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Synthesis and Characterization of Chitosan Nanoparticles for Lithium Metal Ion

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Abstract - Nanoparticles have enormous potential application in drug delivery system. Chitosan nanoparticles have received plenty of attention due to their non-toxicity, biocompatibility, biodegradability and controlled drug release. The objective of the present work was to evaluate the potential of chitosan-tripolyphosphate (TPP) nanoparticles as a carrier in the preparation of Lithium loaded chitosan nanoparticles. Lithium loaded chitosan-TPP nanoparticles were prepared by using Ionotropic gelation method. The cross linking between TPP and chitosan was determined by FTIR studies. The particle size and zeta potential of nanoparticles were studied by dynamic light scattering (DLS) and zeta potential analyser. The particle size for chitosan nanoparticles and Lithium loaded chitosan nanoparticles were found to be148.4 nm and 179.0 nm respectively. The zeta potential for chitosan nanoparticles and Lithium loaded chitosan nanoparticles were found to be+35.70mV and +29mV. These nanoparticles have good loading capacity of 11% for Lithium. These studies suggest that chitosan can complex TPP to form Lithium loaded nanoparticles.

Key Words: Nanoparticles, Chitosan, Ionotropic Gelation, Lithium

1. INTRODUCTION

Recently, polymeric nanoparticles have been widely investigated as a carrier for drug delivery [1]. Because of higher surface to volume ratio, nanoparticles have high drug entrapment efficiency. Nanoparticulate delivery systems offer numerous advantages such as sustained drug release, lesser frequency of dose administration, improved efficacy, reduced toxicity, and protection of the associated drug against enzymatic and chemical degradation in vivo [2].

Polymeric nanoparticles from biodegradable and biocompatible polymers are good candidates for drug carrier to deliver drugs, because they are expected to be adsorbed in an intact form in the gastrointestinal tract after oral administration [3].Chitosan (CS) the second abundant polysaccharide, is a copolymers of glucosamine and N-acetyl glucosamine linked by β-(1–4) linkages [4]. CS is nontoxic, hydrophilic, biocompatible, and because of its cationic nature, has very good mucoadhesive, antibacterial, and membrane permeability properties [5]. Chitosan is positively charged due to its amino groups and able to interact strongly with the negatively charged surface [6]. Chitosan increases the paracellular transport of polar drugs by transiently opening the tight junctions between the epithelial cells [7]. Chitosan has been shown the ability to increase membrane permeability, both in vitro [8-10] and in vivo [11], and be degraded in serum by lysozyme.

Lithium an important element for human beings is present in minerals such as petalite, lepidolite, eucryptite etc. It is responsible for stabilize serotonin transmission in nervous system. Lithium has been reported to play an important physiological role in hematopoiesis. Deficiency of lithium causes an increase in depression, reproductive problems, drug addiction, and poor lipid metabolism while its efficiency results in drowsiness, vomiting etc. In the treatment of Alzheimer's disease, lithium has been as a potential agent.

2. MATERIALS AND METHODS

2.1 MATERIALS

Chitosan (low molecular weight, deacetylation degree>85%) from Aldrich (Germany), Sodium Tripolyphosphate from Aldrich (Germany), Lithium carbonate was purchased from S.D Fine-Chem. Ltd. (Mumbai).

2.1 EXPERIMENTAL/ METHODOLOGY

Chitosan nanoparticles were prepared by using ionotropic gelation process first reported by Calvo et al [12]. Briefly, Chitosan solution was prepared by dissolving 100mg of chitosan in 100mL of acetic acid solution (1% v/v) with constant magnetic stirring for 24h. On addition of 2mL of TPP solution (1mg/mL) to 5mL of the chitosan solution with constant magnetic stirring at room temperature for 1h, nanoparticles were

formed. The resulting nanosuspension was centrifuged thrice at 18,000rpm for 30 minutes, washed with distilled water and freeze-dried.

The loading of prepared chitosan nanoparticles with Li+ has been performed according to method reported by Lifeng Qi et al. [13]. For the preparation of Lithium loaded nanoparticles, 20mg of chitosan nanoparticles were added with 50mL of lithium carbonate solution (100µg/mL) respectively. The mixture was stirred for 12h at room temperature. The resulting nanosuspension was centrifuged thrice at 18,000rpm for 30 minutes, washed with distilled water and freeze-dried.

2.3 Physicochemical Characterization

2.3.1 FTIR Studies

The FTIR spectra of chitosan, chitosan nanoparticles, Li^+ loaded chitosan nanoparticles and were taken using KBR pellets on Perkin Elmer BX FTIR spectrophotometer.

2.3.2 Particle size and Zeta potential

Size of particles and Zeta potential were determined by using Malvern Nano-ZS. The analysis was performed at a scattering angle of 90^0 at a temperature of 25^0C. The measurements were carried out using a suspension of the nanoparticles in deionized and distilled water.

