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Role of pH and Kepok Banana Peel Pectin in β-Tricalcium Phosphate Formation from Windowpane Oyster (*Placuna placenta*) Shells

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A B S T R A C T

β-tricalcium phosphate (β-TCP) has potential as a bone material because it is biodegradable, biocompatible, bioresorbable, and osteoconductive. This study aims to synthesize β-tricalcium phosphate (β-TCP) from windowpane oyster shells and to determine the effect of adding kepok banana peel pectin on particle size and pH on the Ca/P ratio. Synthesis was performed with pH variations ranging from 9 to 11 and pectin concentrations of 0.08-0.16%, using a sintering time of 3 hours at 1100ºC via the precipitation method. The resulting β-tricalcium phosphate was characterized using XRF, FTIR, XRD, and SEM analyses. XRF results showed that variations in pH and pectin concentration affected the Ca/P ratio. All samples had a Ca/P ratio higher than the β-TCP standard (1.5). XRD analysis revealed the formation of peaks identical to the JCPDS β-TCP standard no. 09-0169. However, the formation of calcium pyrophosphate (CPP), hydroxyapatite (HAp), and calcium oxide (CaO) phases was also observed in the results of this study. XRD analysis yielded an average crystal size of 37.48 nm and a degree of crystallinity of 53.55%. FTIR results for all samples showed characteristic phosphate group bands consistent with the β-TCP phase. SEM characterization showed that at pH 10 with the addition of 0.12% pectin, the morphology ranged from semi-spherical to granular with relatively uniform particle size and low agglomeration, with an average particle size of 1.8237 µm and a PDI of 0.08. The sample at pH 10 and a concentration of 0.12% exhibited the highest crystallinity and the smallest particle size. Although the Ca/P ratio did not meet the standard, it was 2.03, the XRD and FTIR test results confirmed the presence of phases and groups identical to those found in β-TCP.

Contribution to Sustainable Development Goals (SDGs):
SDG 3: Good Health and Well-Being
SDG 9: Industry, Innovation, and Infrastructure
SDG 12: Responsible Consumption and Production
SDG 13: Climate Action
SDG 14: Life Below Water

1. INTRODUCTION

1.1. Research Background

The increasing incidence of bone disorders resulting from injury or the aging process has led to a growing demand for biomaterials for bone reconstruction. One widely developed solution is the use of bone substitute materials or bone grafts, which play a role in accelerating bone growth by inducing the formation of new bone tissue [1]. Hydroxyapatite has long been used as a bioceramic material due to its crystalline and stable structure. However, this stability makes hydroxyapatite difficult to degrade in the body, leading to deformities and the risk of bone fractures around the

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implant [2]. Another type of biomaterial that can be used for bone reconstruction is β-tricalcium phosphate (β- $Ca_3(PO_4)_2$, which has a Ca/P molar ratio of 1.5. β-tricalcium phosphate (β-TCP) exhibits biodegradability that aligns with the rate of bone growth and possesses osteoconductive properties, making it a more promising biomaterial [3].

Conventional β-TCP synthesis still relies on synthetic chemicals and energy-intensive processes that are not environmentally friendly. Utilizing natural sources such as windowpane oyster shells can serve as an alternative for producing β-TCP in a more sustainable manner. Windowpane oyster shells contain 96.5% calcium in the form of calcium carbonate ($CaCO_3$) [4]. This approach aligns with the principles of the circular economy, which involves transforming waste into a valuable resource. However, β-TCP synthesized from oyster shells often still exhibits a coarse morphology and high agglomeration [5]. This can be addressed by using a capping agent to achieve a more optimal particle dispersion [6].

One natural material with potential as a capping agent is banana peel [7]. This study utilizes the pectin content in banana peel as a capping agent in β-TCP synthesis. The carboxyl groups ($-COO^-$) in pectin compounds have a high affinity for calcium ions, enabling them to bind and stabilize the forming crystal nuclei [8]. Previous studies have demonstrated the effectiveness of plant-based capping agents, such as *Parkia biglobosa* and peel of prickly pear (*Opuntia ficus indica*), in producing finer and more uniform hydroxyapatite [9][10]. However, the application of pectin as a capping agent in β-TCP synthesis has not yet been reported.

