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**Towards Sustainable Tropical Marine: A Holistic Approach for
Welfare Development of Indonesia**

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Kata Pengantar

Puji syukur kehadiran Allah SWT atas limpahan rahmat dan karunianya sehingga Buku Proceeding 4th ICTROPS tahun 2020 telah dapat diselesaikan. Buku ini sebagai produk keluaran dari kegiatan 4th ICTROPS yang telah dilaksanakan pada 28 Oktober 2020.

Terimakasih disampaikan kepada Prof Dr. Masjaya M.Si. selaku rektor universitas Mulawarman dan Dr. Sc. Mutaid Yusuf selaku Kepala PIU-IsDB Universitas Mulawarman atas dukungan dan arahan yang telah diberikan dalam penerbitan buku ini.

Kami menyadari masih terdapat kekurangan dalam buku ini untuk itu kritik dan saran terhadap penyempurnaan buku ini sangat diharapkan. Semoga buku ini dapat memberi manfaat bagi seluruh civitas akademika di Indonesia pada umumnya dan Universitas Mulwarman pada khususnya.

Samarinda, Oktober 2020
Ketua Pelaksana

Rusfina Widayati, S.T., M.Sc.

Sambutan Rektor



Assalamu 'alaikum wr wb. (May peace be upon you)

First of all, let us thank Allah, the Almighty, for blessing and guiding us into the right path, and for granting all means and opportunities together here to attend this very meaningful occasion.

On behalf of the Mulawarman University, we are greatly honored and pleased to welcome you all to the Second International Conference on Tropical Studies and Its Application (ICTROPS) 2020 on “**Measuring the Readiness of Maritime Aspect to Support Tropical City of The New Capital of Indonesia**”.

The framework of Mulawarman University as a “Center of Excellence for Tropical Studies” as a part of the 4 in 1 initiative. This initiative is projected to excel in Teaching and Learning, Research and Innovation, and Staff Development as soft programs and Campus Infrastructure as hard program. With the assistance of this IsDB program, the 4 in 1 initiative has two umbrella programs of research consortium and curricula development. The outcomes of this initiative are competent graduates, publication, and quality of teaching-learning, excellent services, student quality, academic atmosphere, IPR, prototypes, research collaboration, and employability. In short, the 4 in 1 consortium will have a positive impact on developing nation competitiveness by providing competent graduates, collaboration in research, academic reputation, and excellent services and management.

We would also like to extend our gratitude especially to all speakers, participants and committees.

Wassalamu'alaikum wr. wb.

Samarinda, 2020
Rector,

Prof. Dr. H. Masjaya, M.Si

Sambutan Direktur Eksekutif PIU-IsDB



Assalamu 'alaikum wr wb. (May peace be upon you)

The important role of this IsDB project is as an enabler for Mulawarman University to achieve as a Service Excellence on Center for Tropical Studies (SE-CTS) by developing five key sectors namely Campus Infrastructure (CI), Teaching and Learning (T&L), Research, on Campus Service (CS), and Public Services (PS). The project will indirectly benefit GDP of the nation as a result of higher quality of education. It is expected that the project will increase the quality of graduates, both in academic skills as the core competence and soft skills as an essential added value. It is projected that the project becomes an important trajectory for qualified human resources that are heavily in need to face the embracing ASEAN Economic Community and possibly Trans Pacific Partnerships. Samarinda as the host city for Mulawarman University and East Kalimantan province will also economically benefited through the coming of more young researchers.

The International Conference on Tropical Studies and Its Application annual conference on October 28th 2020, is organized by Mulawarman University in collaboration with Islamic Development Bank (IsDB) and Ministry of Research, Technology, and Higher Education of The Republic of Indonesia.

On behalf of Project Implementation unit (PIU) Islamic Development Bank (IsDB) of Mulawarman University, We would also like to extend our gratitude especially to all speakers, participants and committees.

Wassalamu'alaikum wr. wb.