2.3.3 Loading Capacity

The loading capacity of chitosan nanoparticles for Li^+ metal ions were determined by adding 50mL of metal ion solution to 20mg of freeze-dried chitosan nanoparticles with constant magnetic stirring (1200-1600rpm) for 12h at room temperature. After this, the nanoparticles were separated from the aqueous suspension medium by centrifugation at 18,000rpm for 30 minutes. The amount of free Li^+ in supernatant was evaluated by atomic absorption spectrophotometer using appropriate calibration plot.

$$L.C = \frac{\text{Total amount of metal ion - Free amount of metal ion}}{\text{Weight of nanoparticles}} \text{ X } 100$$

2.4 RESULTS AND DISCUSSION

2.4.1 FTIR Studies

IR spectral analysis has been performed to determine the interaction among metal ions, chitosan, and TPP. The IR spectra of chitosan, chitosan nanoparticles, chitosan nanoparticles loaded with Li^+ metal ions are shown in figure 1-3 respectively and their characteristic peaks are represented in Table No. 1. IR spectrum of chitosan (fig. 1) shows a peak in the range of 3300-3600cm^{-1} which corresponds to the -OH and -NH stretching. Peaks due to -CONH, -NH bending and -C-O-C- are observed at 1657.35cm^{-1}, 1535.00cm^{-1} and 1087.74cm^{-1} respectively. In the IR spectrum of chitosan nanoparticles as shown in figure 2, broadening of peak in the range, 3600-3200cm^{-1} is observed which indicates hydrogen bonding. A shift from 1657.35cm^{-1} to 1643.50cm^{-1} (CONH) and appearance of new peak at 1539.72cm^{-1} (NH bending) in the spectrum of chitosan nanoparticles attributed to the linkage between phosphate groups of TPP with ammonium group of chitosan [13]. The presence of P=O group in the nanoparticles is indicated by appearance of peak at 1151.78cm^{-1}.

In the IR spectra of chitosan nanoparticles loaded with Li^+ metal ions as shown in figure 3, sharp combined peak of -OH and -NH stretching at 3399.95cm^{-1}, 3421.86cm^{-1} and 3429.19cm^{-1} are observed respectively which reflects the interaction of –OH and $–NH_2$ group with metal ions [14]. A peak for CONH at 1657.35cm^{-1} in chitosan is found to be shifted to 1657.35cm^{-1}, 1637.33cm^{-1}, 1638.54cm^{-1} and 1638.60cm^{-1} and a common new peak at 1537cm^{-1} appears for Li^+ loaded chitosan nanoparticles. This observation indicates the linkage between phosphate group of TPP and ammonium group of chitosan.

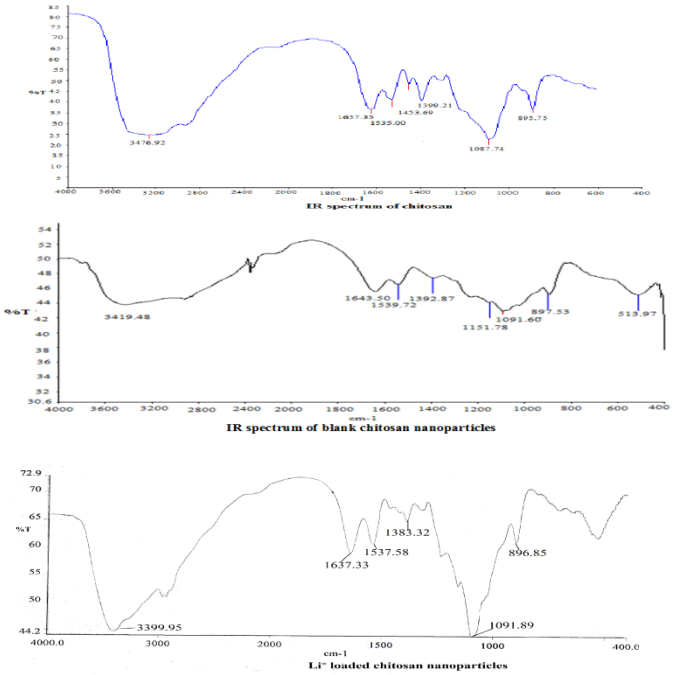

Fig.1.FTIR spectra of chitosan (A), chitosan nanoparticles (B), and Lithium loaded chitosan nanoparticles (C).

Table No. 1. Characteristic peaks in IR spectra of metal ions loaded chitosan nanoparticles

Samples	Characteristic peaks in cm^{-1}		
	-OH and -NH stretching	-CONH	-NH bending
Chitosan	3600-3300	1657.35	1535.00
Chitosan Nanoparticles	3600-3200	1643.50	1539.72
Li^+ loaded chitosan Nanoparticles	3399.95	1637.33	1537.58

2.4.2 Particle Size and Zeta Potential of Chitosan Nanoparticle Loaded with Lithium

The size of blank chitosan nanoparticle is found to be 148.4nm whereas the size of nanoparticles loaded with Li^+ is found to be 179.0nm. The increase in the size of nanoparticles after loading with metal ion can be attributed to the adsorption of metal ions on their surface [15].

The zeta potential of blank chitosan nanoparticle is found to be +35.70 mV. The positive zeta potential indicates positive charge on the nanoparticles, which is due to the amino group of chitosan. At 100µg/mL, the observed zeta potential of chitosan nanoparticles loaded with Li^+ metal ions is +29.0mV, which reflects high stability of these nanoparticles.

Loading Capacity of Lithium

The loading capacity of nanoparticles was calculated by determining the amount of free Lithium in the supernatant measured by atomic absorption spectrophotometer. The loading capacity of chitosan nanoparticles was greatly influenced by Lithium Concentration. However, Chitosan nanoparticles show maximum loading capacity of 11% for Lithium metal ion.

3. CONCLUSION

Chitosan-TPP nanoparticles were successfully prepared by ionotropic gelation method. The physicochemical properties of Chitosan-TPP nanoparticles such particle size and zeta potential gets affected on loading of Lithium metal ion. Chitosan nanoparticles show maximum loading capacity of 11% for Lithium metal ion.

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