β-TCP can be synthesized using various methods, such as sol-gel, hydrothermal, solvothermal, and precipitation. Among these methods, the precipitation method was chosen because of its simple mechanism, low cost, and fast reaction rate [11]. This study was conducted with pH variations ranging from neutral to slightly basic to determine their effect on the Ca/P ratio and the crystallinity of the product [12][13]. Additionally, the concentration of banana peel pectin was varied, as it is expected to play a role in controlling particle size and preventing agglomeration, thereby yielding β-TCP with a uniform morphology [7][8]. This study aims to analyze the effects of pH variations and pectin concentration from banana peels on the phase formation, morphology, and crystal structure of β-tricalcium phosphate synthesized from windowpane oyster shells using the precipitation method. Previous studies have primarily employed pectin as a capping agent in the synthesis of hydroxyapatite (HAp) using synthetic calcium precursors. In contrast, the present study investigates the use of kepok banana peel pectin in the synthesis of β-tricalcium phosphate (β-TCP) utilizing calcium derived from windowpane oyster shell waste as a natural calcium precursor. The main focus of this study lies in the integration of two biological waste sources, banana peels and windowpane oyster shells to produce high-value biomaterials through a green synthesis approach. The results of this study are expected to contribute to achieving SDGs Goal 3, which focuses on improving health through the development of safe biomaterials, as well as Goal 12 through the implementation of sustainable production based on organic waste [7][14].

1.2. Literature Review

1.2.1. Windowpane oyster Shells

Windowpane oyster shells contain 96.5% calcium in the form of calcium carbonate ($CaCO_3$) [4], making them suitable as raw material for the production of β-tricalcium phosphate, which is used in bioceramic materials.

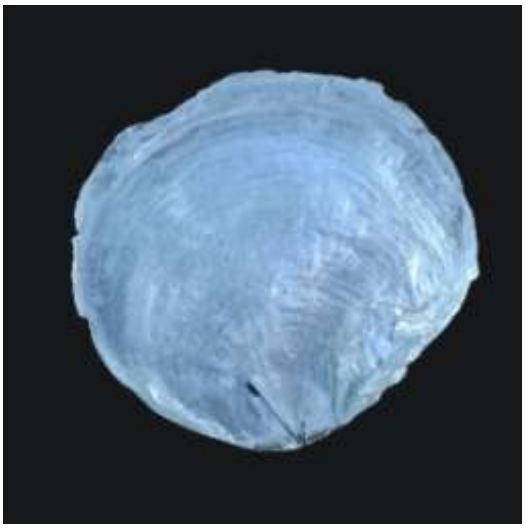

Figure 1. Windowpane oyster shells (*Placuna placenta*)

Based on Figure 1, the windowpane oyster shells has a round shape with a large, symmetrical shell between the left and right valves. The surface of the shell is translucent and displays a gradation of colors when exposed to light. The average shell size of windowpane oyster shells found in Ujung Pangkah, Gresik, and Junganyar Socah, Bangkalan, ranges from 9 to 12 cm [15].

1.2.2. β- Tricalcium Phosphate

β-tricalcium phosphate (β-TCP) is one of the crystalline phases of calcium phosphate with the chemical formula $Ca_3(PO_4)_2$ and a Ca/P molar ratio of 1.5. β-TCP is a calcium phosphate compound with potential for use as a bone material due to its biodegradable, biocompatible, bioresorbable, and osteoconductive properties [1]. β-TCP degrades 10-20 times faster than hydroxyapatite. Implants made of β-TCP generally result in better bone remodeling compared to hydroxyapatite during the final stage of bone formation [16]. β-TCP is highly ideal as a bone graft substitute because it is biocompatible, osteoconductive, and can be formed into powder, blocks, or porous scaffolds that support bone cell growth and vascularization [17].

