

# Creating Novel Functions on Textiles by Applying Organic Chemistry

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## Introduction

Textile chemistry is an applied chemistry in a highly-specialized area of textile materials, applying basic principles of organic, physical, and analytical chemistry and incorporating novel or desired functions and esthetic modifications onto textile materials. The textile materials include fibers, yarns, fabrics, clothing, carpets, upholsteries, composite materials, and beyond. My career as a textile chemist began earlier in the 1980s in China, but I became an independent researcher in 1995, when I was appointed as an assistant professor at the Division of Textiles and Clothing at University of California, Davis, after receiving my Ph.D. in chemistry and postdoctoral training at Auburn University. The focus of research and teaching in my group has been on textile chemistry in the past twenty years, and of course, the achievements made by the group in the area were based on the chemistry as well. Here is a brief summary with a few examples.

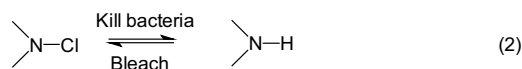
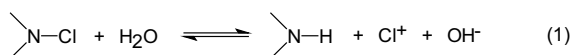
## Biocidal Textiles for Personal Protection

Antimicrobial textiles can be categorized into biocidal and biostatic materials according to their functions against microorganisms. Biostatic functions inhibit growth of microorganisms on textiles and prevent the materials from biodegradation, and are best used in protecting textile materials; biocidal materials are able to kill microorganisms and thus can eliminate growth of microorganisms, and therefore can protect wearers of the textiles from biological attacks, in addition to protecting the materials. Biostatic functions are usually employed in preservation of textile artefacts in museums or for odor control of materials, but cannot prevent transmission of diseases due to the limitation of the functions. In general, for personal protective purposes, biocidal functions that can provide rapid and efficient inactivation of a broad spectrum of microorganisms are desired.

Chemicals that can provide such biocidal functions belong to disinfectants, including aldehydes, alcohols, halogens, halamine structures, peroxy acids, and hydrogen peroxide.<sup>1</sup> However, many of them may have difficulty to be safely employed on textile fabrics. Halamines and peroxide compounds are stable on surfaces of materials and safe to be employed on textiles with intimate skin contacts.<sup>2-5</sup>

## Halamine Chemistry

Fortunately, halamine compounds (N-X, X = Cl, Br, I) have been widely employed as swimming pool disinfectants, with a proven record of safe human skin contacts.<sup>6</sup> Halamine chemistry can be briefly described with the following two equations (Eqs. 1 and 2). Eq. 1 represents the reaction of halamines serving as water disinfectants in swimming pools, by releasing certain amount of free chlorine in water. N-Cl bonds in chloramine compounds are as biocidal as free chlorine,<sup>7</sup> which became the basis of development of surface contact biocidal materials (Eq. 2). Most people can stay in swimming pools for hours, where skin contacts with halamine occur intimately. Thus, halamine structures can be possibly employed in materials with human skin contacts. The reaction in Eq. 2 also serves as the rechargeable biocidal mechanism of halamine solid materials.



N-halamine structures are made of primarily three bonds: imide, amide, and amine (Table I), which may release limited amounts of free chlorine depending on stability of the imide, amide, and amine halamines (Table 1).<sup>1</sup> Imide halamine releases a relatively high





**Table I.**  
Stability of *N*-Halamine Structures

Dissociation Reaction	Dissociation Constants
Imide Structure 	$10^{-2} - 10^{-4}$
Amide Structure 	$10^{-8} - 10^{-9}$
Amine Structure 	$< 10^{-12}$

amount of free chlorine and can be found in water disinfectants in swimming pools and other consumer products, while amine halamine releases extremely low amount of free chlorine based on the dissociation constants of the structures (Table I).

Since *N*-halamine structures are biocidal, and more importantly quite stable in ambient environments, incorporation of the *N*-halamine into polymeric and textile materials will bring biocidal functions to them. Moreover, since Eq. 2 is a reversible reaction, the biocidal functions on the materials are rechargeable with a chlorinating agent, such as chlorine bleach. This rechargeable function is primarily suitable for reusable medical and institutional textiles. Thus, we have developed different processes to incorporate halamine structures to all textile fibers.

