FTIR Spectroscopy XRD, SEM EDX and AFM Studies On Natural Biomaterial

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Abstract: Chitosan is a natural polysaccharide next to cellulose occurring in nature. It consists of a linear (1-4) linked 2 amino 2 deoxy D glucan. It can be chemically prepared from chitin by treatment with alkali at elevated temperature. Chitosan is non toxic, biodegradable and biocompatible polymer. Chitosan is widely existing in nature and has antibacterial effect, heavy metal adsorption effect, anti-oxidation effect and film formability. Chitosan forms viscous solution in various organic acids. These viscous solutions have been used to make functional films. They were readily biodegradable. The main objective of the present research is to develop and a nanocomposite of chitosan films in terms of surface morphology. The effect of plasticizer was also studied. The plasticizer increased the smoothness of surface. The smoothness was confirmed by SEM EDX, AFM studies. The interaction of chitosan and plasticizer was confirmed by FTIR studies and XRD.

Key words: Chitosan; nano composite; glycerol; Films; Plasticizer.

Introduction and Experimental

Chitosan is an abundant heteropolysaccharide obtained from deacetylation of chitin. Chitin is polysaccharide of natural origin and second most abundant next to cellulose. Over the past few years a renewed and increasing interest on the biomass resources for the development of thenew materials has been observed. This is due to scarcity of fossil resources and also of environmental problems associated with their continuous use during the last century considerable interest to researchers’ lies in the cytocompatible, hemocompatible, nontoxic, adsorption filmogenic properties of (1-4). These properties are attracting interest for use in pharmaceutical, biomedical fields, biotechnology, waste water treatment, cosmetic and food industry. However, the films are rigid and need plasticizers to reduce frictional forces between polymer chains such as hydrogen bond or ionic forces to improve mechanical properties (5). The incorporation of polyols in the formation of film can overcome this draw back and keep film stable during the required time. This current research deals with
blending of chitosan with glycerol, preparation of chitosanglycerol film. The blends were characterized using FTIR, SEM, EDX, AFM and XRD methods.

Materials and Method

Chitosan was purchased from panacea Biotech. Glycerol and other reagents used are of AR grade.

Preparation of nanocomposite

The chitosan nanocomposite films were prepared by solvent evaporation technique. Film forming solution of 1% W/V was prepared by dispersing chitosan flakes in 0.1M Acetic acid. 2ml Glycerol is added as plasticizing agent and then stirred with a sterile magnetic stirrer for 24 hour. Film forming solution was then poured on sterile polystyrene plate and left to dry at room temperature on previously leveled surface until the total evaporation of solvent. All films were peeled off after addition of 4N NaOH and were stored in desiccators for further use.

Characterization

The above prepared nanocomposite is subjected to FTIR, XRD, SEM EDX, AFM studies.

Results and Discussion

FTIR

IR Spectra of chitosan showed a strong absorption band OH stretching and NH stretching. The main absorption peaks of pure chitosan was observed at 1651 Cm$^{-1}$ was due to carbonyl stretching vibration of acetamide group, 1558Cm$^{-1}$ amide I 1592Cm$^{-1}$ amino group the 1377 Cm$^{-1}$ assigned to CN stretching. Peak observed at 1651 Cm$^{-1}$ shifts to higher wavelength 1667 Cm$^{-1}$ confirmational change of film when two or more substances are mixed physical blend vs. chemical interaction are reflected by changes characteristics structural peaks.

XRD studies

The change of chitosan structure after the addition of glycerol was investigated by means of XRD which is proven tool to study crystal lattice arrangements and yields very useful information on degree of sample crystallinity. The XRD pattern in this study displayed sharp peak at $2\theta=20^\circ$ revealing crystallinity of chitosan. After the addition of glycerol two peaks ($2\theta=30^\circ$ & $40^\circ$) were seen. The intensity of the peak is less when compared to one without glycerol.

![Figure 1 FTIR spectrum of nanocomposite](image1.png)  
![Figure 2 XRD pattern of nanocomposite](image2.png)

SEM
The microstructure obtained by SEM for the chitosan nanocomposite prepared by solvent casting showed that particles are relatively well dispersed. Figure (3) showed that the chitosan plasticized one was so smooth when compared to without glycerol. In the case of EDX there is no change in composition.

**AFM**

The surface morphology of the film was al inspected by AFM. It is a characterization technique which presents very high possibilities of application in the field of microscopy observation and characterization of various surface. 2D &3D images of nanocomposite were shown in figure(4). The nodules are seen as bright high peaks whereas the pores are seen as dark depressions.

**Conclusion**

The results obtained revealed that chitosan and glycerol are fully miscible blend, the miscibility can be achieved if there is interaction between the two compounds. The interaction is confirmed by FTIR&SEM. The micrographs of surfaces of chitosan without glycerol showed roughness while that with glycerol showed smooth surface. We can conclude that films made with chitosan and glycerol has wide range of industrial applications which depends on the knowledge of the physical and chemical properties of the materials used.

**References**


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