

**SINTESIS DAN KARAKTERISASI NANOPARTIKEL
KITOSAN-TRIPOLIFOSFAT**

SKRIPSI

Diajukan untuk memenuhi sebagian syarat untuk memperoleh gelar Sarjana Sains
Program Studi Kimia



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**Sebuah skripsi yang diajukan untuk memenuhi salah satu syarat
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ABSTRAK

Penelitian ini bertujuan untuk mengetahui kondisi optimum sintesis dan karakteristik-nanopartikel kitosan-tripolifosfat. Tahapan penelitian yang dilakukan meliputi tahap optimasi, sintesis nanopartikel kitosan-tripolifosfat. melalui metode gelasi ionik berikut karakterisasinya melalui uji FTIR (*Fourier Transform Infrared Spectroscopy*), *X-Ray Diffraction (XRD)*, *Scanning Electron Microscopy-Energy Dispersive X-Ray (SEM-EDX)*, dan spektrofotometer UV-Vis. Hasil penelitian menunjukkan bahwa kondisi optimum sintesis nanopartikel kitosan-tripolifosfat diperoleh pada konsentrasi optimum tripolifosfat 1% dan kitosan 1,5% dengan kecepatan pengadukan 700 rpm (randeman 43,33%). Nanopartikel kitosan-tripolifosfat (NPCS) mengalami penurunan ukuran partikel seiring dengan bertambahnya konsentrasi kitosan, dan memiliki ukuran rata-rata sekitar $\pm 24,695-178,88$ nm dengan keberadaan C, O, Na, dan P sebagai unsur penyusun berdasarkan SEM-EDX. Selain itu berdasarkan analisis spektra UV-Vis ukuran rata-rata NPCS yaitu 4,85 – 16,28 nm. Spektra FTIR mengkonfirmasi interaksi antara tripolifosfat berlangsung secara dominan antara gugus P-O dengan gugus amino kitosan. Penambahan konsentrasi kitosan menurunkan intensitas difraksi sinar-X nanopartikel kitosan-tripolifosfat.

Kata kunci : Kitosan, tripolifosfat, Nanopartikel kitosan-tripolifosfat, sintesis, karakterisasi

ABSTRACT

This study aims to determine the optimum conditions of synthesis and characteristics of chitosan-tripolyphosphate nanoparticles. Stages of research carried out include the optimization stage, the synthesis of chitosan-tripolyphosphate nanoparticles through the ionic gelation method and its characteristics through the FTIR (Fourier Transform Infrared Spectroscopy) test, X-Ray Diffraction (XRD), Scanning Electron Microscopy-Energy Dispersive X-Ray (SEM-EDX), and UV-Vis spectrophotometer. The results showed that the optimum conditions of chitosan-tripolyphosphate nanoparticle synthesis were obtained at optimum concentrations of 1% tripolyphosphate and 1.5% chitosan with a stirring speed of 700 rpm (randeman 43.33%). Chitosan-tripolyphosphate (NPCS) nanoparticles experienced a decrease in particle size with increasing chitosan concentration, and had an average size of about $\pm 24,695$ -178.88 nm in the presence of C, O, Na, and P as constituent elements based on SEM-EDX. Also based on UV-Vis spectra analysis the average size of NPCS is 4.85 - 16.28 nm. The FTIR spectra confirm the interaction between the tripolyphosphate predominantly with the chitosan amine group. The addition of chitosan concentration decreases x-ray diffraction intensity in the chitosan-tripolyphosphate nanoparticle.