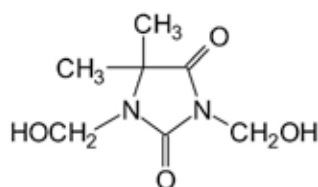
Incorporation of halamine structures into different polymers and fibers could bring biocidal functions to such materials with different biocidal potentials.<sup>8–23</sup> Fabrics with these structures have been prepared in this laboratory and demonstrated rapid inactivation against a full spectrum of pathogenic diseases and even spores. In fact, the halamine fabrics provide the same oxidative function against biological agents as chlorine bleach and could be defined as textile disinfectants.

### Halamine Finishes on Cellulose

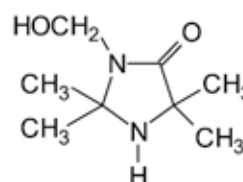
Both amide and imide *N*-halamines have been incorporated into cellulose-containing fabrics by conventional finishing methods with 1,3-dimethylol-5,5-dimethylhydantoin (DMDMH, Fig. 1),<sup>8,9</sup> and the fabrics exhibited rapid biocidal functions. The washing and storage durability of the functions is not so ideal due to the

dominating imide *N*-halamine functionality. But the biocidal functions are the best on the fabrics. DMDMH fabrics can be employed in personal protection products against various biological agents such as bacteria, viruses, fungi, yeasts, and spores. Examples of the treated fabrics demonstrated a complete elimination of pathogens in a contact time as short as two minutes. The biocidal functions could be recharged repeatedly for at least 50 machine washes.<sup>8,9</sup>

To increase washing durability of the *N*-halamine-treated textiles, the more stable amine *N*-halamine has been grafted to cellulose in a similar approach by using 3-methylol-2,2,5,5-tetramethylimidazolidin-4-one (MTMIO) (Fig. 1). The resulting fabrics possess a more stable and less reactive amine *N*-halamine



1,3-dimethylol-5,5-dimethylhydantoin  
(DMDMH)



3-methylol-2,2,5,5-tetramethylimidazolidin-4-one  
(MTMIO)

Fig. 1. Structures of DMDMH and MTMIO



**Table II.**  
Chlorine Loss and Antimicrobial Effects of MTMIO- and DMDMH-Modified Cotton Samples after Repeated Washing without Recharging<sup>13,14</sup>

Washing Cycles	MTMIO			DMDMH		
	Cl ppm	Cl Loss %	Log Reduction	Cl ppm	Cl Loss %	Log Reduction
0	565	—	6	863	—	6
2	507	10.2	5	218	74.7	1.5
5	498	11.9	4	157	81	0.9

structure, thus providing slow, but durable, biocidal functions (Table II). The only disadvantage is that both DMDMH and MTMIO are formaldehyde derivatives, limited to applications only on cellulosic materials.<sup>13,14</sup>

**Halamine Finishes on Other Fibers**

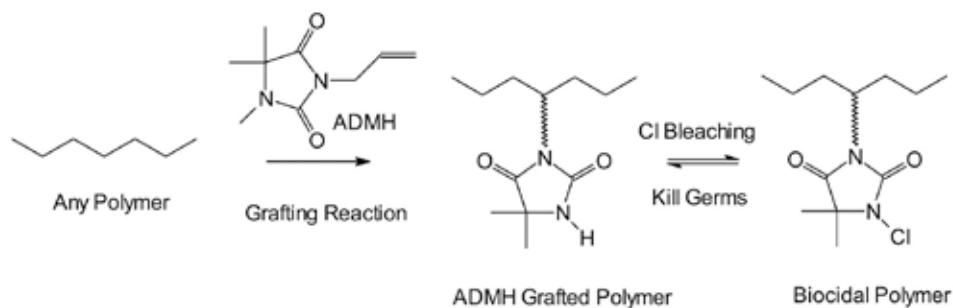
Synthetic polymers and fibers are chemically inert and difficult to be modified. Using radical graft polymerization reactions and vinyl halamine precursor monomers, the biocidal halamine structures were incorporated onto most fibers. A hydantoin-containing vinyl monomer, 3-allyl-5,5-dimethylhydantoin (ADMH, Scheme 1), is a good example,<sup>10</sup> which can incorporate only amide *N*-halamine structures into all possible fibers. Due to the amide structure, the thus-produced fabrics

