



## **Synthesis of Nanoparticles Powder from Seeds Extract Clove by Ionic Gelation**

**Amel D. Hussein**

**College of Dentistry-Wasit University-Iraq**

[adashar@uowasit.edu.iq](mailto:adashar@uowasit.edu.iq)

<https://doi.org/10.32792/utq/utj/vol19/1/5>

### **Abstract:**

In this work, a nanopowder of seed extract clove was prepared in a simple loaded ionic gelation method using several enhancements to prevent particle aggregation as a surfactant to chitosan solutions [Tween 80: 0.5% (v/v)]. The clove seed extract was loaded on polymeric chitosan under a vortex until complete added the quantity. Chitosan Tripolyphosphate (TPP) solution in the ratio (2:1v/v) at room temperature under magnetic stirring was added dropwise using a syringe. The result was characterized by FTIR, SEM, and TEM almost nanorod in shape and the range of size 50 nm-1 $\mu$ m. It is used in agricultural products, biomedical, cosmetics, improving the hardness of dental fillings, a natural analgesic, antibiotic, etc.

**Keywords:** Clove seed, surfactant, chitosan, ionic gelation, acetic acid.

### **Introduction:**

With the development of nanotechnology, new materials are appear which used as compared to their bulk scale, to enhance the properties of the materials such as; carbon nanotubes, metal nanoparticles, etc... These materials are used in many applications such as wastewater treatment, food, agriculture, etc. [1, 2]. "Nanotechnology is a branch of science that deals with materials within a small size between (1-100 nm) in different shapes" [3, 4].

There are various benefits of nanoparticles to modern medicine; since nanoparticles can provide essential improvements [5]. In recent times intended for the reduction of harmful chemical residues by focusing on using sustainable industrial methods [6, 7].

Clove is a rich source of gallic acid, eugenol acetate, and eugenol. So, they are aromatic flower buds of a tree in the family Syzygium aromatic, and Myrtaceae which achieve a modified number of benefits in cosmetics, food, agriculture, and pharmacological activities [8, 9].

The Chitosan process is an abundant natural cationic biopolymer consequential as of partial deacetylation from chitin, so, it is biocompatible, biodegradable, has famous antimicrobial properties, nontoxic, microparticles (CMPs), also chitosan nanoparticles (CNPs), as well as membranes, has attracted significant benefits in applications in the pharmaceutical industry, wastewater treatment, water, food industry, and drug delivery [10-14] which can be scrutinized in Fig.1. Clove nanoparticles have varied uses in the dental field. In this study, the synthesized clove nanoparticle powder with a particle size of less than 50 nm can be used as an analgic for tooth pain, etc.

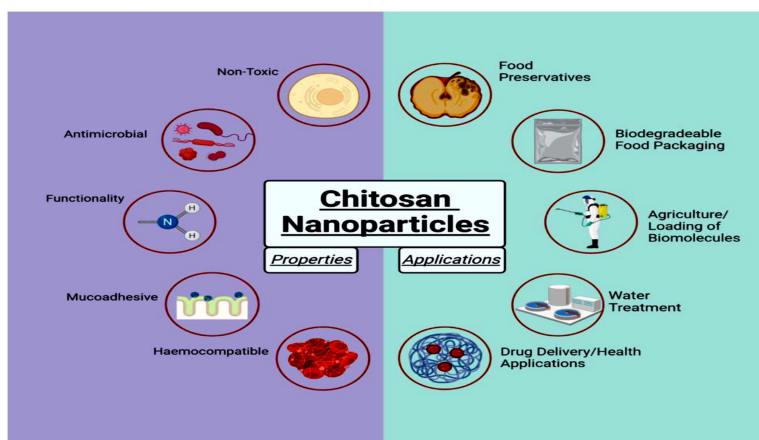


Fig. (1): Expression of the properties and applications of chitosan nanoparticles [15].

### Materials and Methods:

Clove seed extract loads were prepared using the ionic gelation method. The possible mechanism of ionic gelation was illustrated in Fig. 2 where the ionic interaction between negatively charged chitosan with positively charged polyanion sodium tripoly-phosphate (TPP).