1.2.3. Pectin from Kepok Banana Peels

Pectin has the ability to trigger nucleation and crystal growth, thereby enhancing the bone regeneration process [10]. One type of banana that is particularly rich in pectin is the kepok banana. The skin of the kepok banana contains 3.557% pectin [18]. Banana peel pectin is classified as low-methoxyl pectin, with a methoxyl content of 2.62% [19]. Low-methoxyl pectin contains the majority of its carboxyl groups in the free form ($-COO^-$), as only about 20-40% of the carboxyl groups are esterified. The carboxyl groups ($-COO^-$) in pectin compounds have a high affinity for calcium ions, enabling them to bind to and stabilize the forming crystal nuclei. This process prevents agglomeration

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and produces particles with a more uniform size. The use of banana peel pectin as a capping agent aligns with the principles of green chemistry, which emphasize the utilization of renewable raw materials and the reduction of hazardous substances [7].

1.2.4. Synthesis of β-tricalcium phosphate

The synthesis of β-TCP is carried out using calcium and phosphate precursors. The calcium precursor, $CaCO_3$, is obtained from the shells of the windowpane oyster shells, while the phosphate precursor used is phosphoric acid. Calcium carbonate ($CaCO_3$) is heated in a calcination process, decomposing into calcium oxide (CaO). The calcium oxide is then reacted with phosphoric acid to produce tricalcium phosphate and water [13]. The reactions occurring in the synthesis of β-tricalcium phosphate can be expressed in reactions (1)-(2).

$$CaCO_3\,(s) \rightarrow CaO\,(s) + CO_2\,(g) \quad (1)$$

$$3CaO\,(s) + 2H_3PO_4\,(l) \rightarrow Ca_3(PO_4)_2\,(s) + 3H_2O\,(l) \quad (2)$$

1.3. Research Objective

The purpose of this study is to synthesize β-tricalcium phosphate (β-TCP) from windowpane oyster shells (*Placuna placenta*) and to determine the effect of adding pectin from kepok banana peels on particle size and pH, as well as on the Ca/P ratio.

2. MATERIALS AND METHODS

2.1. Materials and Tools

The materials used in this study were windowpane oyster shells powder, kepok banana peel pectin powder (Madeinindonesia Surabaya), sodium hydroxide (NaOH, Merck), phosphoric acid (H_3PO_4 85%, Merck), and distilled water (H_2O). The equipment used in the synthesis process included a magnetic stirrer, thermometer, analytical balance, 100-mesh sieve, oven, and furnace. Characterization instruments included XRF (EPSILON 4), FTIR (SHIMADZU), XRD (PANalytical), and SEM (HITACHI FLEXSEM 1000).

2.2. Methods

2.2.1. Pre-Treatment of Windowpane oyster Shells

The powdered windowpane oyster shells was screened to a particle size of 100 mesh and then calcined at 1000 ˚C for 5 hours. This process yields calcium oxide (CaO), which is the product of the decomposition of calcium carbonate ($CaCO_3$).

2.2.2. Synthesis of β-Tricalcium Phosphate

The synthesis of β-tricalcium phosphate was carried out using a precipitation method. Banana peel pectin from the Kepok variety, at concentrations of 0.08%, 0.12%, and 0.16% w/w, was dissolved in 50 mL of distilled water using a magnetic stirrer at 80˚C and 300 rpm. Subsequently, 1.6824 g of calcium oxide (CaO) dispersed in 50 mL of distilled water was mixed with the pectin solution and continuously stirred for 1 hour. Then, 50 mL of 0.4 M phosphoric acid (H_3PO_4) solution was slowly added dropwise. The mixture's pH was then checked using pH paper. Afterward, 0.5 M NaOH was added to adjust the pH to 9, 10, and 11. The mixing process was carried out in a magnetic stirrer at 300 rpm for 1 hour, maintaining a temperature of 80 ˚C throughout. Afterward, the mixture was allowed to settle for 24 hours, resulting in the formation of tricalcium phosphate ($Ca_3(PO_4)_2$) precipitate. The mixture was then filtered using filter paper, and the precipitate was collected. The precipitate was washed with distilled water until the wash filtrate reached a neutral pH. The precipitate was dried in an oven for 4 hours at 110 ˚C.

2.2.3. Sintering

The tricalcium phosphate precipitate obtained through the precipitation process is placed in a furnace. The sintering process is carried out at a temperature of 1000 ˚C for 3 hours. This heating aims to improve the purity and phase stability of the material. During the sintering process, solid-state diffusion occurs, causing grain growth and material densification, resulting in a more ordered and stable crystal structure in the form of the β-tricalcium phosphate phase.