could demonstrate both powerful and durable biocidal functions. Synthetic fabrics such nylon-66, polyester (PET), polypropylene (PP), acrylics, and aramid fibers, as well as pure cotton fabrics, were successfully modified.<sup>12,13</sup> More recently, ADMH can be incorporated in surfaces of PET fibers by a newly developed controlled radical grafting reaction (Scheme 1).<sup>15,16,24</sup>

Biocidal properties of the modified fibers against *E. coli* and *S. aureus* are shown in Table III. Most of the fibers could completely inactivate a large number of bacteria ( $1 \times 10^6$  CFU) in 10-30 minutes of contact time. In addition, the antibacterial activities of these polymeric *N*-halamines could be easily recovered after usage by simply exposing the fabrics to a chlorine solution again.

**Incorporation of Halamine to Olefin Fibers**

As an effort to incorporate halamine structures to olefin polymers and fibers, a new technology was developed in this lab by using a polymer reactive extrusion process. The polymer can be chemically modified with certain reactive halamine precursor monomers using a radical graft polymerization process (Scheme 2).<sup>20-23</sup> The reaction can be carried out

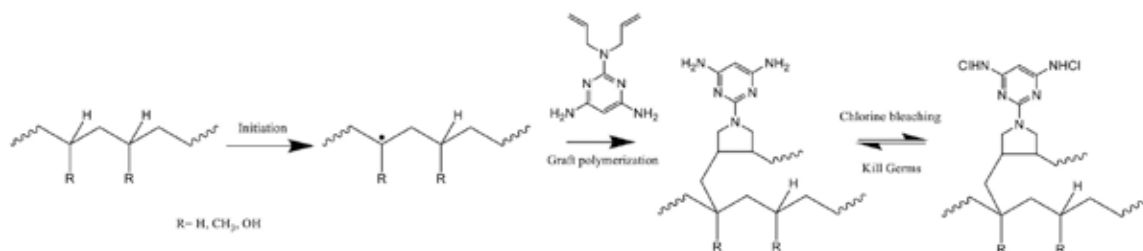


Scheme 1. Structure of ADMH and its grafting reactions on synthetic polymers.

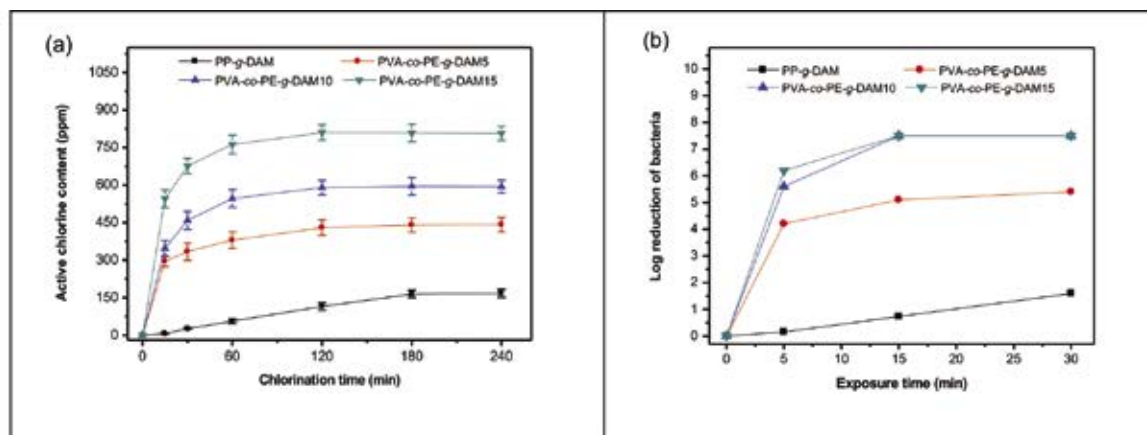
**Table III.**  
Log Reduction of *E. Coli* after Washing and Recharging<sup>12</sup>