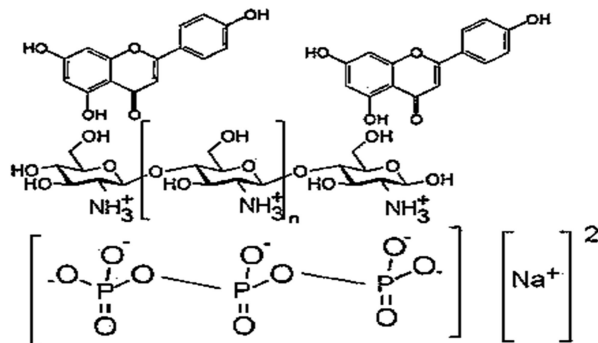


Fig. (2): Illustration the mechanism of cross linking between chitosan and TPP [16].

CNP was prepared by ionic gelation method using surfactant inducing gelation of a chitosan solution added in Sodium TPP. Chitosan was dissolved at room temperature under magnetic stirring in 2% acetic acid aqueous solutions for (20–24 hr.) until a clear solution was structured. Ionotropic gelation is achieved owed to the interaction between negatively charged TPP, in addition to positively charged amino groups. Therefore, a drug in different concentrations of chitosan was prepared. Sodium TTP solution is structured by dissolving 250 mg of TPP in 100 ml deionized water. The resulting solutions were filtered within a 0.22-micron filter. Firstly extract is stirred separately for 3 hours using 1200 rpm at room temperature and under magnetic stirring [17].

Chitosan solution was stirred using a magnetic stirrer overnight at room temperature. PH solution was modified to perform (4.7-4.8) then a syringe filter of (0.45  $\mu\text{m}$ ) chitosan solution was applied. A magnetic stirrer was placed at 4°C, then 20 ml of chitosan solution in a 50 ml round bottom flask, preheated in a water bath for 10 min at 60°C, was put on the magnetic stirrer with 800 rpm. Then, clove seeds extract was loaded on polymeric chitosan under vortex until complete added the quantity. In a magnetic stirring at 800 rpm drop wise TPP solution was added by a syringe with a ratio (2:1v/v) (chitosan: TPP) to chitosan solution at room temperature. It is obtained suspended in surfactant [Tween 80 0.5% (v/v)] or Vitamin E-TPGS, was further added to prevent particles from aggregation. The resulting chitosan particle suspension was centrifuged for 30 min at 12000 rpm. [18].

## Characterization:

### 1. Transmission Electron Microscopy (TEM):

The morphology of CNP was characterized by transmission electron microscopy TEM images of nanoparticle structures with shapes similar to nanorods at the nanoscale, caused by the combination of the particles, poly-distribution, with a few distributions of agglomeration [19]. Fig. 2 confirms images of CNP at different magnifications.

