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Synthesis and Characterization of Tripolyphosphate Chitosans through a Crosslinking Process from High Chemical Weight Chitosans

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Abstract: This study aims to synthesize and characterize chitosan-tripolyphosphate (CTPP) through ionic crosslinking between chitosan and sodium tripolyphosphate. The CTPP powder was characterized using Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM), and surface area analysis (BET), alongside solubility, swelling, and point of zero charge (pHZPC) tests. FTIR spectra showed a peak at 1151.5 cm^{-1} attributed to P=O stretching, confirming the interaction between chitosan and tripolyphosphate. XRD analysis indicated shifts in diffraction peaks, suggesting a successful crosslinking reaction. SEM images revealed that the resulting particles had a compact and aggregated morphology. BET analysis showed a decrease in surface area compared to pure chitosan, which may be attributed to particle agglomeration upon crosslinking. Solubility tests revealed that while chitosan is soluble in acidic media, the CTPP product is insoluble. The CTPP showed a swelling percentage of 60% in acidic conditions, indicating its potential responsiveness. The pHZPC measurement indicated a value of 6.2, with $\Delta pH = 0$, suggesting moderate surface charge behavior in neutral pH conditions. Overall, the study confirms the successful synthesis of crosslinked CTPP with distinct physicochemical properties suitable for further application development.

Keywords: Characterization; Chitosan; Crosslinking; Molecular weight spectroscopy; pHZPC; Synthesis; Tripolyphosphate

Introduction

Chitosan is a poly-(β-1,4-D-glucosamine) derived from the deacetylation of chitin, which is abundantly available from crustacean shells such as shrimp and crabs. It is soluble in dilute organic acids such as acetic, lactic, malic, formic, and succinic acids, and is commonly used in various forms including powders, flakes, beads, membranes, coatings, fibers, hollow fibers, and scaffolds. Due to its biocompatibility, biodegradability, and non-toxicity, chitosan has been extensively applied in biomedical fields, including as a wound dressing material, drug delivery system, and tissue engineering scaffold (Mohanasundaram et al., 2022).

Despite its promising potential, native high molecular weight (MW) chitosan has poor solubility in water, limiting its practical applications. To improve its solubility and biological activities, including antibacterial and plant growth-promoting effects, depolymerization techniques are often employed to reduce its molecular weight. Lower MW chitosan exhibits enhanced solubility and increased biological

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activity, making it more effective in diverse applications such as medicine, agriculture, and environmental remediation (Li et al., 2025).

To address sustainability and valorize waste, researchers have utilized crab shell waste (Portunus sanguinolentus L.) as a raw material for chitosan production. In Maluku Province, Indonesia, where crab production is high, chitosan has been explored as an adsorbent for methylene blue dye (Tanasale et al., 2016). Further developments have included combining chitosan with other materials to improve its performance. For instance, TiO_2/chitosan composites have been used as photocatalysts (Fajriati et al., 2017), silver nanoparticles-chitosan for colorimetric detection (Avissa & Alauhdin, 2014), and zeolite/chitosan composites as slow-release iron fertilizers (Iqbal, 2018). Additionally, chitosan has been applied as a coating on textile surfaces (Saputra, 2016).

However, one key limitation in using chitosan as an adsorbent under acidic conditions is the protonation of the amine groups ($–NH_2$ to $–NH_3^+$), which reduces its capacity to chelate metal ions. Moreover, chitosan tends to dissolve in strongly acidic environments such as sulfuric acid (H_2SO_4), while being only partially soluble in other mineral acids like nitric acid (HNO_3), hydrochloric acid (HCl), and perchloric acid ($HClO_4$) (Hastuti, 2011). These properties limit its structural integrity and functional performance under such conditions (El-Naggar et al., 2024).

To overcome these challenges, chemical modifications have been explored to enhance chitosan's mechanical stability, acid resistance, and adsorption capacity. One widely studied strategy is chemical cross-linking, which creates a covalently bonded matrix, reducing solubility and enhancing structural stability. Common cross-linking agents include glutaraldehyde, ethylene glycol diglycidyl ether, and epichlorohydrin. However, glutaraldehyde's toxicity poses environmental and health concerns (Hassan et al., 2023; El-Naggar et al., 2024).

As an alternative, sodium tripolyphosphate ($Na_5P_3O_{10}$) offers advantages such as being non-toxic, cost-effective, and approved as a food additive. The cross-linking of chitosan with sodium tripolyphosphate occurs through ionic interactions between the protonated amine groups of chitosan and the anionic phosphate groups, forming a stable, insoluble network. This process, often referred to as ionic cross-linking, improves the mechanical strength and acid resistance of chitosan without introducing toxic residues (El-Naggar et al., 2024; Lee et al., 2023).