Washing Times	Log Reduction of <i>E. Coli</i>					
	Nylon	PET	PP	Acrylic	Cotton	PET/Cotton
0	5	5	5	5	5	5
5	5	5	5	5	3	5
15	5	5	5	5	1	5
30	3	3	2	1	UD	3
50	UD	1	1	1	UD	UD
50	5	5	5	5	5	5





Scheme 2. Radical graft polymerization and halamine reaction of olefin polymers

Fig. 2 (a) Active chlorine contents and (b) antibacterial (*S. aureus*) properties of halamine olefin materials.<sup>26</sup>Table IV. Wet Bacterial Penetration Test Results of Submicrosized Fiber Webs (ISO22610)<sup>26</sup>

Agar Plates	15 min	30 min	45 min	60 min	75 min	Reverse
PP-g-DAM	42	8	0	15	23	245
PVA-co-PE-g-DAM5	12	0	0	2	6	4
PVA-co-PE-g-DAM10	0	0	0	0	0	0
PVA-co-PE-g-DAM15	0	0	0	0	0	0

in a twin-screw extruder during the PP compounding process to prepare master batch polymers, and then the modified olefin polymers can be directly extruded into nonwovens. Halamine nonwoven fabrics can serve as basic materials for producing personal protective equipment with biocidal functions and improved protections against bacteria and viruses.

As an example (Scheme 2), diallyl melamine (DAM) grafted PP (PP-g-DAM) and poly(vinyl alcohol-co-ethylene-g-DAM)) fibers were prepared with very high active chlorine (100–750 ppm Cl<sup>+</sup>) and have the ability to completely kill *S. aureus* in a contact time of less than 15 minutes (Figs. 2a and b).<sup>20–23,25,26</sup> It is worth noting that the biocidal results shown in

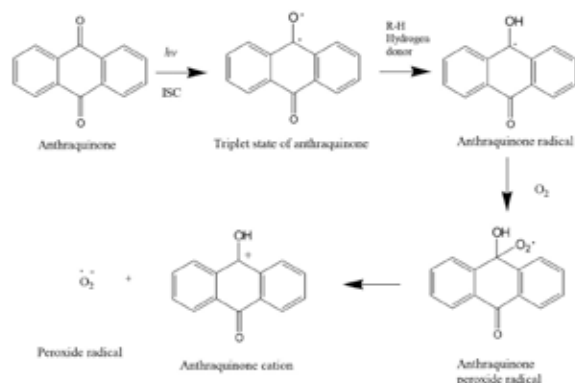
Table IV are results from bacterial wet penetration tests on nonwoven fabrics (submicro and nanosized fibrous membranes) made of the halamine fibers, following an international testing protocol (ISO 22610). The results reveal very fast contact kills against a bacterium (*E. coli*) when the liquid is passing through the

materials while the halamine membrane materials are breathable. When DAM units were over 10% in the copolymers, no live bacteria were able to pass when the liquid penetrated through the nonwovens. The nanofibrous membranes possess ultrahigh surface areas and could further enhance the surface biocidal functions.<sup>20–23,26,27</sup>

### Photochemistry and Light-Induced Biocidal Fibers and Fabrics

Halamine materials are the most powerful biocidal agents, and the biocidal functions are refreshable with chlorine bleach. However, halamine structures are not so stable under ultraviolet irradiation and high temperature and may cause other concerns





Scheme 3. Photo-production of reactive oxygen species (ROS) by anthraquinone compounds.

