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# Synthesis and Characterization of Glutaraldehyde Crosslinked Ternary Beads of Gelatin

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## Abstract

In the current research work, the ternary blended sodium alginate/neem leaf powder/gelatin beads crosslinked with glutaraldehyde were prepared in 2:1:1 ratio. The characterization of the prepared glutaraldehyde cross linked ternary bead was carried out by advanced analytical techniques such as Fourier transform infrared spectroscopy (FT-IR) and X-ray diffraction (XRD) studies. Results of FTIR analysis determined the polymers functional groups, chemical structure of the glutaraldehyde cross linked ternary blended gelatin microspheres, formation of cross links and verified the synthesis of sodium alginate/neem leaf powder/gelatin beads. The reduction in crystallinity of prepared samples was identified from XRD studies and these observed overall results confirm the formation and compatibility of the prepared beads.

**Keywords**

Sodium alginate, neem leaf powder, gelatin, glutaraldehyde, cross linking.

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## INTRODUCTION:

Recently, due to its non toxic, biodegradable and biocompatible nature, the natural polymers have been studied as biological and biomedical resources.[1] In recent years, natural polymers such as konjac glucomannan [2], chitosan [3] and gelatin [4] were remained attractive primarily by many researchers and this was due to its economical, readily available, potentially degradable and compatible nature. Gelatin is a biodegradable natural polymer which can be used to produce microspheres and in order to provide its use in long term applications, certain improvements such as crosslinking reactions are necessary since it possesses aqueous solubility and limited mechanical and thermal properties. [5,6]

Actually, the sodium alginate is a family of linear polysaccharide which is composed of repeating α-L glucuronic acid and β-D-mannuronic acid monosaccharide units, each containing a carboxylate ion. The physical properties of the polymer get affected by the addition of cross links and this will depend upon the degree of cross linking, presence and absence of crystallinity.[7] Neem (*Azadirachta indica*) is the most versatile, multifarious trees of tropics, with immense potential commonly called 'India Lilac' or 'Margosa' which belongs to the family *Meliaceae*,

subfamily *Meloideae*, tribe *Melieae* [8] and this has been reported to have various clinical applications like antibacterial, [9,10] antiviral,[11] anticancer[12] and antidiabetic[13] properties.

The modification of biopolymeric material by means of blending with other polymers was found to be one of the convenient and effective methods to improve physical properties for practical utilization. Another important strategy utilized to improve the performance of the blends is the cross-linking treatment which has raised the attention of both the industrial and the academic world. [14,15] Beads possess attractive properties and applicability and hence have secured a unique position in various applications. Due to its ease of tendency to accumulate in an inflamed area of body, nano- or micro-beads have acquired great importance.[16]

Hence based on literature survey in the present study, we report on solution blending of sodium alginate, neem leaf powder and gelatin with cross linking agent glutaraldehyde which made it in the form of bead. Followed by this, the subsequent characterization of the ternary blended beads using FTIR, XRD studies were done and the obtained results were discussed.

## MATERIALS AND METHODS:

### Materials:

Neem belongs to the *meliaceae* family and is native to Indian sub-continent. Its seeds and leaves have been in use since ancient times for a variety of applications. In the present investigation, from the nearby area, the mature neem leaves used in the present investigation were collected and they were washed thrice with water to remove dust and water-soluble impurities. These collected neem leaves were dried until the leaves become crisp and after this process is over, the dried leaves were powdered. Certain chemicals namely sodium alginate and gelatin were obtained from Nice Chemicals private limited, Chennai and all the chemicals utilized in the present study were of AR grade.

### Preparation of glutaraldehyde cross linked sodium alginate/neem leaf powder/gelatin beads.

The glutaraldehyde cross linked sodium alginate/neem leaf powder/gelatin bead was prepared by the facile method as follows: Initially a homogeneous solution of gelatin was prepared by dissolving 1g of gelatin powder in minimum amount (10ml) of distilled water. To the above prepared homogeneous gelatin solution, about 1g of powdered neem leaf dissolved in minimum amount (10ml) of distilled water and 2g of sodium alginate dissolved in minimum amount (10ml) of distilled water was added and stirred effectively at room temperature for a period of 30 minutes. After this process is over, in order to perform the cross-linking process about 5 ml of the cross-linking agent was added to the ternary blended mixture and this ternary blended mixture was then added dropwise into cold water by using a syringe to form the ternary beads of gelatin. Finally, this glutaraldehyde cross linked sodium alginate/neem leaf powder/gelatin beads were filtrated out, dried and then stored in tight container for further use.