Keywords: *Chitosan, tripolyphosphate, chitosan-tripolyphosphate nanoparticles, synthesis, characterization*

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DAFTAR PUSTAKA

- Ali, S., Khan, I., Khan, S.A., Sohail, M., Ahmed, R., Rehman, A., Ur Ansari, M.S., Morsy, M.A. (2017). Electrocatalytic performance of Ni@Pt core-shell nanoparticles supported on carbon nanotubes for methanol oxidation reaction. *Journal Electroanal Chem.* 795, 17–25. <http://dx.doi.org/10.1016/j.jelechem.2017.04.040>.
- Aranaz, I. (2017). Chitosan Spray-Dried Microparticles for Controlled. *Molecules*, 22, 1–13. <https://doi.org/10.3390/molecules22111980>
- Anitha, R., & Shantha, L. (2010). Bioadhesive chitosan nanoparticles: Preparation and characterization. *Carbohydrate Polymers*, 81, 243-251. <https://doi.org/10.1016/j.carbpol.2010.02.026>
- Antony, R., Arun, T., & Manickam, S. T. D. (2019). Review of Application of chitosan-based Schiff Based. *International Journal of Biological Macromolecules*, 3, 234-235. <https://doi.org/10.1016/j.ijbiomac.2019.02.047>
- Arimoto, H. (1964). *Journal of Polymer Science Part A. 2*, 2283-2295
- Balan, V., & Andreea, I. (2012). Biotinylated chitosan-based SPIONs with potential in blood-contacting applications. *Journal of Nanoparticle Research*, 14(2), 730-743. <https://doi.org/10.1007/s11051-012-0730-y>
- Banach, M., Kowalski, Z., Wzorek, Z., & Gorazda, K. (2009). A chemical method of the production of " heavy " sodium tripolyphosphate with the high content of Form I or Form II. *Journal of Chemical Technology*, 11, 13-20. <https://doi.org/10.2478/v10026-009-0018-x>
- Bibi, S., Jamil, A., Yasin, T., Rafiq, M. A., Nawaz, M., & Price, G. J. (2018). Ultrasound promoted synthesis and properties of chitosan nanocomposites containing carbon nanotubes and silver nanoparticles. *European Polymer Journal*, 105, 297–303. <https://doi.org/10.1016/j.eurpolymj.2018.06.004>
- Bodnar, M., Hartmann, J. F., & Borbely, J. (2005). Preparation and Characterization of Chitosan-Based Nanoparticles. *Biomacromolecules*, 2, 2521–2527. <https://doi.org/10.1021/bm0502258>
- Bunaciu, A. A., Udriștioiu, E., Aboul-enein, H. Y., Bunaciu, A. A., Udriștioiu, E. (2015). *Critical Reviews in Analytical Chemistry X-Ray Diffraction : Instrumentation and Applications X-Ray Diffraction : Instrumentation and Applications*. 8347. <https://doi.org/10.1080/10408347.2014.949616>
- Boonsongrit, Y., Mitrevej, A., & Mueller, B. W. (2006). Chitosan drug binding by ionic interaction. *European Journal of Pharmaceutics and Biopharmaceutics*, 62(3), 267–274. <https://doi.org/10.1016/j.ejpb.2005.09.002>