such as color losses and faint chlorine smell on the products. Aimed at providing other options on biocidal textiles, we also investigated novel self-decontaminating biocidal polymers and textiles without the use of halamine structures.<sup>28-31</sup> One of the innovative approaches was the discovery of light-induced biocidal effects of certain colorants and chemicals on surfaces of fibers, as well as self-storage effects of certain chemical structures on light-induced biocidal materials.<sup>32-45</sup> According to photochemistry, colorants and certain benzophenone derivatives, anthraquinone compounds, and vat dyes are light-active agents. These light-active compounds could primarily produce hydrogen peroxide ( $H_2O_2$ ) and superoxide, so called reactive oxygen species (ROS) under light (Scheme 3), with the help of a hydrogen donor such as cellulose, nylon, and PVA-co-PE nanofibers.<sup>32-45</sup> These ROS can consequently form hydrogen peroxide in the presence of moisture, or more stable peroxy acid

if carboxylic acid groups are available.<sup>28,29</sup> Hydrogen peroxide and peroxy acid groups are stable in ambient environments and also are less toxic to humans. In fact, both are employed in disinfection applications of surface materials and are considered as another group of biocides that could quickly kill pathogens by contacts.

Scheme 3 shows the chemistry of generation of reactive oxygen species (ROS) by an anthraquinone structure. The light sources include UVA (365 nm) and white light lamps that provide light intensity ( $h\nu$ ) similar to a normal room lighting environment.<sup>38,39</sup> Anthraquinone molecules can be excited to a singlet state and then go through inter-system crossing (ISC) to a triplet state. The triplet-excited anthraquinone is more stable and can abstract a hydrogen atom from a donor (R-H, fiber, polymer, solvent, or even microorganisms) to produce an anthraquinone radical and a radical on the donor. The anthraquinone radical can undergo reactions with oxygen in air to form anthraquinone peroxide radical, which then can produce superoxide, hydroxyl radical, and hydrogen peroxide through other reactions.<sup>37</sup> The anthraquinone radicals can be re-oxidized to the original anthraquinone compounds by peroxy radicals or couple with the solvent/polymer radicals (not shown). The formation of the original anthraquinone on the materials explains the durability and rechargeability of the light-active functions. Benzophenone derivatives have the same reaction mechanism to produce ROS. During the entire process, the overall consumed chemicals are mostly oxygen in the air and partially anthraquinone or benzophenone compounds on the surfaces of polymers, while the continuous production of the hydrogen peroxide ( $H_2O_2$ ) and ROS kills microorganisms.

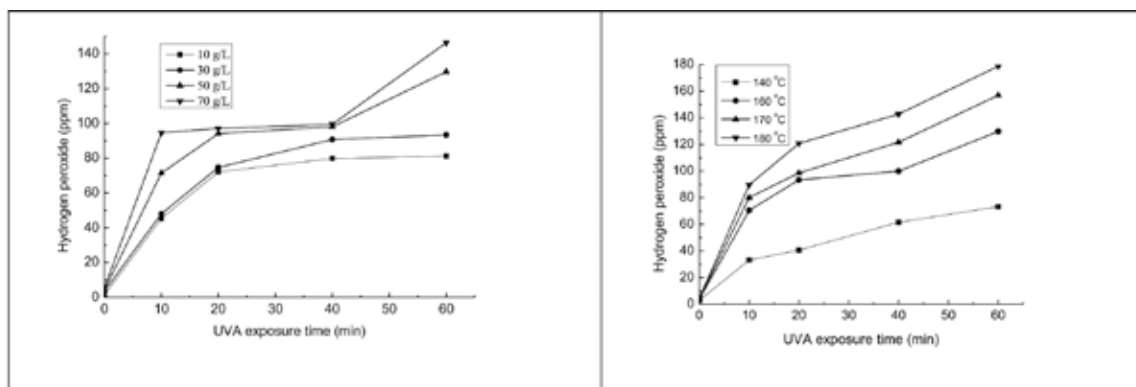


Fig. 3. Amounts of hydrogen peroxide produced by cotton fabrics treated with BPTCA.<sup>45</sup>





