

6-carboxyl Preparation and Characterization of Chitin

Fang Xinlei^{1,2,a}, Xu Ran^{2,a}, Huang Junyun^{2,c}, Li Xiaofang^{2,d}, Yang Yan*,^{1,2,e},
Yang Jianhong^{1,2,f}

1.Changzhou Water&Wood Environmental Protection Technology Corporation,Changzhou
213016,China;

2. School of Environment&Safety Engineering, Changzhou University,Changzhou 213016,China

^aczfxinlei@163.com, ^b124973197@qq.com, ^chjyhy@vip.qq.com,

^d1042522887@qq.com, ^eyy129129@163.com, ^fyangshenyang3@163.com

Keywords: Chitin, modified, characterization

Abstract. Chitin has good biological activity, but the nature of the shell element is difficult to dissolve in water severely restrict its application in many fields. Through chemical modification, as the hydroxyl group to change its characteristics of different preparation of chitin, and were characterized by FT-IR and XRD on it, the results show that the modified alpha into beta chitin chitin success.

Introduction

Chitin has good biological activity. But the nature of the shell element is difficult to dissolve in water severely restrict its application in many fields. Through chemical modification can introduce different group to change its solubility in chitin. To make chitin can better application in the field of wound healing, makes it accord with wet therapy, can be in the induction of water-soluble chitin molecule group has the solubility in water. And representative of a water-soluble chitin is C-6 selective oxidation, including 6-carboxyl of chitin. 6-carboxyl of chitin, the hydroxyl on the C-6 oxidized to carboxyl, to introduce hydrophilic radical satisfies its water-soluble wet therapy.

Experiment method

The preparation of 6-carboxyl of chitin

6-carboxyl chitin preparation method is as follows: the 4g chitin dissolved in 200g 4% NaOH 8% urea aqueous solution, fully mixing into the freezer -20 degree centigrade after freezing. 12h after remove frozen chitin and stir until room temperature quickly, repeat 4 times after preparation of transparent chitin was homogeneous solution. Add it to 500ml of distilled water, after rapid mixing form micelle system, washing with distilled water to neutral. After the spread of chitin to 100ml of distilled water, after homogenizer homogeneous reaction.

Adding 4% sodium hypochlorite (w/v) solution of chitin, and put in the ice water bath, after adding sodium bromide and Tempo. And in the process of reaction, with the pH meter measuring the pH value and gradually HCl drip into the 4M and 0.1M NaOH to control the pH of around 10.8. Finally, add 5ml of alcohol termination reaction. The filtered solution dialysis desalination. Using silver nitrate test for a completely remove chloride ion. After the dialysis, dialysis fluid inside the reduced pressure concentration after freeze-drying 6-carboxyl chitosan powder.

Representation

Chitin because of containing large amounts of hydrogen bonding between molecules to make its high crystallinity. This nature makes the oxidation product of 6-carboxyl chitin rate is only 36%. Often use sulfuric acid[1].after pretreatment of chitin chitin crystal structure so that its crystal structure of alpha chitin into beta, chitin, so as to improve the yield. This topic using NaOH/Urea system freeze-thaw pretreatment of chitin to improve product yield. FT-IR, XRD and use to define

its crystal structure.

FT-IR

Samples after KBr tablet, adopting Avatar 370 type Fourier infrared spectrometer, scanning frequency range is 400 cm^{-1} -4000 cm^{-1} .