In this study, chitosan-tripolyphosphate microparticles were synthesized via ionic cross-linking at varying chitosan concentrations. The resulting materials were then characterized to evaluate particle morphology, chemical structure, solubility, swelling behavior, and the point of zero charge (pH_ZPC). This research aims to contribute to the development of eco-friendly and effective chitosan-based adsorbents suitable for use in acidic environments (El-Naggar et al., 2024; Riaz et al., 2023).

Method

Materials

High molecular weight chitosan (Mw = 310,000–370,000 g/mol) was obtained from Sigma-Aldrich. Sodium tripolyphosphate ($Na_5P_3O_{10}$) was also purchased from Sigma-Aldrich. Acetic acid (CH_3COOH), potassium nitrate (KNO_3), nitric acid (HNO_3), sodium hydroxide (NaOH), and distilled water (aquadest) were acquired from Merck and used as received. All reagents were of analytical grade.

Instrumentation

The instruments used in this study included: Analytical balance (Ohaus), glassware set and spatula, hot plate and magnetic stirrer (Cimarec), 100-mesh sieve, digital pH meter (SevenEasy), Oven (Lab Shell), Fourier Transform Infrared (FTIR) Spectrophotometer, scanning Electron Microscope (SEM, FEI Inspect S40), X-ray Diffractometer (XRD, PANalytical X'Pert Pro), Surface Area Analyzer (Quantachrome tipe Nova 1200e)

Methods

Preparation of Chitosan-Tripolyphosphate

The preparation method followed the procedure described by Ngah et al. (2010) with slight modifications. Chitosan solution was prepared by dissolving 60 mg of chitosan powder in 20 mL of 5% (v/v) acetic acid and allowing it to stand overnight at room temperature to ensure complete dissolution. The chitosan solution was then slowly added dropwise to 100 mL of 0.05 M sodium tripolyphosphate (TPP) solution, which had been previously adjusted to pH 8, under continuous magnetic stirring at 400 rpm. The addition rate was maintained at approximately 1 mL/min to facilitate the controlled formation of chitosan-TPP granules. After complete addition, stirring was continued for an additional 2 hours to ensure thorough cross-linking. The resulting gel was left immersed in the chitosan-TPP mixture for 12 hours at room temperature. The formed chitosan-TPP beads were then collected by filtration, washed several times with distilled water to remove excess reagents, and dried in an oven at 60–80 °C for 6 hours. The dried material was crushed and sieved through a 100-mesh sieve to obtain a uniform particle size.

Characterization of Chitosan-Tripolyphosphate

The synthesized chitosan and chitosan-tripolyphosphate were characterized using Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD, Shimadzu XRD-7000), Scanning Electron Microscopy coupled with Energy Dispersive X-ray Analysis (SEM-EDX, FEI Company Quanta 450), and Surface Area Analyzer (Quantachrome Nova 1200e). FTIR analysis was employed to identify the functional groups present and confirm cross-linking. SEM was used to observe the surface morphology of the samples. XRD analysis provided information on the crystalline structure, while BET was used to determine the specific surface area.

Solubility Test

The solubility of chitosan and chitosan-tripolyphosphate powders was evaluated in three different solvents: 5% (v/v) acetic acid, distilled water, and 0.1 M NaOH solution. A total of 0.05 g of each powder was dispersed separately in 100 mL of each solvent and stirred for 24 hours at room temperature (Ngah et al., 2010). The solubility behavior was assessed visually and is summarized in Table 1.

Table 1. Solubility Effects of Chitosan and Chitosan-Tripolyphosphate

Adsorbent	Solubility Effects."		
	50% Acetic acid	Distilled water	0.1M NaOH
Chitosan	Soluble	Insoluble	Insoluble
Chitosan-tripolyphosphate	Insoluble	Insoluble	Insoluble

Swelling Test of Chitosan-Tripolyphosphate

The swelling behavior of chitosan-tripolyphosphate was evaluated by placing 0.05 g of chitosan-tripolyphosphate powder into a cylindrical tube (4 mm diameter, 75 mm height). The initial height (h_o) of the powder column was marked before adding 1 mL of each solvent: 5% (v/v) acetic acid, distilled water, or 0.1 M NaOH to the tube. The samples were left undisturbed for 24 hours at room temperature. After 24 hours, the swollen height (h_t) of the powder column was measured. The swelling percentage (S) was calculated using the following equation:

$$S = \left(\frac{h_t - h_o}{h_o}\right) \text{x } 100 \qquad (1)$$

Where: h_t = height of the swollen powder (cm) after 24 hours, h_o = initial height of the powder column (cm) (Ngah et al., 2010).

Determination of pHZPC (Zero Point of Charge)

The of pHZPC of chitosan-tripolyphosphate was determined using the pH drift method as described by Shawabkeh et al. (2014). Briefly, 0.05 g of chitosan-tripolyphosphate powder was added to 100 mL of 0.1 M KNO_3 solution. The initial pH of the solution was adjusted between 2 and 11 by adding either 0.1 M HNO_3 or 0.1 M NaOH. The suspensions were stirred for 24 hours at room temperature (22 °C). After equilibration, the final pH values were measured. The difference between the initial and final pH was plotted against the initial pH to determine the of pHZPC.

