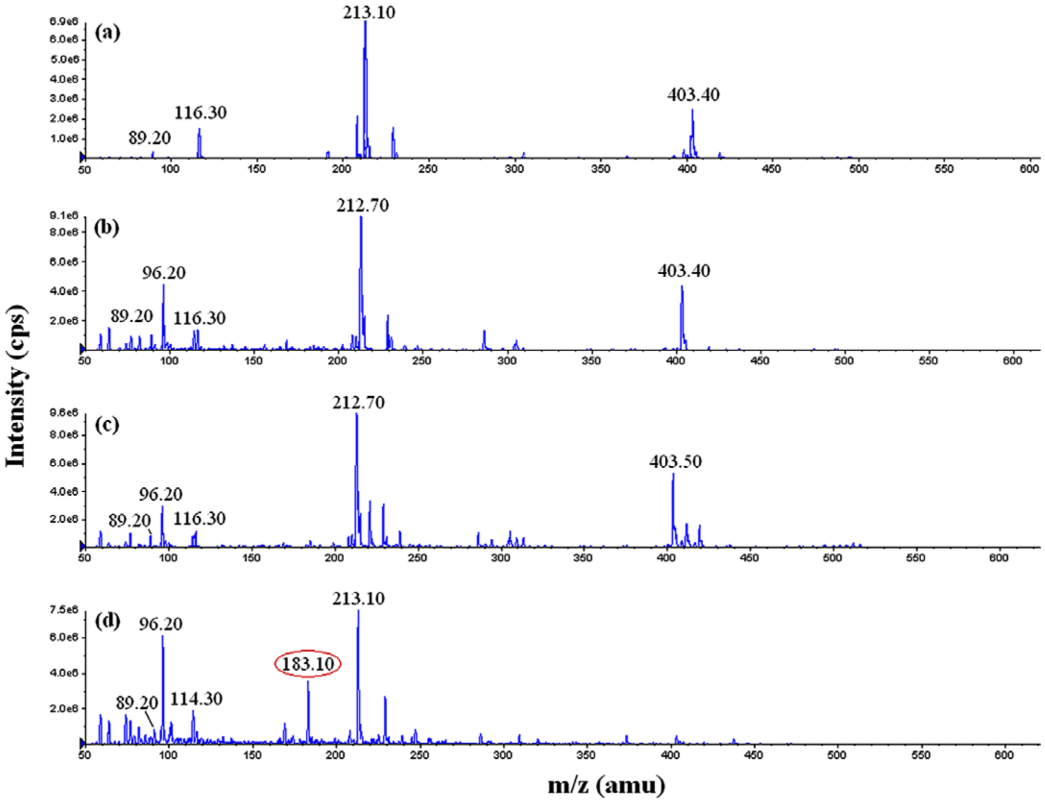


Fig. 5. MS spectra of (a) pure aldicarb aqueous solution, (b) pristine cotton fabric immersed aldicarb aqueous solution, (c) 4-hydroxybenzophenone/BTCA treated cotton fabric immersed aldicarb aqueous solution, and (d) 4-benzoylbenzoic acid/DCC treated cotton fabric immersed aldicarb aqueous solution.

treated cotton fabric was stronger than those of the hydroxybenzophenone derivatives/BTCA treated cotton fabrics. 4-Hydroxybenzophenone treated cotton fabric showed the most powerful antibacterial ability and then in decreasing order were 4,4′-dihydroxybenzophenone treated cotton fabric > 4-chloro-4′-hydroxybenzophenone treated cotton fabric > 4-benzoylbenzoic acid treated cotton fabric. However, 4-benzoylbenzoic acid treated cotton fabric exhibited the best degradation ability against aldicarb, possibly due to the hydrophilicity of the surfaces of the treated cotton fabrics. Therefore, to achieve the optimal functionalities of the finish, it would be necessary to further study on structures and their antibacterial and chemical detoxifying properties of benzophenone groups in the future.

Acknowledgements

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