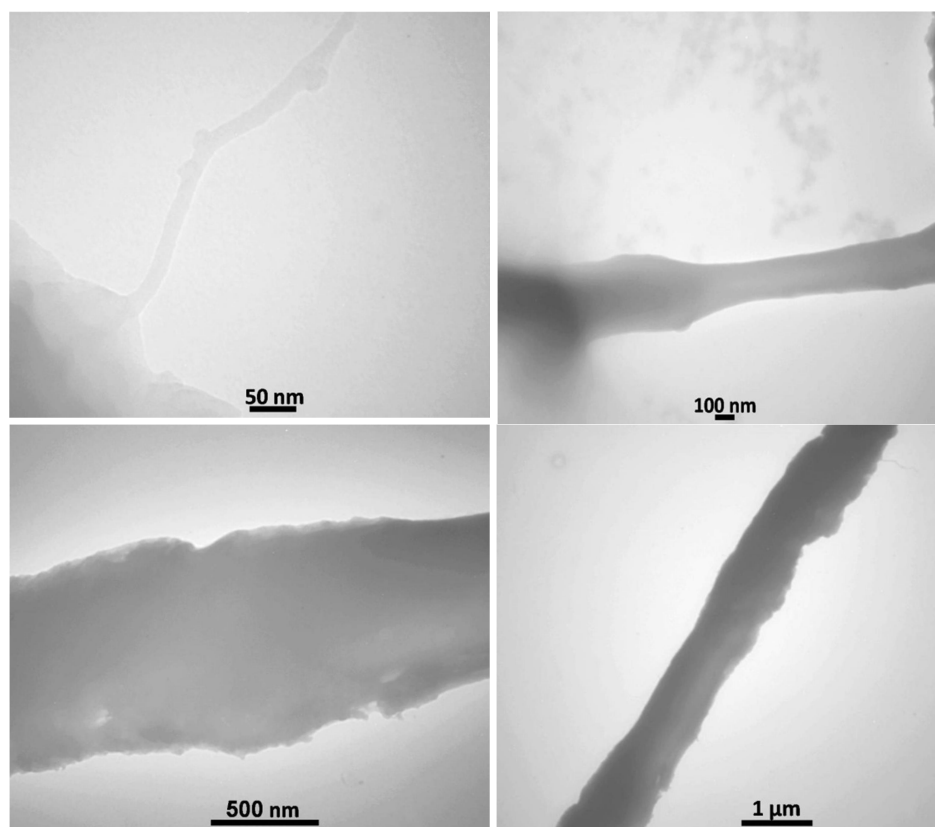


Fig. (3): TEM image of clove nanoparticles.

### 2- Scanning Electron Microscopy (SEM):

Clove NP was analyzed via SEM as seen in Fig. 4, the images taken for clove NP synthesized with ionic gelation generation displayed rod morphology, and a variety of agglomeration caroused

by an incomplete growth phase, resulting in average sizes of less than 50 nm. So, nanoparticles in the range of size <50 nm are obtained through a filtration process [20].

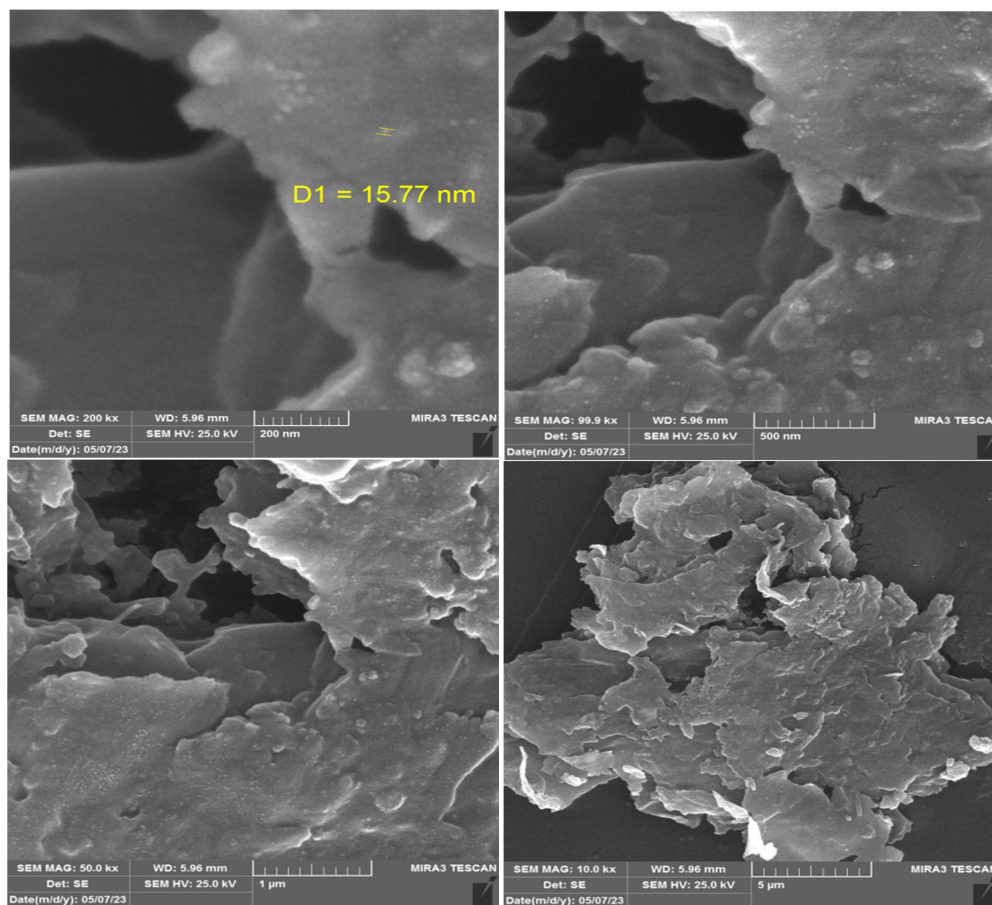


Fig. (4): SEM images of clove nanoparticles with different magnifications.

