

The study on the optimal amount of raw materials produced by the oxidation of Chitosan on the synergy HAc-H₂O₂

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Abstract. In this paper, industrial grade chitosan as the raw materials, HAc-H₂O₂ synergistic oxidative degradation method was used for degradation of raw materials, the influences in the reaction process with different chitosan dosage on the yield, the viscosity and the degree of deacetylation had been studied systematically. The results showed that the amount of 8% is the optimum reaction amount to the degradation of chitosan dosage.

Introduction

HAc-H₂O₂ synergistic oxidative degradation of chitosan acetic acid solution is to dissolve in a certain concentration, adding a certain amount of hydrogen peroxide, reaction at a certain temperature for a certain time, then sodium hydroxide solution is used to adjust the pH value. In the end, the absolute ethanol with 3~5 times was used for the chitosan precipitation, drying to obtain the target product, method of collaborative HAc-H₂O₂ the oxidative degradation of chitosan has the advantages of easy processing, low cost, no residue and other advantages, these features are easy to realize industrialization of environmental protection. Through the collaborative oxidation of industrial grade chitosan, this paper strives to find a high yield, low viscosity, high degree of deacetylation of Chitosan Consumption.

The experimental materials and methods

Experimental apparatus and equipment.

Table 1 Experimental apparatus and equipment

Instrument	Model	Manufacturer
Electronic balance	AL204	Mettler Toledo Instruments Co. Ltd.
Heat collector heated magnetic stirrer	DF-101S	Gongyi Yuhua Instrument Co., Ltd.
Vacuum drying oven	Type DZF-6050	Shanghai Heng Heng Technology Co., Ltd.
pH Meter	ZD-2	Shanghai Hongyi instrument and Meter Co., Ltd.
Rotary viscometer	NDJ-1 type	Shanghai Hengping Scientific Instrument Co. Ltd.
Ultrasonic cleaner	SB-5200D	Ningbo Xinzhi biological Polytron Technologies Inc

Experimental drugs.

Table 2 Experimental drugs

Drug Name	Specifications	Manufacturer
Chitosan	The degree of deacetylation is about 80%	A Dalian company of Chitosan
30% H ₂ O ₂	AR	Tianjin Kermel Chemical Reagent Co., Ltd.
Glacial acetic acid	AR	Tianjin Hengxing Chemical Reagent Factory
Hydrochloric [chlorhydric] acid	AR	Tianjin Damao Chemical Reagent Factory
Caustic soda	AR	Tianjin city Dongli District Tianda Chemical Reagent Factory
Absolute ethyl alcohol	AR	Tianjin Damao Chemical Reagent Factory
Sodium acetate	AR	Tianjin Damao Chemical Reagent Factory
Mixed indicator of methyl orange and aniline blue	(1g/L water soluble Methyl Orange: 1g/L water soluble aniline blue =1:2 volume)	Laboratory preparation

Experiment methods.

(1) Electronic balance is used to accurately weigh a certain amount of industrial grade chitosan, added slowly to a certain concentration of acetic acid solution in the beaker, stirring constantly in the joining process, after the chitosan acetic acid solution mix well, adding once H₂O₂ needed in the experiment and put in the ultrasonic Cuncussion instrument in ultrasonic vibration 2-3 min.

(2) The magnetic vibrator in a beaker, wrap up the mouth of the beaker, and will have to set up the beaker in the water bath heating constant temperature magnetic stirrer with heating temperature specified in the rotor speed is adjusted to 120r/min, constant temperature reaction time.

(3) After the reaction was finished, the beaker was removed and the pH value of the reaction liquid was adjusted to 7.2-7.4 by 2mol/L NaOH solution.

(4) For cooling the reaction liquid to less than 50 DEG C, impurities is filtered from the reaction solution, then about 3-5 times the volume ratio of ethanol applied to the clear liquid to precipitate material.

(5) Filter again, obtained precipitate.

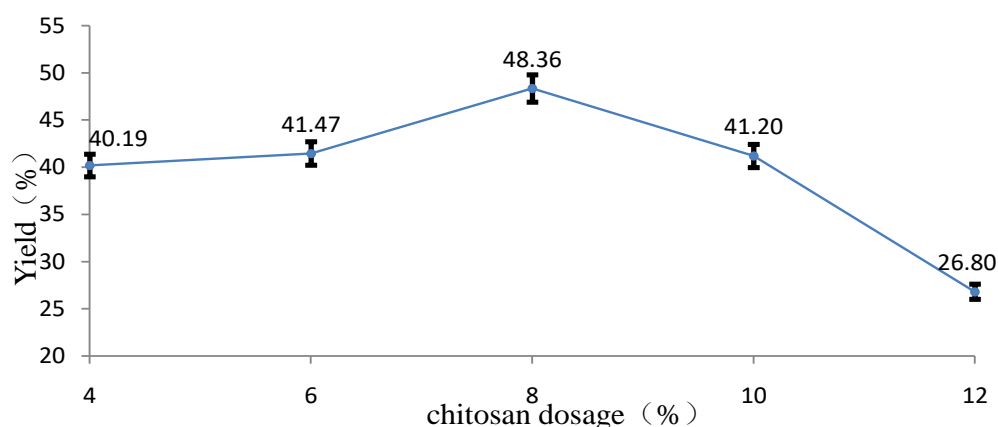
(6) Deionized water is used to wash the precipitate repeatedly to neutral; finally the precipitate has been dried for 12h in the drying oven at 60 degrees. grinding the precipitate dried, water-soluble chitosan oligosaccharide is obtained.