- Chang, K. L. B., Tsai, G., Lee, J., & Fu, W. (1997). Heterogeneous N-deacetylation of chitin in alkaline solution. *Carbohydrate Research*, 303(3), 327–332.
- Cheng, G., & A. R. H. W. (2007). Synthesis and characterization of cobalt / gold bimetallic nanoparticles. *Journal of Magnetism and Magnetic Materials*, 311(1), 31–35. <https://doi.org/10.1016/j.jmmm.2006.11.164>
- Csaba, N., Köping-höggård, M., & Alonso, M. J. (2009). Ionically crosslinked chitosan / tripolyphosphate nanoparticles for oligonucleotide and plasmid DNA delivery. *International Journal of Pharmaceutics*, 382(1-2), 205–214. <https://doi.org/10.1016/j.ijpharm.2009.07.028>
- Divya, K., Vijayan, S., & Nair, J. (2019). Optimization of chitosan nanoparticle synthesis and its potential application as germination elicitor of *Oryza sativa* L. *International Journal of Biological Macromolecules*. 124, 1053-1059. <https://doi.org/10.1016/j.ijbiomac.2018.11.185>
- Dreaden, E.C., Alkilany, A.M., Huang, X., Murphy, C.J., El-Sayed, M.A. (2012). The golden age: gold nanoparticles for biomedicine. *Chem Soc Rev*. 41, 2740–2779. <http://dx.doi.org/10.1039/C1CS15237H>.
- Faculty, P. (1997). Chitosan: properties, preparations and application to microparticulate systems. *Journal Micriencapsul*, 14(6), 689–711.
- Fahim, I., Kheiridine, A & Belaouad, S. (2013). Sodium tripolyphosphate (STPP) as a novel corrosion inhibitor for mild steel in 1 M HCl. *Journal Of Optoelectronics and Advances Materials*. 15(5), 451-456.
- Gan, Q., & Wang, T. (2007). Chitosan nanoparticle as protein delivery carrier - Systematic examination of fabrication conditions for efficient loading and release. *Colloids and Surfaces B: Biointerfaces*, 59(1), 24–34. <https://doi.org/10.1016/j.colsurfb.2007.04.009>
- Gujrati, M., Malamas, A., Shin, T., Jin, E., Sun, Y., Lu, Z.-R. (2014). Multifunctional cationic lipid-based nanoparticles facilitate endosomal escape and reduction-triggered cytosolic siRNA release. *Mol Pharm*. 11, 2734–2744. <http://dx.doi.org/10.1021/mp400787s>
- Haiss, W., Thanh, N.T.K., Aveyard, J., Fernig, D.G., (2007). Determination of Size and Concentration of Gold Nanoparticles from UV–Vis Spectra. *Anal Chem*. 79, 4215–4221. <https://doi.org/10.1021/ac0702084>
- Hasan, S. (2015). A Review on Nanoparticles : Their Synthesis and Types Biosynthesis : Mechanism. *Journal of Recent Sciences*, 4, 9–11

- Hisatomi, T., Kubota, J., Domen, K. (2014). Recent advances in semiconductors for photocatalytic and photoelectrochemical water splitting. *Chem Soc Rev.* 43, 7520–7535. [http://dx.doi.org/ 10.1039/C3CS60378D](http://dx.doi.org/10.1039/C3CS60378D).
- Islam, S., Bhuiyan, M. A. R., & Islam, M. N. (2017). Chitin and Chitosan: Structure, Properties and Applications in Biomedical Engineering. *Journal of Polymers and the Environment*, 25(3), 854–866. <https://doi.org/10.1007/s10924-016-0865-5>
- Kalaivani, R., Maruthupandy, M., Muneeswaran, T., Hameedha Beevi, A., Anand, M., Ramakritinan, C. M., & Kumaraguru, A. K. (2018). Synthesis of chitosan mediated silver nanoparticles (Ag NPs) for potential antimicrobial applications. *Frontiers in Laboratory Medicine*, 2(1), 30–35. <https://doi.org/10.1016/j.flm.2018.04.002>
- Khan, I., Saeed, K., & Khan, I. (2017). Nanoparticles: Properties, applications and toxicities. *Arabian Journal Of Chemistry*. <https://doi.org/10.1016/j.arabjc.2017.05.011>
- Koilparambil, D., Rebello, S., Disease, C., & Shanavas, J. (2014). A Simple and Effective Method for Extraction of High Purity Chitosan from Shrimp Shell Waste. 141-145
- Kolahalam, L. A., Viswanath, I. V. K., Diwakar, B. S., Govindh, B., Reddy, V., & Murthy, Y. L. N. (2019). Materials Today : Proceedings Review on nanomaterials : Synthesis and applications. *Materials Today: Proceedings*, 1, 1-6. <https://doi.org/10.1016/j.matpr.2019.07.371>
- Lin, Y., Sonaje, K., Lin, K. M., Juang, J., Mi, F., Yang, H., & Sung, H. (2008). Multi-ion-crosslinked nanoparticles with pH-responsive characteristics for oral delivery of protein drugs. *Journal of Controlled Release*, 132(2), 141–149. <https://doi.org/10.1016/j.jconrel.2008.08.020>
- Lynch, I., Cedervall, T., Lundqvist, M., Cabaleiro-lago, C., Linse, S., & Dawson, K. A. (2007). The nanoparticle – protein complex as a biological entity ; a complex fluids and surface science challenge for the 21st century. *Advances in Colloid and Interface Science*, 135, 167–174. <https://doi.org/10.1016/j.cis.2007.04.021>
- Mabena, L.F., Sinha Ray, S., Mhlanga, S.D., Coville, N.J. (2011). Nitrogen-doped carbon nanotubes as a metal catalyst support. *Appl Nanosci.* 1, 67–77. <http://dx.doi.org/10.1007/s13204-011-0013-4>.
- Mansha, M., Khan, I., Ullah, N., Qurashi, A. (2017). Synthesis, characterization and visible-light-driven photoelectrochemical hydrogen evolution reaction of