2.2.4. Characterization and Analysis

The synthesized β-tricalcium phosphate was characterized using X-ray Fluorescence (XRF) to determine the Ca/P molar ratio and identify the elemental composition, Fourier Transform Infrared Spectroscopy (FTIR) to identify functional groups, X-Ray Diffraction (XRD) to identify the crystal phase and degree of crystallinity, and Scanning Electron Microscopy (SEM) to determine the surface morphology of the particles and the average particle size using ImageJ and Origin software.

3. RESULT AND DISCUSSION

3.1. Analysis of XRF Results

The material, which was successfully synthesized using a precipitation method based on variations in pH and pectin concentration, was characterized using XRF (X-Ray Fluorescence). The purpose of this was to determine the elemental composition of calcium and phosphorus in the product. Based on the composition of these two elements, the resulting Ca/P molar ratio was then determined. Based on the XRF results, the Ca/P molar ratio is shown in Table 1.

Table 1. Ca/P Molar Ratio of Synthesized Products

pH	Pectin (%w/w)	Ca (%w/w)	P (%w/w)	Ca (mol)	P (mol)	Ca/P molar ratio
9	0.08	72.97	26.44	1.82	0.85	2.14
	0.12	73.21	26.11	1.82	0.84	2.17
	0.16	72.05	27.09	1.79	0.87	2.06
10	0.08	75.22	24.20	1.88	0.78	2.41
	0.12	72.06	27.37	1.79	0.88	2.03
	0.16	72.47	26.80	1.81	0.86	2.10
11	0.08	75.75	23.67	1.89	0.76	2.49
	0.12	72.99	26.34	1.82	0.85	2.14
	0.16	72.80	26.50	1.82	0.86	2.12

From Table 1, it can be observed that variations in pH and pectin concentration in kepok banana peels affect the calcium and phosphorus content of the synthesized products. The obtained Ca/P molar ratios ranged from 2.03 to 2.49, which are higher than the stoichiometric Ca/P ratio of 1.5 for β-TCP. These elevated

values indicate the predominance of calcium-rich phases in the synthesized materials, reflecting an excess of calcium incorporation during the precipitation process. Ca/P ratio greater than 1.5 may indicate the formation of secondary calcium phosphate phases, including hydroxyapatite (HAp) or calcium-deficient hydroxyapatite (CDHA), which exhibit a stoichiometric Ca/P ratio of 1.67, higher than that of β-TCP. The existence of these phases can be verified through further characterization using X-ray diffraction (XRD) analysis [20]. The sample closest to the standard Ca/P molar ratio was the sample with a pH of 10 and a pectin concentration of 0.12%, yielding a Ca/P molar ratio of 2.03.

3.2. *The Effect of pH on the Ca/P Molar Ratio*

Based on the XRF results, a correlation can be established between the effect of pH and the Ca/P molar ratio. This correlation is illustrated in Figure 2.