Result and Discussion

Chitosan with a pKa of 6.3 is a polycationic which will produce NH3+ ion when dissolved in acid. Thus at an acidic pH, the amine ionization of chitosan will increase so that there will be a greater potential for the formation of bonds between chitosan and tripolyphosphate (TPP). The chitosan protonation reaction is presented in Figure 1.

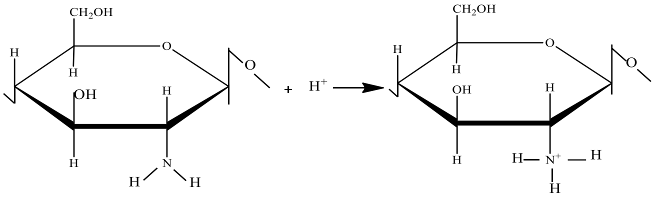


Figure 1. Chitosan protonation reaction

If the TPP solution is in alkaline conditions, deprotonation and ionic cross-linking between chitosan and TPP will occur. Hydroxyl ions and TPP will compete to react ionically with the groups in NH_3^+ chitosan through the formation of ionic crosslinking between chitosan and TPP and the chitosan deprotonation reaction (Figure 2). Chitosan has a pKa of approximately 6.3–6.5, meaning that at the synthesis pH of 8, most of the amino groups ($-NH_2$) exist in their non-ionized form, favoring cross-linking with TPP rather than ionic interactions with protons. Therefore, the pH of the TPP solution, which was maintained at pH 8, is crucial in the synthesis of chitosan-TPP. Chitosan cross-linking with TPP produces a yellowish colour of Chitosan-TPP powder with a yield of 95% (Bodini et al., 2020; Nuraini & Hakim, 2017).

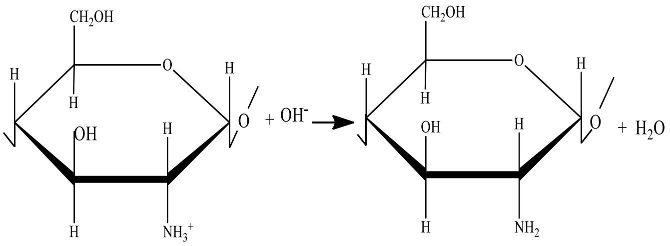


Figure 2. Chitosan deprotonation reaction

Characterization of Chitosan and Chitosan-Tripolyphosphate Fourier Transform Infrared (FTIR)

The formation of chitosan-TPP crosslinks can be seen from the IR absorption in Figure 3. The results of the characterization of chitosan and chitosan-TPP using FTIR are presented in Table 2. In the IR spectra of chitosan tripolyphosphate, a span appears at wave number 1151.5 cm-1 which is a P=O stretching vibration indicating that a bond between chitosan and tripolyphosphate has been. The absorption band at wave number around 3593.38 cm-1 indicates the presence of –OH groups which are thought to cover the NH2 peak due to the formation of wide bands in the same wave number region. The absorption band at wave number 2883.58–2947.23 cm-1 shows the vibration of the aliphatic C–H stretching of CH2. The absorption band in the N–H bending vibration of the primary amide appears at wave number 1651.07 cm-1 while the absorption at wave number 1539.20 cm-1 appears due to the interaction between ammonium ions and phosphate ions.