carbazole-containing conjugated polymers. *Journal Hydrogen Energy*.
<http://dx.doi.org/10.1016/j.ijhydene.2017.02.053>.

- Marie, D. (1993). A study of the UV-visible chlorine absorption spectrum of molecular. *Journal of Photochemistry and Photobiology A: Chemistry*, 70(3), 205–214 [https://doi.org/10.1016/1010-6030\(93\)85045-A](https://doi.org/10.1016/1010-6030(93)85045-A)
- Mardliyanti, E., El, S., & Ria, D. (2012). Sintesis Nanopartikel Kitosan-Trypolly Phosphate Dengan Metode Gelasi Ionik : Pengaruh Konsentrasi dan Rasio Volume Terhadap Karakteristik Partikel. *Prosiding Pertemuan Ilmiah Ilmu Pengetahuan dan Teknologi Bahan*. 90-91
- Mohanraj, V. J., & Chen, Y. (2006). Nanoparticles – A Review. *Journal of Pharmaceutical Research*, 5, 561–573.
<http://doi.org/10.4314/tjpr.v5i1.14634>
- Nikoli, G. S. (2011). *Fourier Transforms - New Analytical Approaches and FTIR Strategies*. <https://doi.org/10.5772/2040>
- Ngoy, J.M., Wagner, N., Riboldi, L., Bolland, O. (2014). A CO₂ capture technology using multi-walled carbon nanotubes with polyaspartamide surfactant. *Energy Procedia*. 63, 2230–2248. <http://dx.doi.org/10.1016/j.egypro.2014.11.242>.
- Omidi, S., & Kakanejadifard, A. (2019). Modification of chitosan and nanoparticle by long chain pyridinium compounds: Synthesis, characterization, antibacterial, and antioxidant activities. *Carbohydrate Polymers*, 208, 477–485. <https://doi.org/10.1016/j.carbpol.2018.12.097>
- Paul, K., Simon, W & James, M. (2018). Determination of Gold Nanoparticles Sizes via Surface Plasmon Resonance. *Journal of Applied Chemistry*. 11(7), 25-29. <https://doi.org/10.9790/5736-1107012529>
- Raval, A. J., & Patel, M. M. (2011). Preparation and Characterization of Nanoparticles for Solubility and Dissolution Rate Enhancement of Meloxicam. *Journal of Pharmaceuticals*, 1(2), 42–49.
- Rawat, M.K., Jain, A., Singh, S., Mehnert, W., Thunemann, A.F., Souto, E.B., Mehta, A., Vyas, S.P. (2011). Studies on binary lipid matrix based solid lipid nanoparticles of repaglinide: in vitro and in vivo evaluation. *Journal Pharm Sci*. 100, 2366–2378. <http://dx.doi.org/10.1002/jps.22435>.
- Sahu, R. K., Hiremath, S. S., Sakka, Y., & Ghorbani, H. R. (2017). A review on the classification , characterisation , synthesis of nanoparticles and their application A review on the classification , characterisation , synthesis of nanoparticles and their application. *Materials Science and Engineering*, 263,