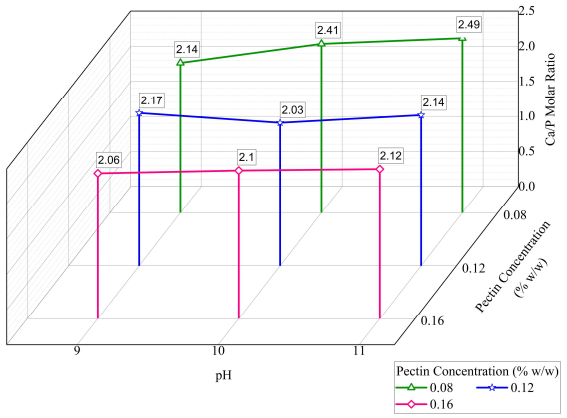

Figure 2. The Effect of pH on the Ca/P Molar Ratio

Figure 2 shows that changes in the synthesis pH affect the composition of the precipitated calcium and phosphorus in synthesized products. In general, the Ca/P molar ratio increases as pH rises, indicating a direct relationship between pH and Ca/P. This increase in the ratio suggests that a basic environment enhances the tendency for calcium-rich phases to form. At higher pH conditions, the speciation of phosphate in the solution shifts from $H_2PO_4^-$ to HPO_4^{2-} and PO_4^{3-}, thereby increasing reactivity toward Ca^{2+} ions. Nucleation and precipitation rates increase, causing some Ca2+ ions to precipitate more quickly onto the crystal nucleus surface before fully reacting with phosphate. Consequently, calcium tends to be incorporated into the precipitate in greater amounts before reacting stoichiometrically with phosphate, resulting in a product with a high calcium content. This phenomenon indicates that the precipitation process occurs faster than the processes of diffusion and ionic composition equilibration in the solution [21]. In addition, research by Irwansyah et al. [22] shows that an imbalance between calcium and phosphate ions during the nucleation process can lead to the formation of a calcium phosphate phase mixture, resulting in a higher Ca/P molar ratio in the product than the theoretical value for β-TCP. Consistent with these findings, Sadlik et al. [23] suggested that Ca/P ratios above 1.5 may indicate the presence of calcium-rich phases or residual $Ca(OH)_2$ remaining after synthesis. Although excess calcium can potentially enhance the bioactivity of the material through a more rapid release of Ca^{2+} ions. However, deviations from the ideal Ca/P ratio can affect the phase stability and dissolution properties of the material, thereby potentially modifying its degradation rate under physiological conditions when applied as a bone substitute.

Ca/P molar ratio closer to the theoretical value for β-TCP can be achieved by optimizing the precursor composition and synthesis conditions. Reducing the concentration of the calcium precursor or increasing the concentration of the phosphate precursor can be done to balance the ion composition in the solution. The reaction temperature can be increased and the reaction time extended to allow the reaction between calcium and phosphate ions to proceed more optimally [24]. This is done to produce purer β-TCP with a Ca/P ratio closer to 1.5.

3.3. *FTIR Spectra Analysis*

FTIR characterization was performed to identify the functional groups present in the synthesized β-tricalcium phosphate samples. The FTIR spectra of each sample are presented in Figure 3 and discussed in detail based on the identified absorption bands.

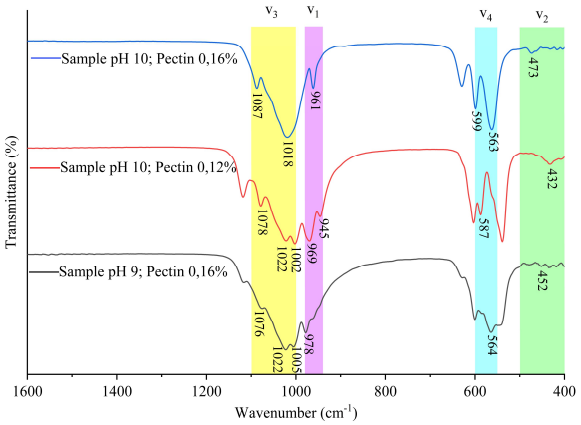

Figure 3. FTIR Spectra of Product Synthesized at Different pH Levels and Pectin Concentrations

The FTIR spectra of the three samples in Figure 3 show absorption bands characteristic of the phosphate group (PO_4^{3-}). The phosphate group (PO_4^{3-}) has four main vibration modes, namely symmetric stretching (v_1), symmetric bending (v_2), asymmetric stretching (v_3), and asymmetric bending (v_4) [25].

Table 2. Wave numbers for the functional groups of β-TCP

Functional Group	(PO_4^{3-}) (cm^{-1})			
	v_1	v_2	v_3	v_4
pH 9; 0.16%	978	452	1005-1076	564
pH 10; 0.12%	945-969	432	1002-1078	587
pH 10; 0.16%	961	473	1018-1087	563-599

All samples exhibited characteristic phosphate group bands consistent with the β-TCP phase. These results are consistent with the study [26], which states that pure β-TCP exhibits characteristic bands due to the symmetric stretching vibration (v_1) at 940-980 cm^{-1}, the asymmetric stretching vibration (v_3) at 1000-1100 cm^{-1}, the symmetric bending vibration (v_2) at 400-500 cm^{-1}, and the asymmetric bending vibration (v_4) at 550-600 cm^{-1}.