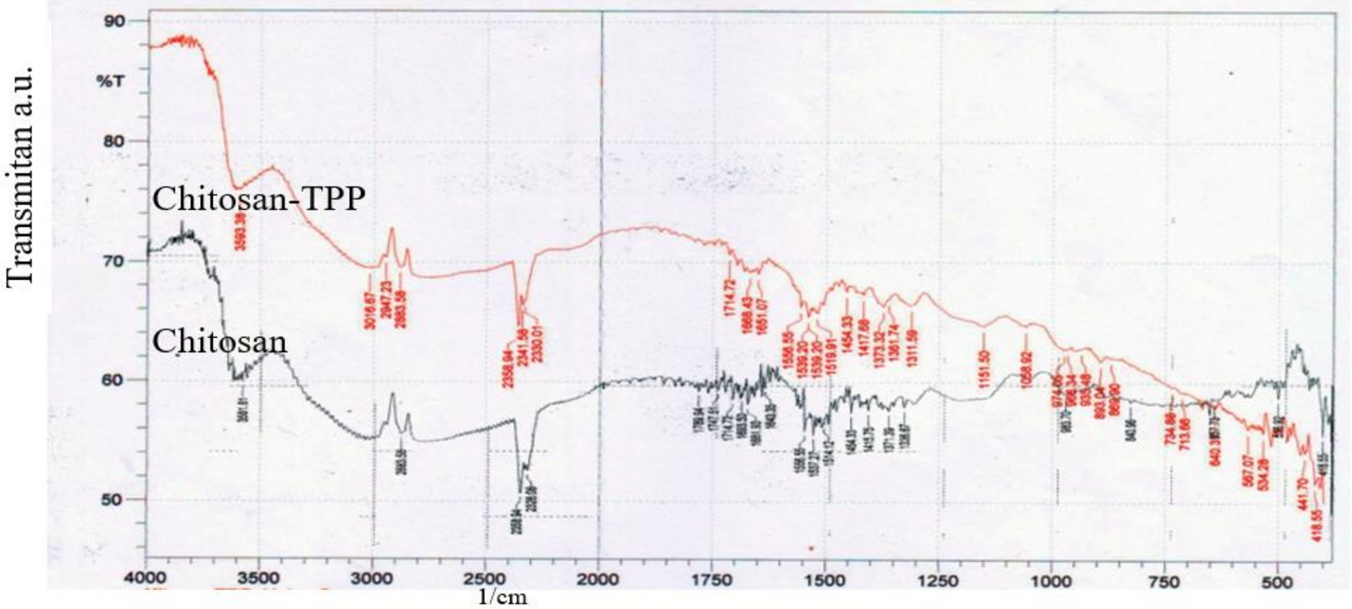


Figure 3. IR spectra of chitosan and chitosan-TPP

Table 2. FTIR Characterization Results of Chitosan and Chitosan-TPP

Sample	Wave number (cm^{-1}) Alauhdin et al. [17]	Tanasale et al. [5]	This research	Functional group
Chitosan		3617.56	3581.81	O–H
		2894.24	2883.58	C–H
		1564.30	1643.35	aliphatic
			1415.75	N–H
				CH3
Chitosan-	3449		3593.38	O–H
TPP	2900		2883.58-2947.23	C–H
	1627-1636		1651.07;1539.20	aliphatic
	1087-1096		1058.92	N–H
	1150-1200		1151.5	C–O
				P═O

In the IR spectrum of chitosan, the peak appears at wave number 1643.35 cm^{-1} which indicates the presence of an amide group. However, two new peaks appeared in the IR spectrum of chitosan tripolyphosphate, namely at 1539.20 cm^{-1} and 1651.07 cm^{-1}. This is due to the interaction between tripolyphosphate ions and NH_3^+ from chitosan. The absorption of the CH_3 bend at wave number 1400s cm^{-1} still appears which indicates the presence of acetyl groups in the tripolyphosphate chitosan because the chitosan used is not completely deacetylated. The C–O range was identified at wave number 1058.92 cm^{-1}.

X-Ray Diffraction (XRD

The data obtained from the XRD analysis is in the form of a diffractogram of the relationship between the X-ray diffraction angle in the sample and the intensity of the light reflected by the sample. The chitosan and chitosan-TPP diffractograms are presented in Figure 4.

Research conducted by the x-ray diffraction pattern of the initial chitosan samples showed a characteristic peak at 2θ = 19.97°. In the chitosan-TPP diffraction pattern, the peak at 2θ = 19.97° shifts to 2θ=19.54°; 19.56°; 19.30°; 19.20°; and 20.42° for chitosan-TPP with chitosan concentration of 0.1; 0.2; 0.3; 0.4; and 0.5, respectively. The results shown were similar to this study, the chitosan x-ray diffraction pattern showed characteristic peaks at 2θ = 8.9281° and 19.7361° and from this pattern it can be seen that chitosan has an amorf structure. In the chitosan diffraction pattern, the peak at 2θ = 19.7361° is shifted to 2θ = 23.1380° for chitosan-TPP. The peak shift in chitosan-TPP indicates that there has been a cross-linking process between chitosan and tripolyphosphate. Sivakami et al explained that the shift in peaks appeared after the cross-linking process, namely from 20 to 25.