2-5. <https://doi.org/10.1088/1757-899X/263/3/032019>

- Sanguansri, P., & Augustin, M. A. (2006). *Nanoscale materials development e a food industry perspective*. 17. <https://doi.org/10.1016/j.tifs.2006.04.010>.
- Saeed, K., Khan, I., 2016. Preparation and characterization of singlewalled carbon nanotube/nylon 6,6 nanocomposites. *Instrum Sci. Technol.* 44, 435–444. <http://dx.doi.org/10.1080/10739149.2015.1127256>.
- Singh, I. H. N. B. (2016). Green synthesis of nanoparticles and its potential application. *Biotechnology Letters*, 38(4), 545–560. <https://doi.org/10.1007/s10529-015-2026-7>
- Sionkowska, A., Wisniewski, M., Skopinska, J., Kennedy, C. J., & Wess, T. J. (2004). *The photochemical stability of collagen – chitosan blends*. 162, 545–554. [https://doi.org/10.1016/S1010-6030\(03\)00397-6](https://doi.org/10.1016/S1010-6030(03)00397-6)
- Sivakami, M. S., Gomathi, T., Venkatesan, J., Jeong, H., Kim, S., & Sudha, P. N. (2013). International Journal of Biological Macromolecules Preparation and characterization of nano chitosan for treatment wastewaters. *International Journal of Biological Macromolecules*, 57, 204–212. <https://doi.org/10.1016/j.ijbiomac.2013.03.005>
- Solomon, S. D., Bahadory, M., Jeyarajasingam, A. V., Rutkowsky, S. A., & Boritz, C. (2007). Synthesis and Study of Silver Nanoparticles. 84(2), 322–325. <https://doi.org/10.1021/ed084p322>
- Sullivan, D. J., Cruz-Romero, M., Collins, T., Cummins, E., Kerry, J. P., & Morris, M. A. (2018). Synthesis of monodisperse chitosan nanoparticles. *Food Hydrocolloids*, 83, 355–364. <https://doi.org/10.1016/j.foodhyd.2018.05.010>
- Talu'mu, M. D. (2011). Sintesis Kitosan Nanopartikel dengan Metode Sonokimia , Gelasi. *J.Prog.Kim.Si*, 1(2), 130–137.
- Tiyaboonchai, W. (2018). *Chitosan nanoparticles : A promising system for drug delivery Chitosan Nanoparticles : A Promising System for Drug Delivery*. (January 2003).
- Thomas, S., Harshita, B.S.P., Mishra, P., Talegaonkar, S. (2015). Ceramic nanoparticles: fabrication methods and applications in drug delivery. *Curr. Pharm. Des.* 21, 6165–6188. <http://dx.doi.org/10.2174/1381612821666151027153246>.
- Venkatesan, B., Tumala, A., & Subramanian, V. (2016). Synthesis and characterization of chitosan nanoparticles for in vivo delivery of siRNA-Npr3 : Targeting NPR-C expression in the heart. *Data in Brief*, 8, 441–447. <https://doi.org/10.1016/j.dib.2016.05.074>
- Yoshida, A., Kaburagi, Y., & Hishiyama, Y. (2016). Scanning Electron Microscopy. In *Materials Science and Engineering of Carbon*. <https://doi.org/10.1016/B978-0-12-805256-3.00005-2>

Zahedi, S., Safaei Ghomi, J., & Shahbazi-Alavi, H. (2018). Preparation of chitosan nanoparticles from shrimp shells and investigation of its catalytic effect in diastereoselective synthesis of dihydropyrroles. *Ultrasonics Sonochemistry*, 260–264. <https://doi.org/10.1016/j.ultsonch.2017.07.023>