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However, there are differences in peak sharpness and band separation among the samples. The β-TCP sample synthesized at pH 9 with 0.16% pectin showed a broader peak. In the pH 10 sample with 0.12% pectin, the phosphate peak was still slightly broadened. Meanwhile, the pH 10 sample with 0.16% pectin exhibited the sharpest peak. The sharpness of this peak indicates greater crystal-structure regularity and more complete β-TCP phase formation than in the other two samples. As pectin concentration and pH increased, the peak became sharper.

Furthermore, the FTIR results showed no absorption bands characteristic of organic pectin compounds, such as carbonyl (C=O) or carboxyl (COO-) groups. This indicates that the pectin decomposed during sintering and was not present in the material's final structure. Pectin acts as a complexing agent or template during the synthesis process without becoming part of the β-TCP structure after sintering [7].

3.4. XRD Pattern Analysis

XRD characterization was performed to analyze the degree of crystallinity of the synthesized product by identifying the 2θ values and the intensity of the resulting spectrum. The diffractogram of the sample is shown in Figure 4. The analysis was conducted by observing the positions of the diffraction peaks, their relative intensities, and their agreement with the standard β-TCP data.

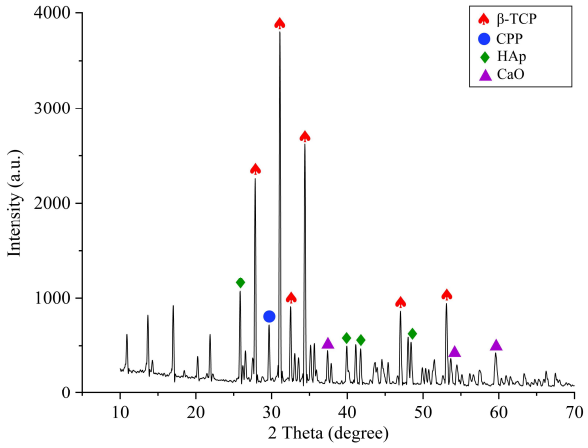

Figure 4. XRD Pattern of Product Synthesized at pH 10 and a Pectin Concentration of 0.12%

Based on Figure 4, the XRD diffraction pattern indicates that the composition of the β-TCP synthesized from windowpane oyster shell is qualitatively similar to the JCPDS β-TCP standard no. 09-0169 [27]. The β-TCP crystalline peak was detected at a 2θ angle of 27,9º, 31,2º, 32,7º, 34,6º, 47,1º, and 53,1º. Although the dominant phase formed in all three samples was β-TCP, the XRD diffraction pattern still showed several additional peaks identified as resembling the Although the dominant phase formed in all three samples was β-TCP, the XRD diffraction patterns still showed several additional peaks identified as being similar to calcium pyrophosphate (CPP), hydroxyapatite (HAp), and calcium oxide (CaO), indicating incomplete phase conversion.

The calcium pyrophosphate (CPP) phase appears at a 2θ angle of approximately 29.6º, which is consistent with JCPDS data no. 09-0346 [28]. The formation of the calcium pyrophosphate phase results from the condensation of hydrogen phosphate ions (HPO_4^{2-}) which remain in the precipitated precursor into pyrophosphate ions ($P_2O_7^{4-}$) during the high-temperature sintering process. These pyrophosphate ions then react with Ca^{2+} ions to form the $Ca_2P_2O_7$ phase [5]. Meanwhile, the presence of HAp in the samples is indicated by peaks at 25,9º, 39,9º, 41,9º, and 48,4º, consistent with JCPDS No. 09-0432 [29]. The presence of HAp indicates that some of the calcium and phosphate ions reacted to form a compound with a higher Ca/P ratio compared to β-TCP. Calcium oxide (CaO) was also detected in the samples, characterized by peaks at angles of 37,4º, 53,7º, and 59,7º, consistent with JCPDS No. 37-1497 [30]. The presence of CaO indicates that some of the calcium resulting from the calcination of simping shell has not reacted completely with phosphoric acid during the synthesis process. This condition may be attributed to a slight imbalance in the precursor composition or inhomogeneity during synthesis [29].

In addition to identifying crystal phases, XRD data can also be used to calculate the crystallite size of a material. The calculation of the β-TCP crystallite size using the Scherrer equation, based on the FWHM values of several major diffraction peaks, is presented in Table 3.