Samarinda, 2020
Executive Director of PIU-IsDB,

Dr. Sc. Mustaid Yusuf, M.Si

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Synthesis and Characterization of Chitosan-Tripolyphosphate from Clam Shells (*Cerithidea obtusa*) and Its Application to Sorption of Cadmium Ion

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Abstract

Synthesis and characterization of chitosan-tripolyphosphate (Chi-TPP) from Clam Shells (*Cerithidea obtusa*) and its application to sorption of cadmium ion has been researched. The Chi-TPP was synthesized using gelation methods by addition tripolyphosphate 0,1%. The chitosan was prepared by deacetylation from chitin and this was isolated from Clam Shells (*Cerithidea obtusa*). The Chi-TPP was characterized using Fourier Transform Infra-Red (FTIR), X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM). FTIR characterization was indicated the appearance peak of a N-H vibration at 1635 cm^{-1} , functional group of NH_3^+ deformation at 1534 cm^{-1} , stretching vibration of a C-O- at 1072 cm^{-1} and vibration of P-O at $1026,91\text{ cm}^{-1}$ was indicated the presence of TPP. XRD pattern of Chi-TPP showed broad diffraction peaks at 2θ values of 19.85° , which are thypical fingerprints of Chi-TPP. The lower intensity of Chi-TPP was revealed that its was amorphous. SEM image of Chi-TPP showed cross-linker TPP on chi and stabilizes them. The sorption application of cadmium ions on Chi-TPP was optimum at pH 4 ie 29.12 mg/g.

Keywords: chitosan-tripolyphosphate, Clam Shells, cadmium ion

Introduction

Clam shells (*Cerithidea obtusa*) is clam-species from Potamididae family and existing is very abundant in beach of Pondong Village, Paser Regency, East Kalimantan. The shells as a waste and unutilized. The utilization of shells waste is processed into chitin and chitosan product. Chitin is poly (β -(1 \rightarrow 4)-N-acetyl-D-glucosamine from the shell of clam, crab and shrimp. Chitin is collected by deproteinization and demineralization of crustaceans' wastes. The deacetylated form of chitin is chitosan. The result of dyacetylation produces many functional group of amine (-NH₂) dan hydroxil (-OH) (Dutta, *et al.*, 2004). Compared with chitin, chitosan have higher solubility, lower crystallinity, degree of deacetylation, particle size, formation of particle and agregation (Tiyaboonthai, 2003).

Application of chitosan exhibits the promising to removing of organic ang inorganic polutant like dyes, phenol and heavy metal (Bhatnagar *et al.*, 2009], Pb dan Pt (Kondo, *et al.*, 2015), Fe(II) and Mn(II) (Ali *et al.*, 2018). The removing capacity is due to the presence of functional group that can form to coordination bonds (Kondo, *et al.*, 2015). The properties of chitosan exhibits biodegradable, non-toxic, biocompatible and can be resized in nano-size. Modification of chitosan into chitosan nanoparticle with a crosslinker can expands its usefulness as drug delivery (Kavaz, 2010). organ transplants and restoring organ function (Kumar and Koh 2012) and efficient sorption of heavy metal (Holban *et al.*, 2016).

Synthesis of chitosan nanoparticle have five methods are presently available are ionic gelation method, microemulsion, emulsification solvent diffusion, polyelectrolyte complex and reverse micellarmethod (Tiyaboonthai, 2003). The most widely used method is ionic gelation method. This method utilizes the electrostatic interaction between the amine group of chitosan and a negatively charged group of polyanion such as tripolyphosphate (TPP) (Bhumkar and Pokharkar, 2006). The TPP is a type of harmless anionic crosslinker and can interact by inter- and intramolecular with chitosan to provide better spherical morphology, increase the flexibility of the polymer increase its stability (Koiparambil and Shanavas, 2017).

In this work, isolation of chitin from Clam shells (*Cerithidea obtusa*,) and transformed into chi. , chitosan-TPP was prepared using gelation method and characterized by functional group using FT-IR, crystal structure using XRD, and morphological surface using SEM. The effect of pH to application sorption on Chi-TPP.

Materials and Methods

Materials

Clam Shells (*Cerithidea obtusa*) was collected from beach Pondong Paser, Analytical grade NaOH, HCl, tripolyphosphate (TPP), CH₃COOH 25% and Cadmium.