**Table V.**  
Wash Durability of Antimicrobial Functions of 2-Ethyl-anthraquinone (AQ) and Vat Yellow GCN (Vat Dye) Dyed Cotton Fabrics (60 min of light exposure)<sup>40</sup>

Dyeing Concentration (owf)	Bacterial Reduction (%)					
	<i>E. coli</i> (10 <sup>5</sup> CFU/mL)			<i>S. aureus</i> (10 <sup>4</sup> CFU/mL)		
	No Wash	5 Washes	10 Washes	No Wash	5 Washes	10 Washes
6% AQ	99.99	99.99	99.99	99.9	87.6	67.6
9% AQ	99.99	99.99	99.99	99.9	95.6	61.9
3% Vat Dye	99.99	99.99	99.99	99.9	97.8	86.8

**Table VI.**  
Bacterial Reduction (%) caused by AQC Functionalized Nanofibrous Membranes under Different UVA Exposure Time<sup>44</sup>

Bacterium	<i>S. aureus</i>		<i>E. coli</i>	
	30 min	60 min	30 min	60 min
PVA-co-PE27-AQC	87.623	>99.999	80.443	>99.999
PVA-co-PE32-AQC	86.893	>99.999	78.012	>99.999
PVA-co-PE44-AQC	87.920	>99.999	82.375	>99.999

Both anthraquinone structures and 3,3',4,4'-benzophenone tetracarboxylic acid (BPTCA) were incorporated onto cellulose, and the cotton products demonstrated very promising photo-induced generation of hydrogen peroxide and antimicrobial functions (Fig. 3 and Table V).

When the same anthraquinone structure is incorporated onto nanofibers such as poly(vinyl alcohol-co-ethylene) (PVA-co-PE) nanofibrous membranes, the product demonstrated even more powerful biocidal functions due to the increased surface areas (Table VI).<sup>44</sup> More recently, photo-induced effects were observed on the materials containing the above structures without direct exposure to light. Such an effect is extremely interesting and important for development of light-active biocidal textiles, which is still under investigation in the laboratory.

Peroxides and ROS act as oxidizing agents and broad spectrum biocides by denaturing microorganisms and proteins. Peroxyl compounds vary in their oxidative activities based on different structures. The advantages of employing ROS as biocides on textiles are the combination of colorants or other functional agents with biocidal functions, self-generation of hydrogen peroxide and other ROS, sustainability, and stability during storage and light exposure.

## Functional Cotton Textiles

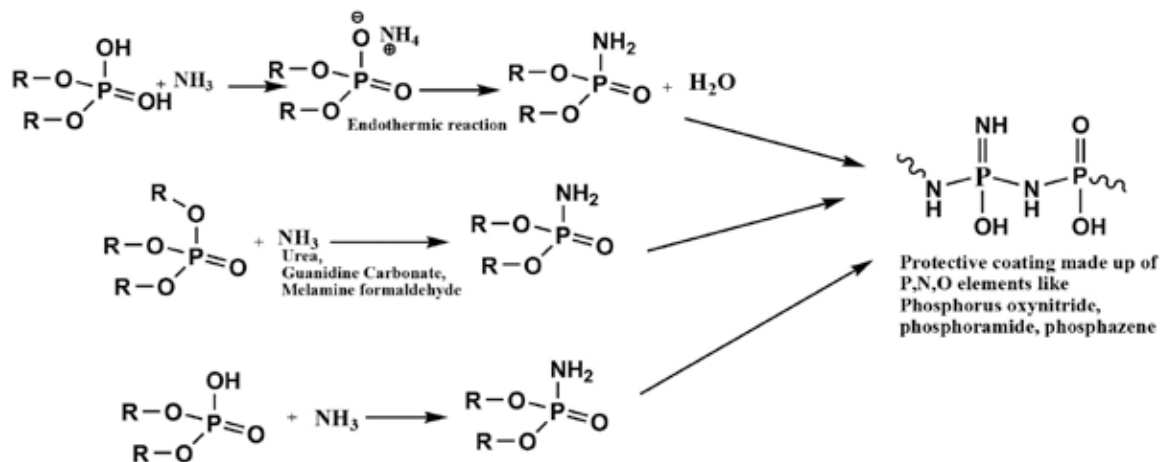
In addition to the development of biocidal textiles, my research group has also explored many different functional finishes on textiles, including phosphorous-nitrogen synergistic flame retardancy, non-formaldehyde crosslinking, and non-fluoride water repellency of cotton fabrics, as well as novel fabrication processes of thermoplastic nanofibers and functional nanofibers.<sup>20–23,50–53</sup> Two additional examples of our contributions to textile chemistry are discussed in the following sections.