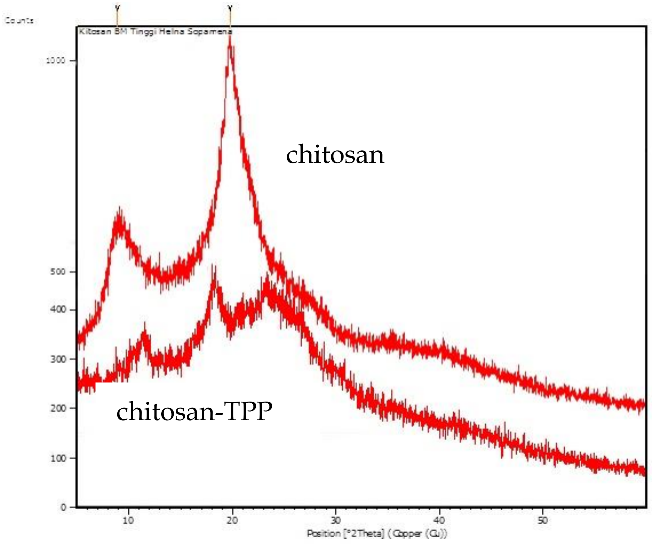
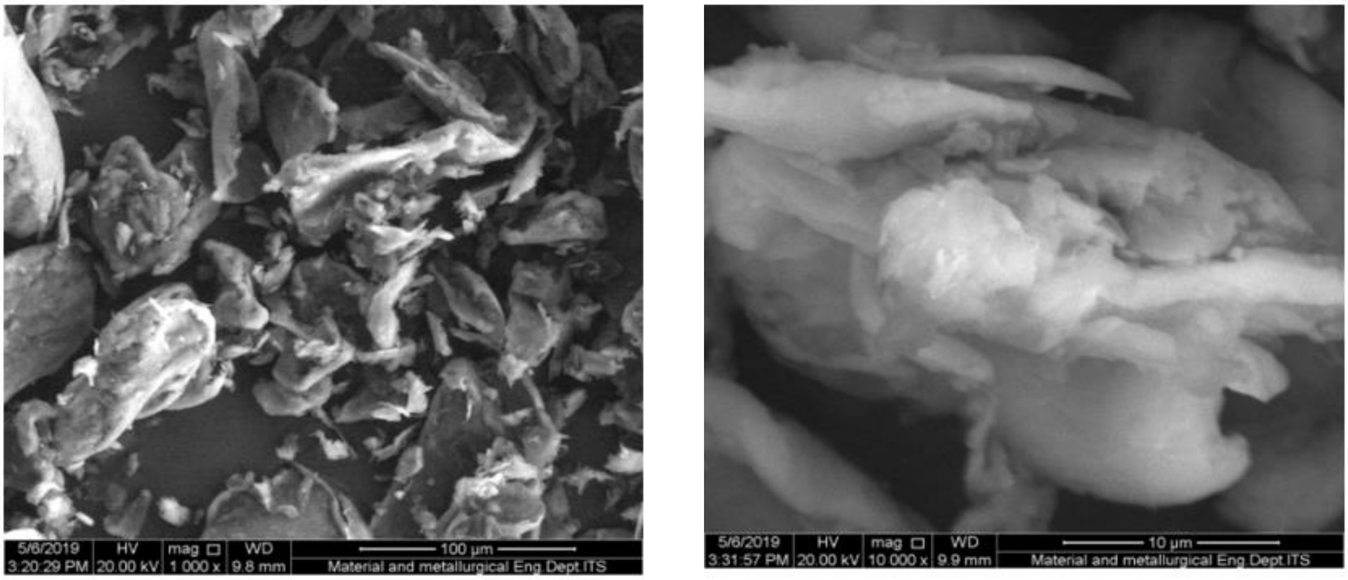


Figure 4. Chitosan and Chitosan-TPP XRD diffractograms

Scanning Electron Microscopy-Energy Dispersive X-ray (SEM-EDX)

The SEM micrographs of native chitosan prior to crosslinking with tripolyphosphate (TPP) were obtained at two different magnifications: (a) 1000× and (b) 10000×, as shown in Figure 5. These images provide a detailed view of the initial surface morphology and structural characteristics of unmodified chitosan. In image (a), recorded at 1000× magnification, the macroscopic structure of chitosan is visible, revealing an uneven surface with an indistinct pore structure. This morphology is typical of raw chitosan, where the surface appears rough and disorganized due to the absence of any crosslinking treatment. Such structural features may limit its mechanical stability and reduce its suitability for applications requiring controlled release or molecular interaction (Bodini et al., 2020).

At higher magnification, image (b) offers a closer look at the microstructure of chitosan. The surface exhibits aggregated particles and a coarse texture, lacking a well-defined porous network. The absence of a uniform and dense structure suggests that the native chitosan matrix is not yet optimized for advanced functional applications such as drug delivery systems or nanoparticle carriers. This comparison between the two magnifications illustrates the baseline morphology of chitosan before chemical modification. The disordered and loosely packed surface structure observed here is expected to undergo significant changes upon crosslinking with TPP, a process intended to improve surface uniformity, porosity, and structural stability (Bodini et al., 2020).



(a) (b)

Figure 5. SEM results of chitosan with (a) 1000x and (b) 10000x magnification

In the EDX spectrum shown in Figure 6, chitosan has three main elements or components namely C, O, and Na. Similar to previous research which produced elements C (34.9%), O (44.3%), Na (14.7%), and N (6.0%). In this study, no peak appeared for the element N, possibly due to the small amount of N that could be detected by EDX.