### CHARACTERIZATION:

The FTIR spectra of the prepared glutaraldehyde cross linked sodium alginate/neem leaf powder/gelatin beads were recorded in the spectral range of 400–4000 $cm^{-1}$ using 200 FT-spectrophotometer and the X-ray diffraction patterns of ternary beads of gelatin were analyzed using an SHIMADZU X-ray diffractometer using a Ni-filtered Cu-Kα radiation source power 40 kW, the Bragg's angle (2 θ) in the range of 5-80º and scanning speed of 2θ = 10/min.

## RESULTS AND DISCUSSION:

### FT-IR studies:

Fourier transformation is a spectrum of the signal at a series of discrete wavelength. This FT-IR study confirms the cross linking of sodium alginate/neem leaf powder/gelatin beads by glutaraldehyde and as well as to confirm the presence of certain chemical cross linkings between the three added components namely sodium alginate, gelatin, neem leaf powder and the cross-linking agent glutaraldehyde. The FT-IR spectral details of sodium alginate/gelatin/neem leaf powder bead were represented in **Figure-1.**

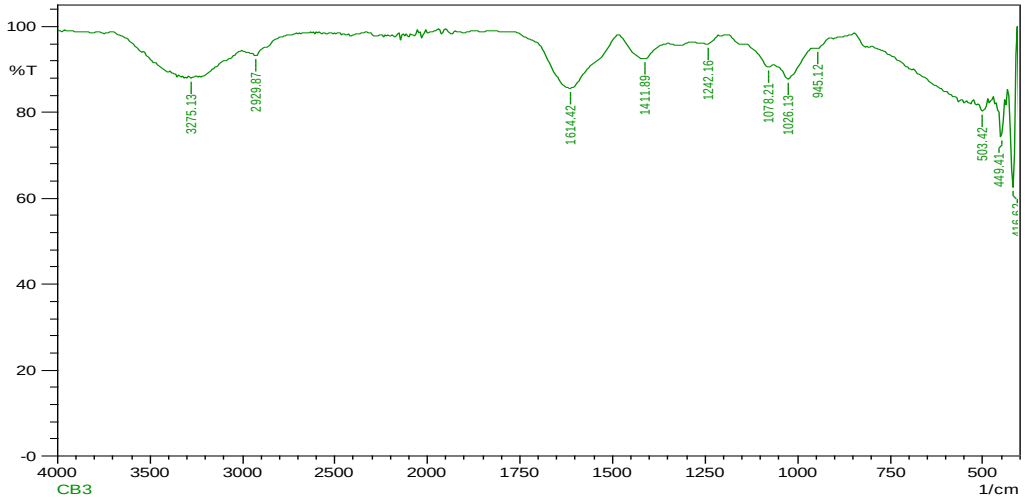


**Figure.1 FT-IR Spectrum of glutaraldehyde cross linked sodium alginate/neem leaf powder/gelatin beads**

The FT-IR spectrum of glutaraldehyde cross linked sodium alginate/gelatin/neem leaf powder beads shows an absorption peak at around 3275.13cm$^{-1}$ which is assigned to the stretching vibration of hydroxyl group with strong hydrogen bonding as intra- and/or intertype [17,18] and NH stretching of secondary amide C=O stretching in gelatin.[19] The band at 2929.87cm$^{-1}$ is mainly attributed to the aliphatic CH stretching in methylenic group. A strong absorption band observed at 1614.42cm$^{-1}$ is assigned to the C=N stretching and this obtained peak concludes the cross linking of the sodium alginate with the cross linking agent glutaraldehyde.[20] Certain strong bands obtained at 1411.89cm$^{-1}$ 1242.16 cm$^{-1}$,1078.21cm$^{-1}$1026.13cm$^{-1}$,945.12cm$^{-1}$and 503.42cm$^{-1}$ was mainly attributed to the OH bending in alcohols, C-N stretching in amines, C-O stretching in alcohols, C-O-C linkage, assymetrical C-O-C stretching in epoxide and C-C bending [21] respectively. From the above obtained FT-IR results it was concluded that the three materials namley sodium alginate, gelatin and neem leaf powder were blended effectively in the bead formation.