### 3- Fourier Transforms Infrared Spectroscopy (FTIR) analysis:

FT-IR spectra of clove nanoparticles are presented in Fig. 5. Spectrum of the clove show peaks at 3426, 2365, and 1649  $\text{cm}^{-1}$ . Fig. 5 illustrates, various absorption peaks with some of them due to the structure of dominant bioactive materials that existed in the clove extract that correspond to [O-CH, -OH, =CH-, -C-O] functional groups as indicated in [21].

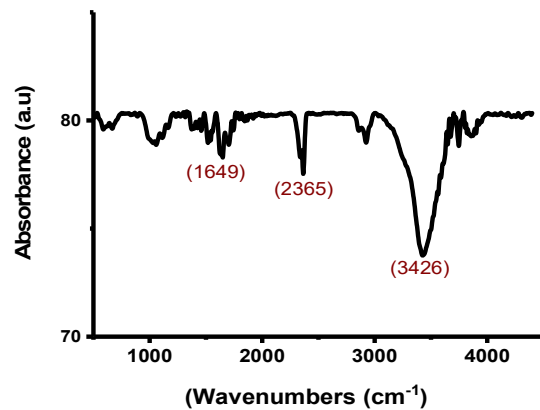


Fig. (5): FTIR spectra of clove nanoparticles prepared using ionic gelation generation process.

## Conclusions

Clove nanoparticles were synthesized using an ionic gelation generation process which is a low cost, sample method, and safety. The TEM, SEM, and FTIR were used to study the structural properties of nanopowder cloves. The analyzing apparatuses above show nanoparticle structures with different shapes, and scales with average sizes of 50 nm. The FTIR spectra of clove nanoparticles showed a maximum peak at 3426  $\text{cm}^{-1}$  that can be used in many applications.

## References:

- [1] A. Vahidi, H. Vaghari, Y. Najian, M.J. Najian, H. Jafarizadeh-Malmiri, Evaluation of three different green fabrication methods for the synthesis of crystalline ZnO nanoparticles using Pelargonium zonale leaf extract, *Green Processing and Synthesis* 8(1) (2019) 302-308.
- [2] S.O. Ogunyemi, Y. Abdallah, M. Zhang, H. Fouad, X. Hong, E. Ibrahim, M.M.I. Masum, A. Hossain, J. Mo, B. Li, Green synthesis of zinc oxide nanoparticles using different plant extracts and their antibacterial activity against *Xanthomonas oryzae* pv. *oryzae*, *Artificial cells, nanomedicine, and biotechnology* 47(1) (2019) 341-352.
- [3] I. Khan, K. Saeed, I. Khan, Nanoparticles: Properties, applications and toxicities, *Arabian journal of chemistry* 12(7) (2019) 908-931.
- [4] A.D. HUSSEIN, FABRICATION SENSORS BASED ON NANOCOMPOSITES ZnO/PVDF, *International Journal of Applied Sciences and Technology* 4(3) (2022) 124-128.



