Characterization of Curcumin Encapsulation in Chitosan-Sodium Citrate Nanoparticles

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ABSTRACT

The encapsulation of curcumin in chitosan-sodium citrate nanoparticle was studied with ionic gelation method. Identification of functional groups was determined by Fourier Transform Infrared (FTIR) spectrophotometry method. The crystallinity characteristic was tested by X-Ray Diffraction (XRD). Particle size determination was performed using Transmission Electron Microscopy (TEM), whereas the morphological characterization was tested by Scanning Electron Microscopy (SEM). The results showed that the electrostatic interaction between the amine group and a carboxyl group can cause a shift in the uptake of chitosan-sodium citrate nanoparticles at wave number 1635 cm\(^{-1}\) to 1581 cm\(^{-1}\). Qualitative data of XRD spectra show that there are reducing crystallinity of curcumin. Nanoparticles have a size of ±95nm.

Keywords: encapsulation, nanoparticles, curcumin, chitosan, sodium citrate

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1. Introduction

Curcumin is a hydrophobic polyphenol contained in turmeric (Curcuma longa). Curcumin has advantages in a variety of pharmacological activities such as anti-oxidant, anti-inflammatory, antitumoral and antimicrobial. Curcumin pharmacological activities can be used to cure diseases like cancer. However, the clinical application is limited because curcumin has low solubility in water, rapid hydrolysis at neutral and alkaline pH, experienced rapid systemic metabolism and elimination, as well as lead to poor absorption decrease the bioavailability. Various strategies have been made to overcome the limitations of the use of curcumin and allowed therapeutic applications, such as improvements in delivery systems (Mazzarino et al., 2012).

Curcumin delivery to the carrier nanoparticles has been successfully used to improve the bioavailability of curcumin and to protect from rapid metabolism and degradation. The nanoparticles coated with a mucoadhesive polysaccharide emerged as a promising strategy to extend the residence time and to increase the absorption of the drug through the mucosa. Chitosan has been widely investigated for its ability to interact with negatively charge from mucosal surface and to increase the absorption of the drug by opening narrow connectors between mucosal cells (Tiyaboonchai et al., 2003). Chitosan is a polyelectrolyte compound can be combined with a polyanion, such as sodium citrate.

Fig 1. Structure of chitosan-citrate (Pieróg et al., 2009)

Covalent crosslinking agents are generally toxic, in order to overcome this, an ionic crosslink agent is reversible. Chitosan is a polycationic polymer. These properties cause interaction with negatively charged (anionic) components. The ionic interactions occur between the negative charges of the crosslinking agent and the positive charge of chitosan. The ionic crosslink method is a simple and easy procedure. The presence of ionic crosslinking enables modified chitosan formed into a variety
of drug delivery systems, such as microparticles and nanoparticles (Berger et al., 2004).

Based on the explanation above, it would require the development of more efficient methods of synthesis, namely the ionic gelation method. This method is based on the electrostatic interaction between polycation and polyanion with high-speed stirring. Ionic gelation method requires simple preparation, which is carried out at room temperature. Researchers Le et al. (2013) have reported that the results of the synthesis of chitosan by ionic gelation method can be used as carriers of curcumin to the target cancer cells.

In this study, the researchers carried out the manufacture of chitosan-sodium citrate nanoparticles using the ionic gelation method for encapsulation of curcumin. This study focused on the synthesis and characterization of chitosan-sodium citrate nanoparticle to encapsulate curcumin and analyzed it as an effective delivery system.

2. Materials and Procedures

2.1. Materials

The studies used chitosan with the deacetylation degree of 81%, sodium citrate, aquabidest, curcumin, ethanol, and acetic acid (Merck). The instruments used for characterization included Fourier Transform Infrared Spectrophotometer (FTIR, Shimadzu Prestige-21), X-Ray Diffraction (XRD, Rigaku Miniflex-600), Scanning Electron Microscopy (SEM, JEOL JSM 6510LA), and Transmission Electron Microscopy (TEM, JEOL JEM-1400).