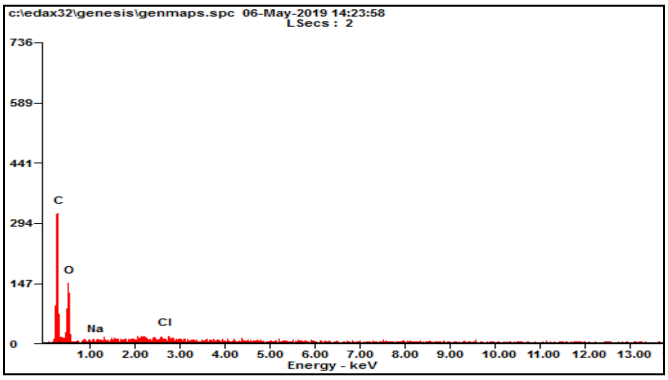


Figure 6. Chitosan EDX results

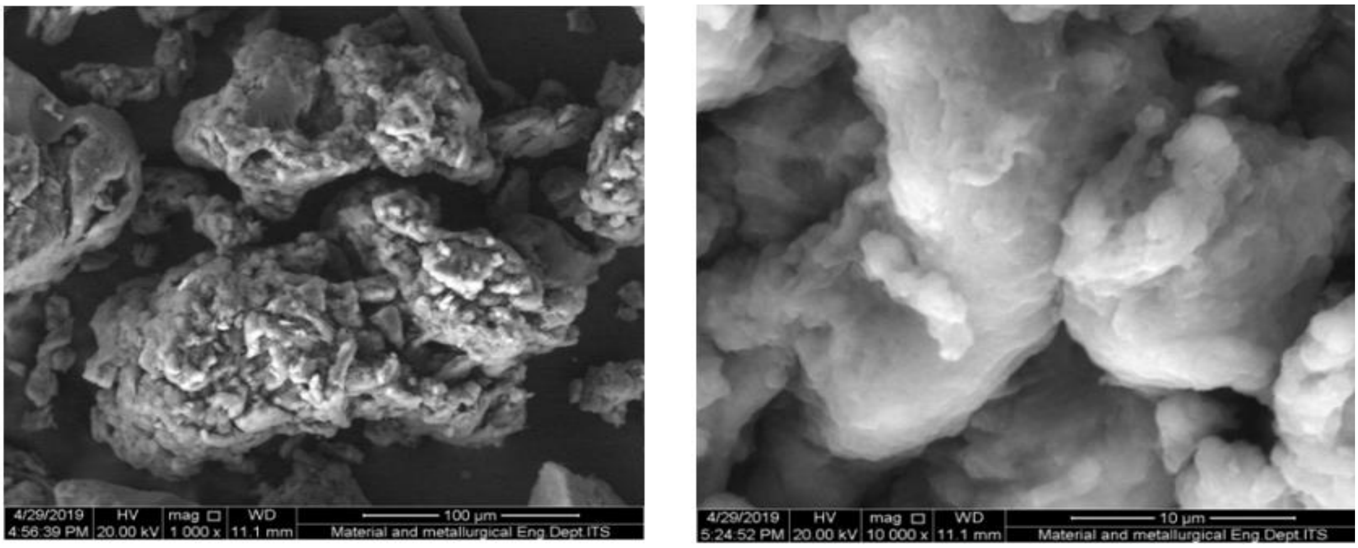
The elemental composition of the synthesized chitosan-tripolyphosphate (chitosan-TPP) was determined using Energy Dispersive X-ray Spectroscopy (EDX). The results showed that the major elements present were carbon (C) at 46.48%, oxygen (O) at 50.11%, sodium (Na) at 1.50%, and chlorine (Cl) at 1.71% (Table 1). The high content of carbon and oxygen is consistent with the organic structure of chitosan, which contains hydroxyl and amine functional groups. However, the presence of sodium and chlorine at relatively high concentrations suggests the existence of residual impurities in the synthesized product. These impurities may originate from the chemical reagents used during the synthesis process, such as sodium-containing salts or buffer solutions, or may result from insufficient washing or purification steps (Bodini et al., 2020).

This raises concerns regarding the overall purity and accuracy of the characterization data. The presence of ionic residues such as Na^+ and Cl^- can influence the physicochemical properties of the chitosan-TPP, particularly its surface charge and adsorption capacity. Therefore, further purification steps such as repeated washing or dialysis are recommended to minimize the presence of such contaminants. To ensure the reliability of the elemental analysis, future studies should include repeated EDX measurements at multiple locations on the sample surface (Bodini et al., 2020).

Table 3. Elemental Composition of Chitosan Based on EDX Analysis

Element	Weight (%)
C	46.48
O	50.11
Na	1.50
Cl	1.71

The morphology of chitosan-TPP nanoparticles was examined using scanning electron microscopy (SEM) at magnifications of 1000× and 10000×, as shown in Figure 3 (a) and (b). The SEM images provide insight into the surface characteristics and particle aggregation behavior, which are essential for evaluating the potential applications of the nanoparticles. At a lower magnification (1000×), the chitosan-TPP particles display an irregular morphology with agglomerates of various sizes and relatively coarse surfaces (Figure 3a). These agglomerated clusters suggest strong interparticle interactions, likely caused by electrostatic attractions between the positively charged amino groups of chitosan and the negatively charged phosphate groups of TPP (Rahmawati & Yusuf, 2021; Bodini et al., 2020). Inadequate stabilization during the ionic gelation process may further promote particle aggregation. Such morphology is commonly observed in biopolymer-based nanoparticles synthesized via ionic crosslinking (Nuraini & Hakim, 2017; Zhang et al., 2023).