### Phosphorus-Nitrogen Synergistic Effect on Cellulose

Organophosphorus compounds have been widely employed as the main fire retardants for cotton, together with nitrogen-containing compounds, which can provide a synergistic effect. Such a combination ensures formation of chars from the treated cotton and prevents further decomposition of the materials into flammable chemicals, thereby extinguishing fires. However, not all nitrogen-containing compounds could provide the synergistic effect with phosphorus. Lack of understanding on the mechanism of this synergistic effect inhibits development of new flame retardants for textiles, especially to cellulosic materials.<sup>46</sup>







Scheme 4. Proposed synergistic effect of P-N<sup>48</sup>

We selected several representative organophosphorus compounds and nitrogen-containing compounds to treat cotton fabrics, and systematically analyzed structural and compositional changes of the treated cotton during thermal degradation processes. The results revealed that the phosphorus compounds mostly decompose to phosphoric acid rapidly, causing a subsequent dehydration reaction and char formation of cellulose.<sup>46</sup> For nitrogen-containing compounds, if they are able to quickly release ammonia under heat, ammonia could catalyze dehydration of cellulose by phosphoric acid and further increase formation of chars, which is the cause of the synergistic effect (Scheme 4).<sup>48,49</sup> As a result, only nitrogen compounds that can release ammonia under heat can serve as good synergistic agents to organophosphate.

### Crosslinking Cellulose with Polycarboxylic Acids

Polycarboxylic acids, such as 1,2,3,4-butanetetracarboxylic acid (BTCA) and citric acid, have been investigated as non-formaldehyde crosslinking agents in wrinkle-resistant finishing of cotton fabrics for decades. In recent years, we also explored aromatic polycarboxylic acids, such as 3,3',4,4'-benzophenone tetracarboxylic acid (BPTCA), as a potential functional crosslinker. We found that BPTCA has an advantage in reaction with cellulose, having a one step-direct esterification reaction, different from BTCA and other aliphatic polycarboxylic acid.<sup>41,54</sup>

Due to the fact that sodium hypophosphite (SHP) serves as a good catalyst to both direct and indirect esterification reactions, we further investigated the catalytic mechanism of SHP and other salts in both reactions and found that acidity (pKa) of corresponding acids of salt anions play an important role in a Fischer esterification reaction mechanism, while large cations help the anhydride formation step from aliphatic polycarboxylic acids.<sup>55,56</sup> In addition, both the sizes of the acid and catalysts and their affinities to cellulose also impact the crosslinking reaction efficiency,<sup>57,58</sup> which has been considered as a structural factor in causing disparity of the measured wrinkle recovery angle (WRA) and durable press (DP) performance of the treated fabrics. Overall, polycarboxylic acids should have good affinity to cellulose and a smaller size to diffuse rapidly into cellulose to result in effective crosslinking. Taking a balanced consideration, citric acid has been considered as an ideal crosslinker, except for the yellowing effect caused by a dehydration reaction and formation of a conjugated system. However, effective bleaching processes could remove the yellow color without significantly affecting the crosslinking structures.<sup>59,60</sup> In addition, the yellow color is only noticeable on light-colored cotton fabrics and not on darkly dyed ones.

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