



Synthesis, characterization and application of novel bio-polymeric Schiff base from chitosan and 5-chloro-3-methyl-1-phenyl-1H-pyrazole-4- carboxaldehyde

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Abstract

Schiff base of chitosan and 5-chloro-3-methyl-1-phenyl-1H-pyrazole-4-carboxaldehyde was prepared by reaction of chitosan with 5-chloro-3-methyl-1-phenyl-1H-pyrazole-4-carboxaldehyde in the presence of acetic acid as a solvent. Some reaction parameters have been investigated such as temperature effect, time effect and mol ratio and optimize the product. The product has been ascertained by FT- IR spectroscopy. Antifungal activities of the Schiff base against *Aspergillus Niger* (A. Niger) and *Penicillium* were measured through the agar well diffusion method.

Keywords

Chitosan, Schiff base, reaction parameters, characterization, antifungal activity.

INTRODUCTION

Chitin is natural polysaccharide usually obtained from the exoskeletons of shellfish and insects. Although, chitin is the second most abundant natural polysaccharide next to cellulose. It is a copolymer of 2-acetoamido-2-deoxy-D-glucose (N-acetyl-glucosamine, GluNAc) and 2-amino-2-deoxy-D-glucose (N-glucosamine, GluN) units randomly or block distributed throughout the biopolymer chain depending on the processing method used to derive the biopolymer. When the degree of deacetylation of chitin reaches about 50% (depending on the origin of the polymer), it becomes soluble in aqueous acidic media and is called chitosan. The solubilisation occurs by protonation of the -NH_2 function on the C-2 position of the D-glucosamine repeat unit, whereby the polyelectrolyte in acidic media. Chitosan is the only pseudo natural cationic polymer and thus, it finds many applications that follow from its unique character. Since, last decade so many modifications have been carried out due to presence of free -NH_2 and -OH groups on chitosan. Such as preparation of complexes, drug delivery, coating hydro gel, cross linking polymer, Nano particles and Nano composites, and quaternary salt. Due to presence of both reactive amino and hydroxyl groups that can be used to chemically alter its properties under mild reaction conditions. Among these substituted biopolymers there are the Schiff's bases obtained by the reaction of free amino groups of chitosan with an active carbonyl compound such as Aldehyde and ketone. In the present work, we prepared bio-polymeric Schiff base of chitosan and 5-chloro-3-methyl-1-phenyl-1H-pyrazole-4-carboxaldehyde and it has been ascertained by FT-IR spectroscopy. Antifungal activities of synthesized Schiff base were investigated against *Aspergillus Niger* (A. Niger) and *Penicillium* were measured through the agar well diffusion method.

MATERIALS AND METHODS

Chitosan was kindly supplied as a gift sample by Mahtani chitosan Pvt. Ltd., Veraval, Gujarat. The degree of deacetylation was 90%. 3-Methyl-1-Phenyl-2-pyrazolin-5-one was purchased from Aldrich Chemical Co. and used without further purification. Phosphorous oxychloride (S.D.Fine Chemicals Ltd. Bombay) was used as received. Acetic acid, dimethyl form amide (DMF) and ethanol was supplied by S.D.Fine Chemicals Ltd. Bombay. Dimethyl formamide was distilled at $152\text{--}154^\circ\text{C}$ and used. Analytical grade acetic acid (Qualigens, Glaxo

India Ltd.) was used as received. Absolute ethyl alcohol of a high degree of purity (99.5 %) was supplied by S.D.Fine Chemicals Ltd. Bombay. Ammonia (40%, S.D.Fine Chemicals Ltd. Bombay) was used as received.

RESULTS AND DISCUSSION

The synthesized bi-polymeric Schiff base is light yellow to yellow coloured solid materials. The formation of bi-polymeric Schiff base was confirmed by FT-IR spectra of chitosan and Schiff base. The main evidence of Schiff base formation is certainly the appearance of strong absorption band at 1634 cm^{-1} attributed to the -C=N vibration characteristic of imines which is not observed in chitosan. The characteristic absorption peak for -NH_2 group at 1592 cm^{-1} decrease in its intensity due to the decrease in its content indicating that the reaction on amino groups in chitosan with aldehyde to give Schiff base. On the other hand there is no evidence of bands characteristic of free aromatic aldehyde near to 1680 cm^{-1} .

Conclusion

The Schiff base of chitosan was synthesized by the reaction of chitosan with 5-chloro-3-methyl-1-phenyl-1H-pyrazole-4-carboxaldehyde. The optimized reaction condition obtained in the preparation of bi-polymeric Schiff base of chitosan and 5-chloro-3-methyl-1-phenyl-1H-pyrazole-4-carboxaldehyde is Chitosan : 0.5 g (dry basis), [Acetic acid] : 0.15 mol/L [mol] : 6.00:1.00, Time : 18h, Temperature : 450°C . The value of maximum percentage yield, in the case of bi-polymeric Schiff base of chitosan and 5-chloro-3-methyl-1-phenyl-1H-pyrazole-4-carboxaldehyde is found to be 69.71%. The antifungal activities of chitosan and Schiff base of chitosan were investigated against *Penicillium* and A. Niger and the result indicates that the Schiff base of chitosan has better antifungal activities than chitosan. The antifungal activity of Schiff base increases with an increase in concentration. As a novel chitosan derivative, the Schiff base of chitosan improves the antifungal activity of chitosan and expands the antifungal spectrum compared with chitosan itself.

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