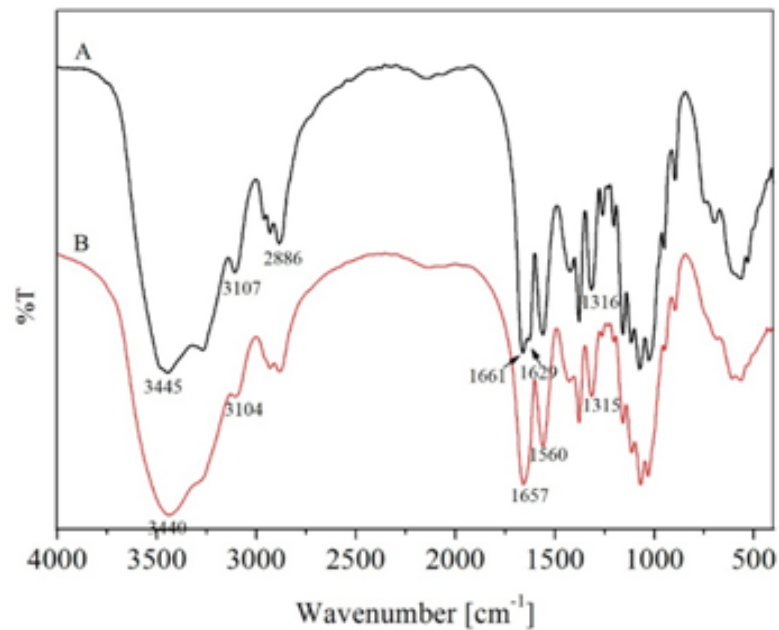


Fig.1 Original chitin A and chitin B after pretreatment of infrared spectrum

Fig.1 is the original chitin A, chitin and 6-carboxyl of chitin after pretreatment of carboxyl chitin infrared spectrogram. In A and B is one of the most obvious changes in the original chitin amide I band at 1661 cm^{-1} and 1629 cm^{-1} appeared two peaks, and treated with NaOH/Urea freeze-thaw chitin only appeared at the 1657 cm^{-1} A peak, it shows that after the pretreatment of chitin are by alpha into beta, also demonstrated the processed product molecular inter-atomic forces reduced[1]. In 1661 cm^{-1} , 1316 cm^{-1} and 1156 cm^{-1} of the peak represent the chitin amide I, II, III band, broad peak around 3445 cm^{-1} is hydroxyl stretching vibration absorption peak, peak wide that exists between the hydroxyl strong intermolecular and intramolecular hydrogen bonding[2,3].

XRD

Samples of the X-ray diffraction pattern of 20 degree centigrade use Lab XRD-6000-X-ray diffractometer (Shimadzu corporation Japan) record, Cu K alpha source, 50mA pipe 40Kv voltage, current, the DS/SS0.5°, scanning speed 4°/min (2θ), shooting range (2θ) to 5-50°.

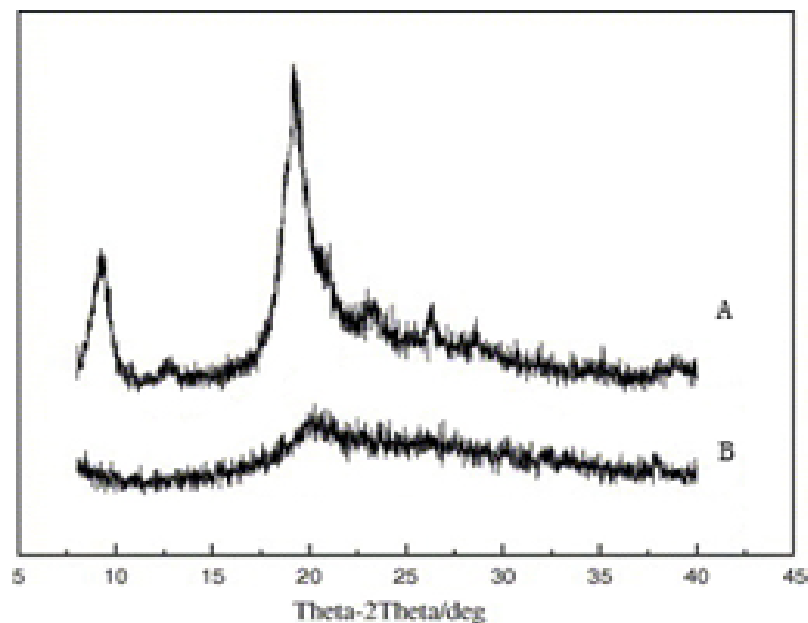


Fig.2 Chitin A and after the pretreatment of raw chitin B XRD diffraction pattern

The original chitin and after freeze-thaw treatment of chitin using XRD analysis of its crystal structure, the results are shown in fig. 2. The primary difference between A and B in figure 2θ 9.3, 12.4 and 19.1[4]. After NaOH/Urea freeze-thaw pretreatment in 9.3 and 19.1 in B 2θ almost disappeared, the peak and 12.4, in 2θ peak almost disappear. These show that chitin by NaOH/Urea freeze-thaw its crystal structure cause significant change after pretreatment, its crystal structure into beta successfully by alpha chitin.

Conclusion

(1) The NaOH/Urea chitin after freezing and thawing processing only appeared at the 1657 cm^{-1} peak, showed that after the pretreatment of chitin are by alpha into beta.

(2) Chitin by NaOH/Urea after freezing and thawing pretreatment peak at 9.3 and 19.1 2θ almost disappeared, and 12.4 2θ peak almost disappear. Show its cause significant change of crystal structure, its crystal structure into beta successfully by alpha chitin chitin.

Acknowledgements

This work was financially supported by Changzhou city science and technology support program (social development, Grant No.CE20125036).

References

- [1] L.Sun, Y.Du, J. Yang, et al. Carbohydrate Polymers. Forum Vol .66(2006),p.168-175
- [2] X.Hu, Y.Du, Y.Tang, et al. Carbohydrate Polymers. Forum Vol.70(2007),p.451-458
- [3] B.Focher, A.Naggi, G.Torri, et al. Carbohydrate Polymers. Forum Vol.17(1992),p.97-102
- [4] K.Vandavelde, P.Kiekens. Carbohydrate Polymers. Forum Vol.58(2004),p.409-416