Table 3. Crystallite Size Based on the Scherrer Equation

Diffraction angle (2θ)	FWHM (º)	Crystallite size (nm)
27.9	0.1864	41.36
31.2	0.1869	40.95
32.7	0.1987	38.38
34.6	0.1894	40.05
47.1	0.2176	33.46
53.1	0.2314	30.70
Average		37,48
Standard Deviation		4.01

Further analysis of the XRD data showed that the crystallite size, calculated using the Scherrer equation, was 37,48 nm with a standard deviation of 4.01. This result is smaller compared to the study [31], which did not use pectin in its synthesis and reported a crystallite size in the range of 40-43 nm. The smaller crystallite size indicates that the resulting β-TCP has a more uniform and stable crystal structure [7].

XRD analysis shows that the β-TCP material has a high degree of crystallinity, as indicated by the presence of sharp diffraction peaks and high intensity. The percentage of crystallinity was calculated using the following equation [7]:

$$\text{Crystallinity} = \frac{\text{area of the crystalline diffraction peaks}}{\text{total area of all diffraction peaks in the sample}} \times 100\%$$

The crystallinity value for the pH 10 sample with a pectin concentration of 0.12% was 53.55%. These results indicate that these conditions are optimal for β-TCP formation. In addition to the effect of pH, pectin helps produce a more homogeneous distribution of Ca^{2+} ions through the interaction of its carboxyl groups, resulting in more uniform crystal growth [8].

3.5. Analysis of SEM Results

SEM analysis was performed on a synthesized product at pH 10 with a pectin concentration of 0.12% using magnifications of 1,000× and 5,000×. This was done to determine the surface morphology of the synthesized material. The results of the SEM characterization of the synthesized product is shown in Figure 5.

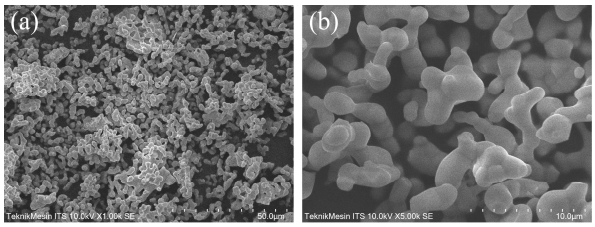

Figure 5. SEM Images Synthesized Product at pH 10 and a Pectin Concentration of 0.12% (a) Magnification 1,000× (b) Magnification 5,000×

Based on Figure 5, the synthesized product at pH 10 with 0.12% pectin addition exhibited particle shapes ranging from semi spherical to granular, with a relatively uniform size distribution and low agglomeration. The quantitative shape analysis supports these observations, with a circularity value of 0.89, indicating that the particles were nearly spherical. An aspect ratio (AR) of 1.51 indicates slight elongation, while a roundness value of 0.79 indicates that the particles maintained a predominantly rounded morphology despite granular features. Overall, these results indicate that the synthesized β-TCP particles possessed a relatively uniform semi-spherical to granular morphology with limited anisotropic growth.

These results are consistent with the study by Pangestu et al. [13], which reported that β-TCP particles typically exhibit morphologies ranging from semispherical to granular. The granular morphology is generally associated with the ripening process during the nucleation and growth stages of calcium phosphate crystals, in which larger crystals tend to absorb or merge with smaller neighboring particles, resulting in the formation of more compact and granular structures. Meanwhile, according to research by Santoso et al. [25], the synthesis of β-TCP without a capping agent such as pectin yields a morphology of small, irregularly shaped particles, many of which have aggregated into clusters. These results indicate that the synthesis of β-TCP without a capping agent leads to significant agglomeration, so that the ideal particle shape of β-TCP is not observed. This indicates that adding pectin, which acts as a capping agent, can control crystal growth and particle dispersion during precipitation. The carboxyl groups in pectin can interact with Ca^{2+} ions, thereby slowing excessive crystal growth and producing more controlled particle sizes while minimizing agglomeration [8].