(a) (b)

Figure 7. SEM results of chitosan-TPP with (a) 1000 and (b) 10000x magnification

In contrast, at higher magnification (10000×), the SEM image (Figure 3b) reveals finer surface details. The surface of the chitosan-TPP nanoparticles appears uneven with the presence of microporous structures. These features indicate an increased surface area, which may enhance the material's adsorption capacity or interaction with active molecules, depending on the intended application. The rough and non-uniform surface texture is indicative of an amorphous or semi-crystalline structure, in agreement with previously reported characteristics of chitosan-based nanoparticles crosslinked with TPP (Hosseinzadeh & Aghaei, 2021). Overall, the observed morphological features suggest that the chitosan-TPP nanoparticles possess physical characteristics suitable for applications in drug delivery systems, biosorbents, or other biomedical and environmental fields requiring high surface area and biocompatibility (Nuraini & Hakim, 2017; Rahmawati & Yusuf, 2021).

Chitosan-TPP cross-linked is characterized by the presence of the element P in the EDX spectrum presented in Figure 8. Cross-linked chitosan-tripolyphosphate (chitosan-TPP) is characterized by the presence of phosphorus (P), as observed in the EDX spectrum shown in Figure 7. The elemental composition obtained from EDX analysis is presented in Table 4. A phosphorus content of 14.39% was detected, indicating the successful crosslinking of chitosan with tripolyphosphate. This reaction typically involves ionic interactions between the protonated amino groups of chitosan [$(C_6H_{11}NO_4)n$] and the anionic phosphate groups of tripolyphosphate [P_3O_{10}]$^{5-}$, forming a stable crosslinked structure. Sodium (Na) was also detected at 2.56%, likely originating from the sodium tripolyphosphate ($Na_5P_3O_{10}$) used as the crosslinking agent. The presence of chloride (Cl) was not observed in this spectrum, which could be due to its concentration being below the detection limit of EDX (approximately 0.1%) (Khoerunnisa et al., 2021).

Compared to previous studies—for instance, crosslinked chitosan-TPP synthesized at pH 9 showed C (41.93%), O (38.17%), N (14.40%), and P (5.50%), (the P content in this study appears relatively high). This discrepancy may be attributed to differences in crosslinking conditions, chitosan concentration, or insufficient washing, which may lead to residual phosphate retention in the material.

Table 4. Results of Analysis of Chitosan-TPP with EDX

Element	Weight (%)
C	34.29
O	48.76
Na	2.56
P	14.39

BET (Brunauer–Emmett–Teller)

BET (Brunauer–Emmett–Teller) surface area measurements were performed to evaluate the effect of the crosslinking process on the surface characteristics of chitosan and its TPP-crosslinked derivatives synthesized from high molecular weight chitosan. The native chitosan exhibited a relatively high specific surface area of 867.474 m²/g, indicating a loosely packed, porous structure. In contrast, the crosslinked chitosan-TPP samples showed a substantial decrease in surface area, with values of 1.268 m²/g and 1.587 m²/g for materials prepared from 1% and 2% chitosan solutions, respectively. A further sample of chitosan-TPP, analyzed in this study, exhibited a specific surface area of 280.827 m²/g.

The marked reduction in surface area is attributed to the formation of a more compact and aggregated structure during the ionic crosslinking between chitosan and tripolyphosphate. This agglomeration effect likely decreases the accessible surface available for interaction with external molecules. Consequently, the lower surface area of the chitosan-TPP composites may negatively influence their adsorption efficiency, which is a critical parameter for applications such as pollutant removal or drug delivery. These findings underscore the impact of crosslinking conditions and initial chitosan concentration on the morphological and functional properties of the resulting materials (Zhang et al., 2023).

Solubility Test

The solubility test of chitosan and chitosan tripolyphosphate in Table 5. The data from Table 5 shows that in a weakly acidic medium, chitosan is soluble, but chitosan-TPP is insoluble. The solubility of chitosan takes place through a protonation reaction, where the amine group in chitosan will accept the H^+ released by acetic acid so that it becomes positively charged (NH_3^+). The presence of NH_3^+ ions cause chitosan to dissolve. In chitosan-TPP is insoluble in acidic media, so it can be concluded that modification of chitosan by cross-linking makes chitosan-TPP have less solubility in acidic media compared to chitosan. In alkaline media, chitosan is insoluble, while chitosan-TPP is partially soluble. The presence of ionic cross-linking reactions between chitosan and tripolyphosphate and hydroxyl ions causes water to be adsorbed properly in the chitosan-TPP polymer chains (Sari & Putri, 2023).