Preparation of chitin and chitosan

Chitin from Clam Shells (*Cerithidea obtusa*) was prepared by a demineralization and deproteination. The product was transformed into chitosan by deacetylation process. The resulting of chitosan was separated, washed with distilled water to neutral pH.

Synthesis of Chi-TPP

Five grams of chi were dissolved into 25 mL CH₃COOH 1% and then 5 mL crosslinker of tripolyphosphate (TPP) was added into chi solution. The mixture was shaken by magnetic stirrer for 1 hour to homogeneous. After the completion of the reaction, the product of Chi-TPP was washed by deionized water to neutral.

Characterization.

The functional group of Chi-TPP was analyzed by Shimadzu 8201 PC Fourier transform spectrometer (FTIR). The crystal structure were characterized by Shimadzu X-ray diffraction (XRD) using CuK α radiation ($\lambda = 1.5406 \text{ \AA}$) operated at 40 kV and 30 mA). Morphological surface was examined by JEOL SSM-6510 LA Scanning electron microscopy (SEM)

The sorption of cadmium ion on Chi-TPP.

Sorption application of optimum pH was carried out by a batch adsorption method. A series of 50 mg.L⁻¹ cadmium ion were adjusted to pH 2; 3; 4; 5; 6; 7; 8; 9; 10; 11 by adding HCl or NaOH solutions. Interaction of cadmium solution on 20 mg of Chi-TPP by shaking for 120 minutes. After adsorbent was separated, concentration of cadmium was analyzed using AAS.

Results and Discussion

Calm shells (*Cerithidea obtusa*) were obtained from beach of Pondong Village, Paser, East Kalimantan. The size of shell is 5 cm, blackish brown color and existing is very abundant and unutilized. Isolation of chitin from Calm shells (*Cerithidea obtusa*) by a demineralization and deproteination and then was transformed into Chi by deacetylation. The Chi-TPP was synthesized by gelation method that is the complexation of Chi with TPP as a crosslinker. The form physical of Chi and Chi-TPP were the same, but coloring of Chi-TPP is more white and easily to dissolve in acetate acid solution. The form physical of Chi and Chi-TPP are shown in Fig.1.



Figure 1. material of a) Chi and b) Chi-TPP

Material of Chi-TPP was successfully complexed by the gelation method which was indicated change in the characteristics of the FTIR, XRD and SEM analysis. The FT-IR spectra of chitin, Chi, Chi-TPP are shown in Fig.2.

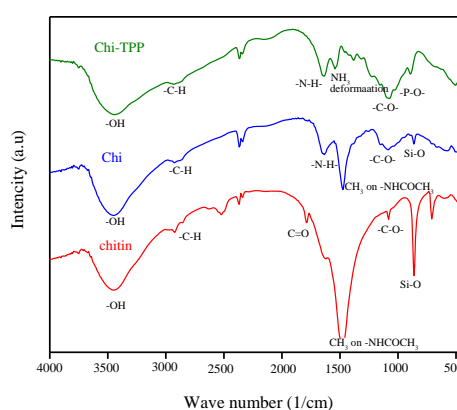


Figure 2.. FTIR Spectra of chitin, Chi and Chi-TPP

The functional group of chitin has a wavenumber at 3425 cm⁻¹ due to the -OH groups. The -CH₂ group was showing at 2924 cm⁻¹ as attributed to the symmetrical stretching, the band at 1473 cm⁻¹ was -CH₃ group on amide (-NHCOCH₃), wave number at 1798 cm⁻¹ due to the C=O group on amide (-NHCO) and then at 1080 cm⁻¹ the stretching vibration of C-O-C in glucose circle. The impurities spectra of silica mineral on chitin at wave numbers 864, 11 cm⁻¹ and 709,80 cm⁻¹. (Silverstein *et al.*, 1981).

The FTIR spectra of Chi has similar to a chitin, this has a wavenumber at 3448 cm⁻¹ due to the -OH groups, -CH₂ group at 2931 cm⁻¹, stretching vibration of C-O-C at 1087 cm⁻¹, CH₃ group on amide (-NHCOCH₃) at 1473 cm⁻¹, wave number at 1798 cm⁻¹ due to the C=O group on amide (-NHCO). (Silverstein *et al.*, 1981) and then presence of silica mineral at wave number 864 cm⁻¹. The difference FTIR spectra of chitin and Chi was loss of C=O group of amida (-NHCO-) ((Silverstein *et al.*, 1981).