3.6. Particle Size Distribution

Particle size distribution was determined from SEM micrographs using ImageJ software. At least 150 particles were randomly selected and measured to ensure statistical representativeness. The obtained particle size data were subsequently processed using Origin software and fitted with a Gaussian distribution function to determine the average particle size (xc). Although this image-based method provides valuable inrmation on particle morphology and size distribution, it is less accurate than techniques such as dynamic light scattering (DLS) or laser diffraction, particularly for agglomerated particles. The particle size distribution graph of synthesized product at pH 10 with a pectin concentration of 0.12% is shown in Figure 6.

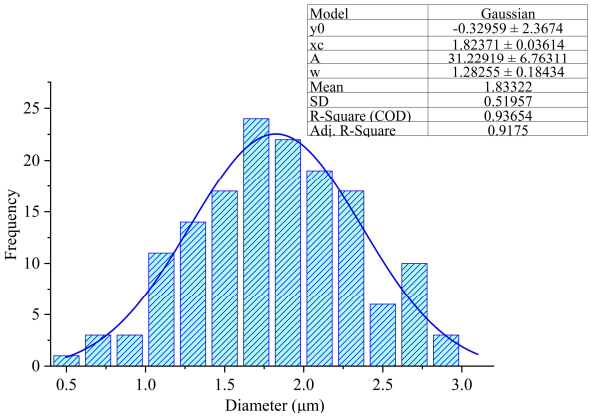

Figure 6. Particle Size Distribution Graph for Synthesized Product at pH 10; 0.12% Pectin

Figure 6 shows that the average particle size (xc) of synthesized product at a pH of 10 and a pectin concentration of 0.12% was ± 1.8237 μm, with a size range of approximately 0.5-3 μm and a standard deviation of 0.5196 μm. The particle size distribution graph also yields an R-square value of 0.93654. The R-square value describes the degree of fit of the approach method to the data, the closer the value is to 1, the more it indicates that the Gaussian method is appropriate for describing the particle size distribution of β-TCP. The degree of particle-size uniformity can be evaluated using the Polydispersity Index (PDI), with values ranging from 0 to 1. If the PDI value is less than 0.1, the particles are classified as monodisperse and if the PDI value is greater than 0.1, the particles are classified as polydisperse. The PDI is calculated as the square of the standard deviation divided by the average particle diameter [32]. The PDI value of the synthesized product pH 10 sample with a pectin concentration of 0.12% is 0.08.

Based on the results of the particle size analysis and PDI values, it can be concluded that the β-TCP synthesized with the addition of pectin exhibits a relatively uniform particle size distribution and is classified as monodisperse. Meanwhile, according to a study by Santoso et al. [25], the synthesis of β-TCP without the addition of a capping agent such as pectin yields particle sizes in the range of 0.8-5.6 μm, resulting in a broader particle size distribution than that of β-TCP synthesized with the addition of pectin. This indicates that pectin acts as a capping agent that controls crystal growth through interactions between carboxylate groups (-COO-) and Ca^{2+} ions, thereby inhibiting excessive crystal growth and reducing particle agglomeration [8].

The average particle size obtained (1.8237 μm) indicates that the synthesized β-TCP material can be classified as a β-TCP microparticle. This particle size falls within the typical range reported for β-TCP microparticles used in biomaterials and bone tissue engineering applications, approximately 1-5 μm [33]. Particle size is an important parameter because it influences the physical, mechanical, and biological properties of the material. Smaller and more uniform particles generally provide a larger specific surface area, which can enhance protein adsorption, cell attachment, and interactions between the biomaterial and surrounding tissues. Furthermore, these characteristics contribute to improved biocompatibility, better osteoconductive performance, and enhanced mechanical strength, thereby increasing the effectiveness of β-TCP as a biomaterial for bone implant applications [1].

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4. CONCLUSION

The synthesis of β-tricalcium phosphate (β-TCP) from windowpane oyster shells with the addition of kepok banana peel pectin via a precipitation process was successfully carried out. Variations in pH affected the Ca/P molar ratio, with the value closest to the β-TCP standard being 2.03. FTIR characterization results showed the presence of characteristic β-TCP functional groups in the form of PO_4^3. XRD characterization confirmed the presence of the β-TCP crystalline phase with a crystallinity of 53.55% and an average crystal size of 37.48 nm. SEM analysis revealed particle shapes ranging from semi-spherical to granular, with an average particle size of 1.8237 μm and a PDI of 0.08.

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