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Synthesis and characterization of chitosan based dye containing quaternary ammonium group

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ABSTRACT

A new antimicrobial biopolymer dye was synthesized by reaction of quaternary ammonium salt of chitosan and reactive red x-3b. And quaternary ammonium salt of chitosan was produced by grafting glycidyltrimethylammonium chloride on chitosan. The synthesized materials were characterized by Fourier transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD), solubility test and antimicrobial test. Results show that the antimicrobial biopolymer dye was combined by $-N^{+}(CH_3)_3$ of quaternary ammonium salt of chitosan and sulfonic group of reactive red x-3b. Water solubility of chitosan biopolymer dye was increased as well as pH value. In addition, antibacterial property of new synthesized dye was excellent, whose antibacterial rates of *Staphylococcus* and *Escherichia coli* were both bigger than 99%. These results may provide new perspectives on improving the decorative properties and antimicrobial properties in wood industry.

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1. Introduction

Chitosan is a modified biopolymer obtained from chitin, one of the most abundant natural amino polysaccharide (Corazzari et al., 2015), by deacetylation and formed primarily of repeating units of beta-(1,4)-2-amino-2-deoxy-D-glucose (or D-glucosamine) (Basturk, 2012). It is biodegradable, nontoxic, renewable, antimicrobial and fungistatic (Myllytie, Salmi, & Laine, 2009; Sargin, Kaya, Arslan, Baran, & Ceter, 2015). Due to its special physic-chemistry properties, chitosan has been broader application in a number of fields including as adsorbent in water treatment of heavy mental (Wang & Chen, 2014) and dye (Vakili et al., 2014), as drug-delivery in biomedical field (Mati-Baouche et al., 2014), as antibacterial agent in finishing textile (Babu & Ravindra, 2015) and paper (Park, Kang, & Lim, 2010) and so on. However, this extraordinary carbohydrate polymer has a high molecular weight resulting in low solubility in water which limits its application in wood, food (Sargin et al., 2015) and the like industries.

To improve the water solubility of chitosan, several physical and chemical methods have been tried to prepare a water soluble chitosan with low molecular weight (Xia, Wu, & Chen, 2013) or more water-soluble group. Glycidyltrimethylammonium chloride is used in synthesizing to improve the water solubility and antibacterial property of chitosan in this study.

So far, color is a principal aspect in wood industry for determining the degree of appreciation of consumer, meanwhile, wood color has to be all the wood ages long (Romagnoli et al., 2013). Reactive dye is commonly used in wood dyeing for its bright colors and good fixing ability after penetrating wood (Deng, Liao, & Hu, 2004; Wu, Yu, & Zhang, 2014). However, the weight of dye molecule is too small and run off easily from wood which loved by bacterial. Therefore, a different biopolymer dye synthesized with reactive dye and anti-bacterial chitosan would have a huge market and broad prospects in wood industry for particular environment like hospital, *etc.* But the chitosan and reactive dye could not dissolve in the same solution for producing sediment at any pH value.

Hence, the present investigation involves synthesizing chitosan quaternary ammonium salt and antibacterial chitosan biopolymer dye, meanwhile Fig. 1 shows the chemical equation (Perkins, 2004; Xu, Wang, Guo, Lei, & Tang, 2011; Xu, Xin, et al., 2011). In this chemical equation, the symbolic chemical formula $DYE{-}SO_3^-$ means that the $-SO_3^-$ group is contained in the dye molecule. The $-SO_3^-$ group could react with the $-N^+$. According to the stereo-hindrance effect and molecular structure, most of chitosan biopolymer dye molecules have only one reacted $-SO_3^-$ group per molecule, and there can be two reacted $-SO_3^-$ groups on one dye molecule in our products.

In this work, we employed an approach to combination and test the water solubility and antibacterial activity of products. We used Fourier transform infrared spectroscopy (FTIR), X-ray