2.2. Experimental Procedures

2.2.1. Synthesis of curcumin loaded chitosan-sodium citrate nanoparticles

Synthesis of curcumin loaded chitosan-sodium citrate nanoparticles were prepared by ionic gelation method. The Early preparation was done with the fabrication encapsulant. A number of chitosan dissolved in 1% acetic acid and sodium citrate dissolved in aquabidest. Curcumin solution was added to the solution while stirring sodium citrate. The suspension was evaporated in a water bath temperature of 40°C for 30 minutes. Stirring was carried out for 4 hours for further centrifugated at 7000 rpm for 90 minutes. The supernatant was washed using centrifugation results aquabidest and dried with freeze dryer.

2.2.2. Characterization of curcumin loaded chitosan-sodium citrate nanoparticles

Characterization of FTIR (Fourier Transform Infrared) was performed with a Shimadzu to confirm the determination of functional groups. Samples formed with KBr pellets. IR spectra were determined in the wave number region between 450-4000 cm⁻¹. Particle size determination was made by TEM (Transmission Electron Microscopy).

The characterization results obtained from TEM sample surface in the form of photos with ranges up to nanometers (nm). Characterization of the surface morphology was confirmed by means of SEM (Scanning Electron Microscopy), while the crystallinity measured by XRD method (X-Ray Diffraction) using Cu Ka (λ=1.54060 Å), with voltage 40 kV and current of 30 mA. The samples performed at 20 of 3.02 to 80 degrees. From the measurements, it obtained diffractionogram pattern for the samples analyzed.

3. Results and Discussion

Study of the interaction between the components of functional groups in the nanoparticulate system can be studied with FTIR spectrophotometry in Figure 2. In this analysis, it shows that the carboxyl group (COO⁻) of the anionic polymers can interact electrostatically with -NH₃⁺ (amino group) of chitosan. This interaction occurs in the FTIR spectra of chitosan-sodium citrate that has been included curcumin. The characteristic spectral shift occurs from the amine group wave number 1635 cm⁻¹ to 1581 cm⁻¹ and C=O of -COO⁻ group shift of the absorption band from wave number 1604 cm⁻¹. The C-O group in curcumin of wave number 1026 cm⁻¹ is also shifted to 1072 cm⁻¹. This indicates that the C-O on curcumin interacts with the NH₂ group of chitosan, so that can be sequestered curcumin of chitosan-sodium citrate nanoparticles. The interaction between keto group from curcumin and the amine group of chitosan can cause drug loading.

![Fig. 2. FTIR Spectra (A) Chitosan-sodium citrate-curcumin, (B) Curcumin, (C) Sodium citrate, (D) Chitosan](image-url)

Identifying the particle size of curcumin loaded has been in the nanoparticles was determined by image analysis method using TEM (Transmission Electron Microscopy) shown in Fig 3. The results of image analysis using TEM explain that the particle size synthesized in the range of less than 100 nm, with an average diameter of ±95 nm. In the visible image that resembles the shape of the particle sphere ball and has a relatively uniform particle size. Particle transport in the body is affected by the particle shape.
Nanoparticle size is an important parameter because it can affect drug release, physical stability, and uptake by cells (Karavelidis et al., 2011).

Fig 3. The observation of curcumin loaded chitosan-sodium citrate nanoparticles size using TEM

Fig 4. describes the structural morphology of nanoparticles. The morphological arrangement of nanoparticles. Scanning electron micrographs of samples of the nanoparticles establish that the drug loaded nanoparticles have a discrete spherical shape.

The crystallinity state of a material is evidenced by the diffraction diffractogram band of X-ray Diffraction (XRD). The results of XRD analysis of curcumin powder without treatment and products loaded curcumin of chitosan-sodium citrate nanoparticles is shown in Fig 5. Fig 5. (A) shows the diffractogram of curcumin early without treatment, whereas image (B) shows the diffractogram of curcumin loaded with chitosan-sodium citrate. Diffractogram initial curcumin shows sharp peak intensity, whereas curcumin loaded in the polymer shows a decrease in crystallinity. This indicates that when the chitosan-sodium citrate nanoparticles, curcumin is dispersed amorphous solid in the polymer matrix after encapsulation.

Fig 5. Diffractogram of XRD, Curcumin powder (A), Curcumin loaded in chitosan-sodium citrate nanoparticles (B)

4. Conclusion

Based on the results of research and discussion, there is a shift of the absorption band FTIR spectra of nanoparticles which shows the presence of electrostatic interactions between chitosan and sodium citrate. The nanoparticle can decrease crystallinity of XRD spectrum. Nanoparticle has size ± 95 nm.

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Conflict of interest: Non declare