The results of the measurement of the swelling test of chitosan and chitosan-TPP are presented in Table 5. Chitosan-TPP has the ability to swell in an acidic medium, which is equal to 60%. This is because the protonation of NH_2 and the presence of tripolyphosphate ions can increase the polar group of the chitosan-TPP structure so that it can increase absorption in acidic media. In distilled water and NaOH media, chitosan and chitosan-TPP experienced swelling. However, the swelling percentage of chitosan-TPP was smaller than that of chitosan, namely 40% and 20% for chitosan-TPP and 75% and 100% for chitosan. The swelling ability of chitosan-TPP depended on the degree of cross-linking. In alkaline media, the deprotonation reaction from NH_3^+ to NH_2 in the presence of OH^- so that the swelling is only affected by the presence of the polar group of the tripolyphosphate ion. In previous research, it was found that the swelling index of chitosan-TPP at pH 3 was higher than that of chitosan-TPP at pH 9 (Nuraini & Hakim, 2017).

Table 5. Solubility Test of Chitosan and Chitosan-Tripolyphosphate

Adsorbent			Solubility effect
	CH_3COOH 5%	Distilled water	NaOH 0.1 M
High MW Chitosan	Dissolved	Insoluble	Insoluble
Chitosan-TPP	Insoluble	Insoluble	Partially dissolved

pH_{ZPC} Test

The value of pHZPC, is often used as an important parameter for analysing electrostatic surface changes. Based on the results of measurements and calculations, the graph in Figure 8 is obtained which shows the surface acidity of the resulting chitosan is the difference in pH of the solution before and after the addition of chitosan-TPP into the potassium nitrate solution measured as a function of the pH of the initial solution.

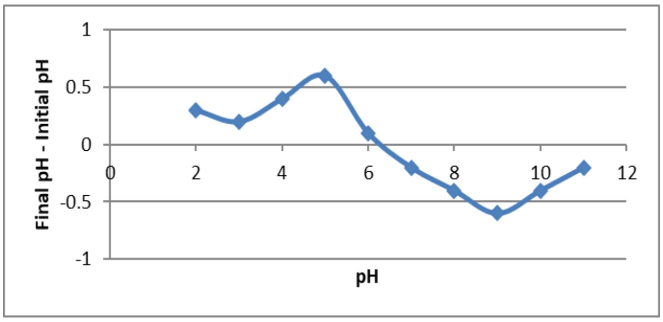


Figure 8. Graph of Chitosan-TPP pHZPC

In this study, the pHZPC of chitosan-TPP was 6.2 which is close to the pKa of chitosan which was 6.3. This means that if chitosan-TPP is in amedium with pH < 6.2, the surface charge of chitosan-TPP will be positively charged, whereas if chitosan-TPP is in medium with pH > 6.2, the surface charge of chitosan-TPP will be negatively charged. In other words, in medium with pH < 6.2, chitosan-TPP is more likely to attract negatively charged molecules or anions, whereas in medium with pH > 6.2, chitosan-TPP is more likely to attract positively charged molecules or cations (Sari & Putri, 2023).

Conclusion

Based on the results obtained, it can be concluded that the synthesis of chitosan-tripolyphosphate (chitosan-TPP) was successfully carried out by reacting a chitosan solution with a tripolyphosphate solution through a cross-linking process, achieving a reaction efficiency of 95%. Characterization using FTIR confirmed the presence of P=O vibration at a wavenumber of 1151.5 cm^{-1}. XRD analysis showed a shift in diffraction peaks, indicating that a cross-linking reaction had occurred between chitosan and tripolyphosphate. SEM analysis revealed that chitosan particles exhibited irregular surface folds, while morphological changes were observed in chitosan-TPP, suggesting structural modification due to cross-linking. EDX spectra confirmed the presence of phosphorus (P) at 14.39%, supporting the incorporation of tripolyphosphate into the chitosan matrix. Solubility tests indicated that chitosan was soluble in acidic media, whereas chitosan-TPP was not. Swelling tests showed that chitosan-TPP had a swelling ratio of 60% in acidic conditions. The pHZPC test revealed that at $\Delta pH = 0$, the point of zero charge (pHZPC) was 6.2. BET analysis showed that pure chitosan had a surface area of 867.474 m^2/g, which significantly decreased to 280.827 m^2/g after cross-linking with tripolyphosphate. This decrease in surface area is likely due to surface agglomeration caused by the cross-linking process, as further supported by morphological changes observed in SEM images. If chitosan-TPP is to be used as an adsorbent, the reduced surface area may negatively impact its adsorption capacity.

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Author Contributions

Methodology.; validation; formal analysis; L. I. A.; investigation; resources.; data curation.; writing— original draft preparation. A. S.; writing—review and editing: visualization: Q. F. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest

The authors declare no conflict of interest.

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