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Preservation of Chitosan Aqueous Gel under Neutral Conditions

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Abstract

An acid smell free chitosan neutral hydrogel was prepared by the addition of sodium hydroxide aqueous solution to chitosan acetic acid salt solution and by the extensive rinse with distilled water to remove sodium acetate. This resulted in chitosan hydrogel that could dissolve quickly by the addition of a minimum amount of acids such as hydrochloric acid, acetic acid and so on. Chitosan solution became a transparent solution without any filtration following to the addition of acid to chitosan neutral hydrogel. The chitosan solution obtained by the addition of hydrochloric acid or acetic acid progressed its stability of viscosity for around 300 hours probably due to luck of free acid to hydrolyze glycoside linkage.

Introduction

Though chitosan becomes water-soluble following the formation of salt with organic acids, Shamov *et al.*, (2002), it is hard to maintain its molecular weight constant for long standing at room temperature. But the stability of chitosan aqueous solution was found to be fairly stable in spite of less water solubility of chitosan-HCl salt than chitosan-acetic acid salt. But the dissolution of chitosan was achieved quickly by the addition of concentrated hydrogen chloride, when chitosan neutral hydrogel was applied which was prepared by vigorous mechanical agitation of chitosan precipitate following to neutralization of chitosan-acetic acid aqueous solution.

The chitosan neutral hydrogel was found to be stable for two more weeks at room temperature and a clear, viscous chitosan-HCl or chitosan-acetic acid aqueous solution was prepared immediately after the addition of acids without any filtration procedure. On the comparison of stability of both chitosan aqueous solutions at room temperature for long standing, viscosity of chitosan-acetic acid aqueous solution was faster to reduce than that of chitosan-HCl aqueous solution.

On the deodorization of chitosan aqueous solution, there was little acidic smell of chitosan aqueous solution probably due to complete neutralization and dialysis.

Since resulted chitosan neutral hydrogel was confirmed to maintain its antimicrobial activity, predominantly biomedical applications of chitosan hydrogel would be expected in the near future.

Experimental

Chemieals: Chitosan was kindly donated from the Koyo Chemical Co. Ltd. The degree of deacetylation was 82% and molecular weight was estimated by viscometric measurement to be 1.6×10^6. Other reagents of reagent grade were purchased from Wako Pure Chemical Co. Ltd. and used without further purification.

Preparation of chitosan neutral hydrogel: 10 g of chitosan powder was dissolved in 200 ml of 4w/v% acetic acid aqueous solution followed by filtration to remove insoluble material. The chitosan solution was then brought to pH 10 by the addition of 10w/v% sodium hydroxide aqueous solution at room temperature. The precipitate was collected by filtration followed by extensive rinse.

Viscosity: Viscosity measurement was achieved by applying an Ubbelohde type viscometer at 25°C. The molecular weight of chitosan was estimated by applying the viscosity equation.

Results and discussion

Chitosan neutral hydrogel: The molecular weight of chitosan was found to be stable for fairly long periods even at room temperature when chitosan was in hydrogel form. The stability was prolonged by sterilization applying autoclave treatment. The solubility of chitosan was remarkably enhanced by hydrogelation due to a minimum amount of acid was requested to dissolve, especially solubility for hydrochloric acid aqueous solution was enhanced remarkably. Only 0.3 to 0.5 moles of hydrochloric acid or acetic acid was requested to dissolve chitosan hydrogel against large amount of acetic acid to dissolve chitosan powder. A similar treatment was required to dissolve chitosan powder by hydrochloric acid aqueous solution.

On the other hand, clear chitosan solutions were obtained immediately after the addition of minimum amount (less than 1:1 molar ratio against mole of amino group) of acid without filtration procedure. Resulted chitosan solutions were fairly stable on standing for 270 hours at room temperature. Acetic acid seemed to be unstable factor of chitosan aqueous solution probably due to the association effect of hydroxyl group of sugar residue with carbonium ion of acetic acid of salt formation member with amino group. The pKa of chitosan was estimated both to be 6.4 through the titration of chitosan hydrogel by hydrochloric acid and acetic acid on the degree of transpearency of chitosan solution.