**X-ray diffraction studies:**

One of the rapid analytical techniques primarily used for phase identification of a crystalline material is the X-ray powder diffraction (XRD) technique which can provide information on unit cell dimensions. **Figure-(2)** represents the X-ray diffraction patterns of sodium alginate/gelatin/neem leaf powder beads prepared in the presence of the crosslinking agent glutaraldehyde.

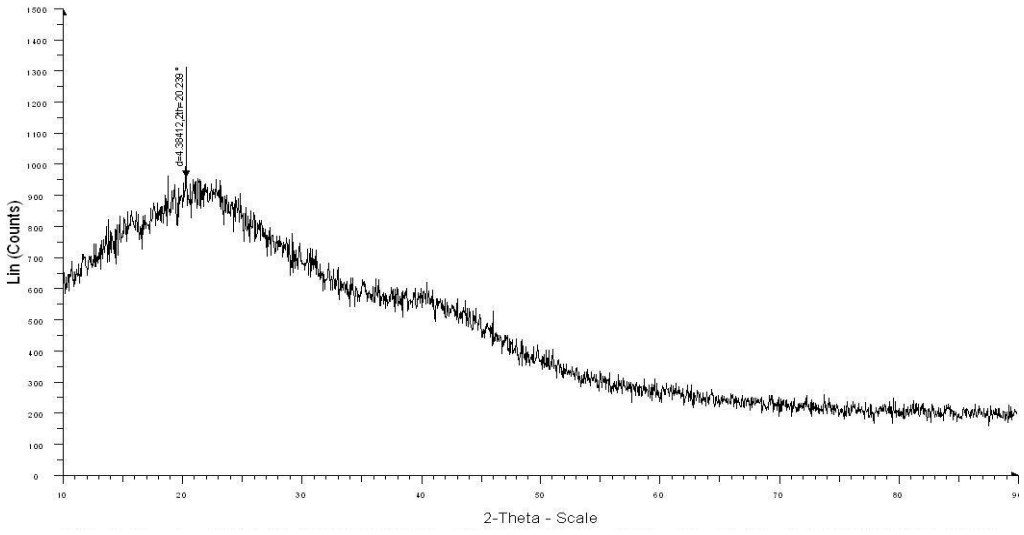


**Figure-2: X-ray diffractogram of glutaraldehyde cross linked sodium alginate/neem leaf powder/gelatin beads**

The obtained results of XRD pattern indicate that the prepared glutaraldehyde cross linked sodium alginate/gelatin/neem leaf powder beads shows a broad peak and possesses lower degree of crystallinity value. This decrease in crystallinity of the glutaraldehyde cross linked sodium alginate/gelatin/neem leaf powder beads is mainly attributed to sodium alginate, gelatin and neem leaf powder interactions, where the polysaccharides could form networks with neem leaf powder and therefore, the resultant beads were considerably more amorphous.[22] The diffraction pattern with a broad amorphous peak indicated that there was a molecular miscibility and interaction between the components.[23] These results concluded that the glutaraldehyde cross linked sodium alginate/gelatin/neem leaf powder beads has more amorphous nature which will be suitable for the adsorption process.

**CONCLUSION:**

In this study, the glutaraldehyde cross linked ternary sodium alginate-based hydrogels was prepared successfully with neem leaf powder and gelatin. The goal of this paper is to achieve a comprehensive understanding of the molecular interactions between the three added components namely sodium alginate, gelatin and neem leaf powder. The observed results suggest that there is strong interaction between the molecular chains of sodium alginate, neem leaf powder and gelatin, which may lead to the miscibility at specific ratios of the three components blended. Also from the FTIR results, it was evident that certain new peaks were observed due to the various functional groups (NH, C=O stretching in acids, C=N) and this shows the effective binding between the added component and the cross linking agent glutaraldehyde in blend formation. The highly amorphous nature of the glutaraldehyde cross linked sodium alginate/gelatin/neem leaf powder beads was elucidated from XRD studies respectively and these new findings suggest that the prepared sample can have a new path for further research in the wastewater treatment.

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