Results and discussion

On the condition that the temperature is 80°C, the concentration of acetic acid is 1%, hydrogen peroxide concentration is 8%, the reaction time is 3h, the only change is the amount of chitosan, with the requirements that the product should with high yield, low viscosity, high degree of deacetylation, optimum dosage of chitosan in HAc-H₂O₂ synergistic oxidative degradation of chitosan is obtained.

Table 3 Effect of chitosan dosage on the reaction

w (%)	clarity	Reactive liquid color	finished color
4	Slightly turbid	yellow	popcorn
6	Slightly turbid	bronzing	yellow
8	Slightly turbid	bronzing	brown
10	muddy	brown	brown
12	muddy	brown	brown

Effect of chitosan dosage on Yield.**Fig.1** Effect of chitosan dosage on the yield

Analysis of Figure 1 shows that under the condition that the only change is the dosage of chitosan, the yield increased with the increasing amount of chitosan, reached the maximum when the dosage is 8%, and then began to decline. When the dosage of chitosan reached 10%, mixing excessive chitosan in acetic acid solution has become more difficult, can not be completely soluble in The reaction solution, In the degradation process ,chitosan can not fully contact with the oxidant, the reaction is not sufficient, the yield will be reduced.

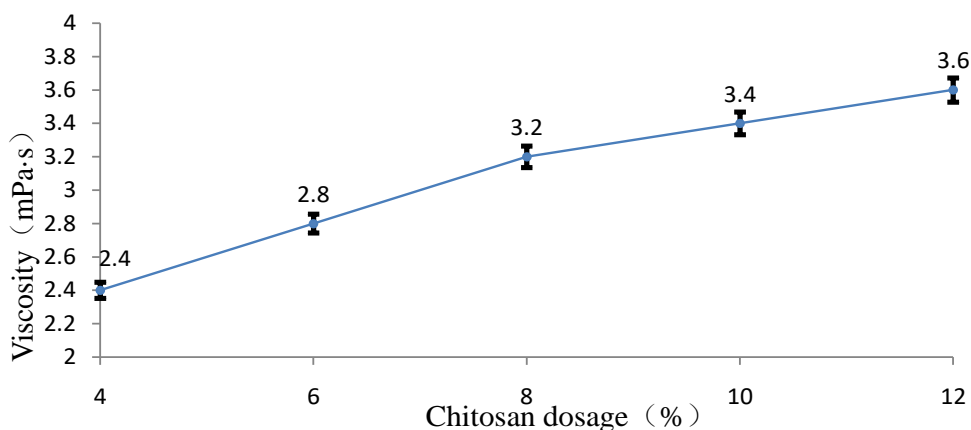
Effect of chitosan dosage on viscosity.**Fig.2** Effect of chitosan dosage on viscosity

Figure 2 analysis showed that the only change in the amount of chitosan under the conditions, the viscosity increased with the increasing amount of chitosan, when the dosage was 4%. The viscosity reaches the minimum value. The less dosage of chitosan ,the more soluble in acetic acid solution ,the more fully contact with oxidizing agent reaction , chitosan with lower viscosity can be degraded with fully reaction.so viscosity increase with the increased of chitosan dosage . the amount of molecular weight increases with the increase of chitosan dosage as well.

Effect of chitosan dosage on deacetylation degree

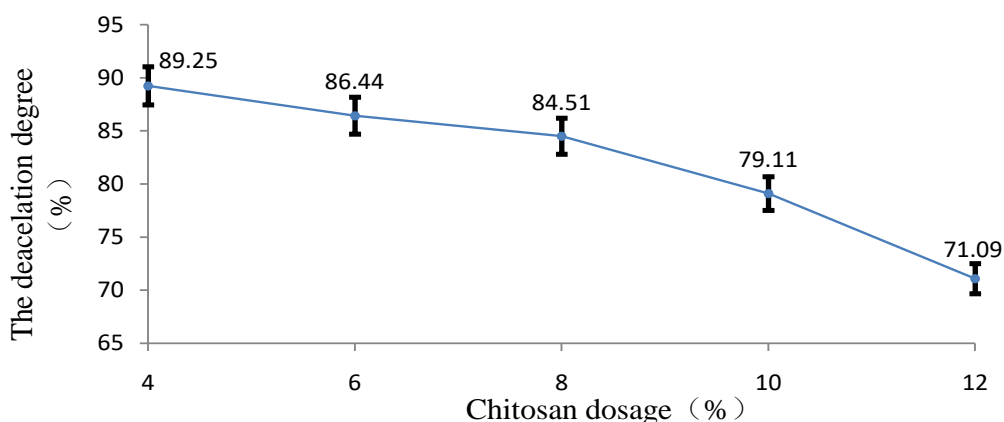


Fig.3 Effect of chitosan dosage on degree of deacetylation

Figure 3 analysis showed that under the conditions that the only change is the amount of chitosan, the degree of deacetylation decrease with the increased of chitosan dosage ,when the dosage was 4%,the degree of deacetylation Reaches the maximum value.The less dosage of chitosan ,the more soluble in acetic acid solution ,the more fully contact with oxidizing agent reaction. chitosan with highest degree of deacetylation can be degraded with fully reaction.So the degree of deacetylation decrease with the increased dosage of chitosan.

Conclusions

The experimental results indicated that when the dosage is 6%, while viscosity quality index and the degree of deacetylation of quality indicators are more remarkable than the dosage of 8% but the yield in 6% dosage is far less than of 8% dosage;And when in 10% dosage , the viscosity, degree of deacetylation and yield are all less than 6%.Synthesizing several kind of situation,the amount of 8% is the most suitable dosage.

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