- [5] V. Vilas, D. Philip, J. Mathew, Catalytically and biologically active silver nanoparticles synthesized using essential oil, *Spectrochimica acta part a: molecular and biomolecular spectroscopy* 132 (2014) 743-750.
- [6] B.A. Ben–Arfa, I.M.M. Salvado, J.M. Ferreira, R.C. Pullar, Clove and cinnamon: Novel anti–oxidant fuels for preparing magnetic iron oxide particles by the sol–gel auto–ignition method, *Journal of Alloys and Compounds* 786 (2019) 71-76.
- [7] H. Gao, H. Yang, C. Wang, Controllable preparation and mechanism of nano-silver mediated by the microemulsion system of the clove oil, *Results in physics* 7 (2017) 3130-3136.
- [8] S.K. Shukla, A.K. Mishra, O.A. Arotiba, B.B. Mamba, Chitosan-based nanomaterials: A state-of-the-art review, *International journal of biological macromolecules* 59 (2013) 46-58.
- [9] G.E.-S. Batiha, A.M. Beshbishy, A. El-Mleeh, M.M. Abdel-Daim, H.P. Devkota, Traditional uses, bioactive chemical constituents, and pharmacological and toxicological activities of *Glycyrrhiza glabra* L.(Fabaceae), *Biomolecules* 10(3) (2020).
- [10] A.J. Al-Manhel, A.R.S. Al-Hilphy, A.K. Niamah, Extraction of chitosan, characterisation and its use for water purification, *Journal of the Saudi Society of Agricultural Sciences* 17(2) (2018) 186-190.
- [11] T. Liu, J. Li, Q. Tang, P. Qiu, D. Gou, J. Zhao, Chitosan-based materials: An overview of potential applications in food packaging, *Foods* 11(10) (2022) 1490.
- [12] U. Garg, S. Chauhan, U. Nagaich, N. Jain, Current advances in chitosan nanoparticles based drug delivery and targeting, *Advanced pharmaceutical bulletin* 9(2) (2019) 195.
- [13] A. Bhatnagar, M. Sillanpää, Applications of chitin-and chitosan-derivatives for the detoxification of water and wastewater—a short review, *Advances in colloid and Interface science* 152(1-2) (2009) 26-38.
- [14] B. Doshi, M. Sillanpää, S. Kalliola, A review of bio-based materials for oil spill treatment, *Water research* 135 (2018) 262-277.
- [15] N. Van Bavel, T. Issler, L. Pang, M. Anikovskiy, E.J. Prenner, A simple method for synthesis of chitosan nanoparticles with ionic gelation and homogenization, *Molecules* 28(11) (2023) 4328.
- [16] A.M. Jasim, E.D. Alhtheal, S.S. Raheem, K.J. Rawaa, A. HAMA, Characterization and Synthesis of Selenium-TPGS Nanoparticles for Target Delivery Clove to Minimize Cytogenic and Liver Damage Induced in Adult Male Rats, *Nano Biomed* 13(2) (2021) 127-136.



- [17] A.A. Tayel, A.F. Elzahy, S.H. Moussa, M.S. Al-Saggaf, A.M. Diab, Biopreservation of shrimps using composed edible coatings from chitosan nanoparticles and cloves extract, *Journal of Food Quality* 2020(1) (2020) 8878452.
- [18] D.H.A. Sudarni, U.O. Aigbe, K.E. Ukhurebor, R.B. Onyancha, H.S. Kusuma, H. Darmokoesoemo, O.A. Osibote, V.A. Balogun, B.A. Widyaningrum, Malachite green removal by activated potassium hydroxide clove leaf agrowaste biosorbent: characterization, kinetic, isotherm, and thermodynamic studies, *Adsorption Science & Technology* 2021 (2021) 1145312.
- [19] M.V.d.O.B. Maciel, A. da Rosa Almeida, M.H. Machado, A.P.Z. de Melo, C.G. da Rosa, D.Z. de Freitas, C.M. Noronha, G.L. Teixeira, R.D. de Armas, P.L.M. Barreto, *Syzygium aromaticum* L.(clove) essential oil as a reducing agent for the green synthesis of silver nanoparticles, *Open Journal of Applied Sciences* 9(2) (2019) 45-54.
- [20] U.P. Agarwal, S.A. Ralph, R.S. Reiner, C.G. Hunt, C. Baez, R. Ibach, K.C. Hirth, Production of high lignin-containing and lignin-free cellulose nanocrystals from wood, *Cellulose* 25 (2018) 5791-5805.
- [21] A. Mohamed, R. Atta, A.A. Kotp, F.I. Abo El-Ela, H. Abd El-Raheem, A. Farghali, D.H.M. Alkhalifah, W.N. Hozzein, R. Mahmoud, Green synthesis and characterization of iron oxide nanoparticles for the removal of heavy metals ( $Cd^{2+}$  and  $Ni^{2+}$ ) from aqueous solutions with Antimicrobial Investigation, *Scientific Reports* 13(1) (2023) 7227.