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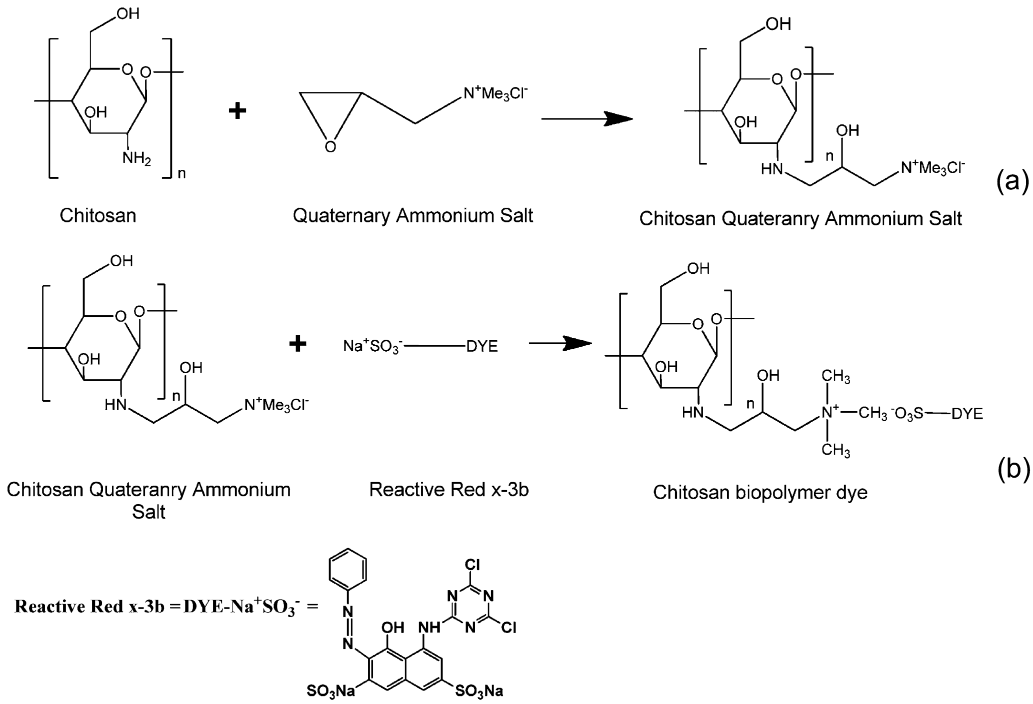

Fig. 1. Chemical reaction equations of chitosan quaternary ammonium salt (a) and chitosan biopolymer dye (b).

photoelectron spectrometer (XPS), X-ray diffraction (XRD) to prove the formation of chitosan based dye. We evaluated the antibacterial activity of chitosan biopolymer dye with JIS Z 2801:2000 control. The water solubility of dye was tested by UV–visible spectroscopy (UV–vis).

2. Experimental

2.1. Materials

Chitosan powder which had an 85% degree of deacetylation, glycidyltrimethylammonium chloride (liquid state) and commercial-grad reactive red x-3b (C.I. Reactive Red 2) which was suitable for textile and wood were used in this study. All other chemicals were analytical grade and supplied by Beijing Chemical Works.

2.2. Synthesis of chitosan quaternary ammonium salt

N-(2-hydroxyl)propyl-3-trimethyl ammonium chitosan chloride was prepared by a modified method according to references (Lim & Hudson, 2004; Mati-Baouche et al., 2014). Chitosan (6 g) was dispread in 240 mL distilled water under pH value of 6 at 85 °C. Glycidyltrimethylammonium chloride (5.6 g) was added dropwise. After 8 h of reaction with stirring, the resultant products were poured into cold acetone and kept in refrigerator for 24 h. Then, acetone was decanted and the remaining products were dissolved in methanol. The mixture was precipitated in 4:1 acetone–ethanol solution and the sediment was collected by filtration. After that sediment was washed with ethyl alcohol for 5 times. Finally, the resultant products were obtained by freeze-drying for 48 h.

2.3. Synthesis of chitosan biopolymer dye containing quaternary ammonium group

Chitosan quaternary ammonium salt (3 g) was dissolved in 230 mL distilled water and reactive red x-3b (0.4 g) was dissolved in 10 mL distilled water. Dye solution was inserted into chitosan quaternary ammonium salt solution dropwise with stirring. After stirring for 4 h at 40 °C, the mixture was precipitated in ethyl alcohol and extraction filtrated repeatedly until filtrate was colorless. Finally, the sediment was freeze-dried and pure quaternary ammonium salt chitosan dye was achieved.

2.4. Characterization

Fourier transform infrared spectroscopy (Nicolet 6700, Thermo Scientific, USA) in transmission mode, was used in the wavenumber range of 4000–400 cm^{-1} to analyze the linkage of chitosan, chitosan quaternary ammonium salt and chitosan biopolymer dye. The composition and structure of the chitosan, chitosan quaternary ammonium salt and chitosan biopolymer dye were analyzed by elemental analyzer (Vario EL cube, Elementar, Germany), X-ray Spectrometer (Axis Ultra, Kratosx, Japan) and X-ray diffraction (D/max2550HB+/PC, Rigaku, Japan). JIS Z 2801:2000 control was used to evaluate the antibacterial property against *Escherichia coli* and *Staphylococcus aureus* of birch veneers which were dyed with the chitosan biopolymer dye. Water solubility can be measured by UV–vis spectroscopy (Chen, Ou, Cheng, & Xie, 2011; Chi et al., 2006). In this study, the water solubility of chitosan biopolymer dye (w/v = 50 mg/20 mL) was measured in the pH values of 3, 5, 7, 9, 11 and 13 at λ_{max} of 540 nm while 0.25 wt.% reactive red x-3b solution whose concentration approximate to the chitosan biopolymer dye was used as reference.

3. Results and discussion

3.1. FTIR spectroscopy

Identification of the synthesis of chitosan quaternary ammonium salt and chitosan polymer dye was confirmed by FTIR. The FTIR spectra of chitosan and chitosan quaternary ammonium salt are shown in Fig. 2(a) and (b). Compared with chitosan (a), chitosan biopolymer dye (b) showed disappearance of $-NH_2$ at 1600 cm^{-1},

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