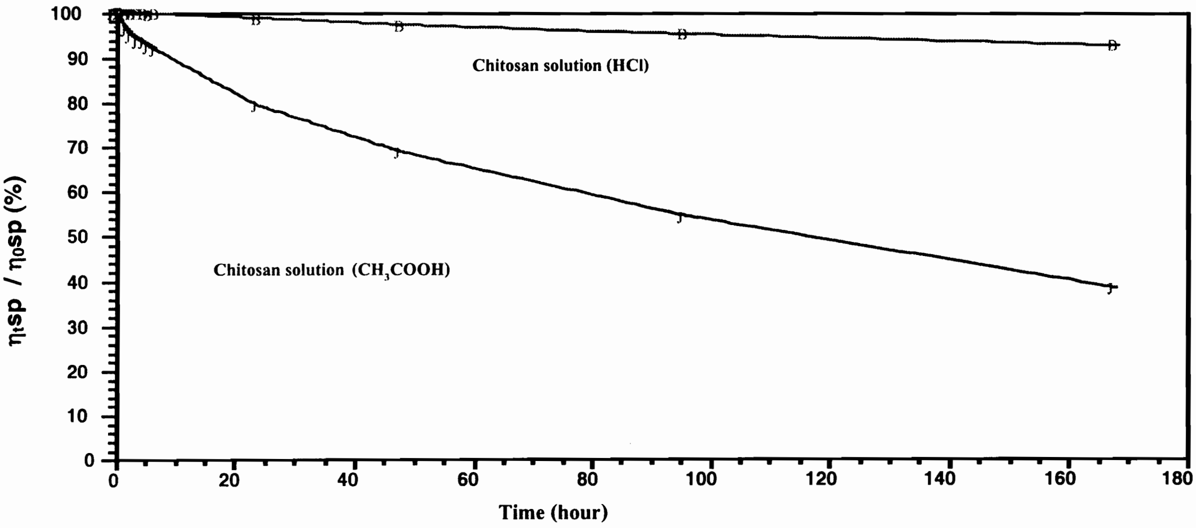

Figure 1. Time dependent viscosity changes of chitosan aqueous acetic acid solution (pH 3.85) prepared from chitosan powder and 4v/v% aqueous acetic acid. Chtosan solution (HCl) was prepared from chitosan neutral hydrogel by the addition of 0.5 ml of concentrated hydrochloric aid (pH 5.24).

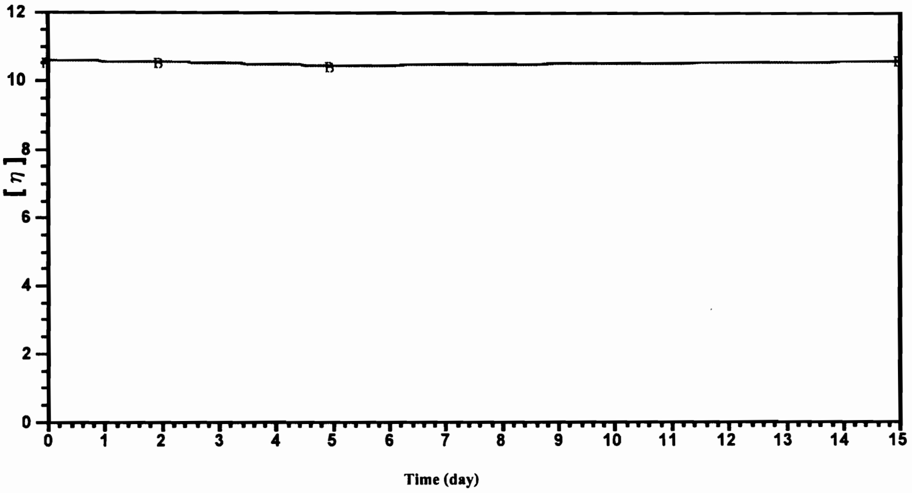

Figure 2. Stability of the intrinsic viscosity of chitosan aqueous hydrochloric solution which was extracted from preserved chitosan neutral hydrogel at room temperature.

The stability of the chitosan molecule was confirmed by the stability of intrinsic viscosity as shown in Figure 2 when chitosan hydrogel was kept at room temperature for 15 days in sealed glassware even if without any pasteurization.

One of the predominant advantages of chitosan hydrogel would be the long standing at room temperature for long periods without reduction of molecular weight and preparation of smooth chitosan membrane directly from hydrogel. As chitosan hydrogel was found to be effective for wound healing. A mixed hydrogel of chitosan with chitin hydrogel would be expected of wider application for various degrees of wound healing.

Conclusions

Chitosan acid salt aqueous solution has been investigated to progress the stability in aqueous solution on standing for long period at room temperature and to remove acid smell from chitosan solution or chitosan products, because chitosan aqueous solution was limited to applications by these defects.

Acknowledgements

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