Characteristic FTIR spectra of Chi-TPP has peak at 1534 cm⁻¹ was assigned to deformation of -NH, the band at 1651 cm⁻¹ was bending vibration of N-H group

from amine ($-NH_2$) (11) and vibration of P-O on TPP at 1026 cm^{-1} . The loss FTIR spectra of Chi-TPP were C=O group, CH₃ group on amide ($-NHCOCH_3$) and impurities of silica mineral. (Silverstein *et al.*, 1981). The XRD pattern of Clam shells, Chi and Chi-TPP are shown in Fig.3.

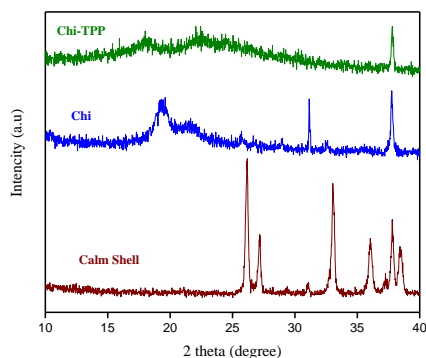


Figure 3.. XRD pattern of Clam shells, Chi and Chi-TPP (magnification 10.000x)

The diffraction of Clam Shell has crystalline structure because there are still many minerals. XRD pattern of Chi has two characteristic peaks at $10,15^\circ$ and $19,94^\circ$ but still has impurities in peaks at 32° and 38° . The characteristic XRD pattern of Chi-TPP has a broad at $19,85^\circ$ and 23.50° . (Bhumkar dan Pokharkar, 2006). Diffraction of Chi is crystallite structure, Cross-linking of TPP on Chi where these two peaks disappeared in XRD pattern of Chi-TPP, which indicates amorphous shape.

SEM image of Chi and Chi-TPP are shown in Figure 4. The morphological surface of Chi has rigid colloid-shaped and then SEM image of Chi-TPP showed threads-shaped of cross-linker TPP on chi-surface and stabilizes them.

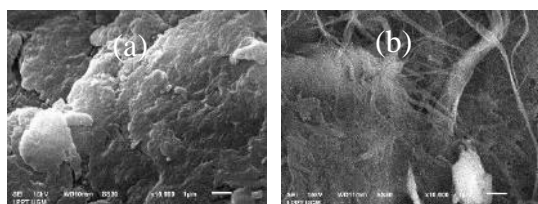


Figure 4.. SEM image of Chi and Chi-TPP

Determination of the optimum pH was carried out to determine the optimum pH conditions to adsorb cadmium ion using Chi-TPP. The effect of pH adsorption of cadmium ion on Chi-TPP are shown in Fig.5

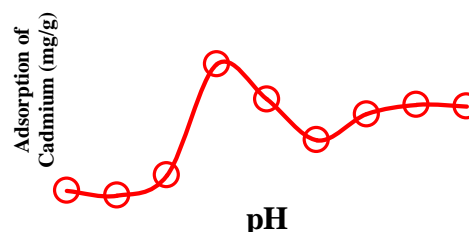


Figure 5.. The Effect of pH adsorption of cadmium ion on Chi-TPP

Based in Figure 5, pH range experiment was varied from 1.0-9.0. At low pH, ion H^+ has high concentration and compete with Cd ion for sorption. However, if pH solutions was increased, the sorption of Cd was increased because concentration of ion H^+ due to decreased. Optimum pH for sorption of Cd ion on Chi-TPP at pH 4.0 Increasing the pH further was resulted in a decreased for sorption until at pH 7. At pH 8-9, the sorption of cadmium ion was increased because Cd ion has started to precipitate (Seyedi *et al.*, 2013)

Conclusions

The successful isolation of chitin from Clam Shells (*Cerithidea obtusa*) and synthesis of Chi-TPP from chitin. XRD pattern of Chi-TPP has characteristic peak is amorphous shape. SEM image of Chi-TPP is rigid-colloid with cross-linking of TPP. Optimum pH Adsorption of cadmium ion on Chi-TPP at pH 4.

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