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Synthesis of precipitated calcium carbonate using chicken bones as a raw material for paper

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Abstract: This research discusses the synthesis of precipitated calcium carbonate (PCC) standard for raw paper materials from chicken bones. In the paper industry, PCC is used as a coating and filler to enhance paper's smoothness, brightness, and opacity. The primary material used in this research is chicken bones from the frozen food industry waste, solving environmental problems. Chicken bones were dried in an oven at 100°C, then crushed and sieved to 100 mesh. X-ray Fluorescence (XRF) analysis shows that chicken bones contain calcium at 98.83%. The high content can make chicken bones a raw material for PCC. This research uses variables of Na_2CO_3 solution and precipitation time with a temperature of 30°C. The target of this research to produce high quality Precipitated Calcium Carbonate (PCC) with a calcite crystal structure suitable for industrial applications particularly as raw material for the paper industry. Based on the analysis results, the best conditions were obtained at a Na_2CO_3 concentration of 2.5 M and a precipitation time of 40 minutes. The XRF and XRD test analyses showed that PCC was a polymorph calcite with a particle size of 2.14752 µm. This research proves that chicken bones can be an alternative raw material for PCC by ISO 3262-2:1998 parameters for industry standards.

Keywords: *precipitated calcium carbonate (PCC), chicken bones, paper industry, x-ray fluorescence (XRF), calcite crystal structure*

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INTRODUCTION

Precipitated Calcium Carbonate (PCC) is a form of calcium carbonate obtained through a chemical reaction process from raw materials containing natural calcium carbonate. Precipitated calcium carbonate can form three main types of crystals, calcite, aragonite, and vaterite (Jimoh et al., 2017, p. 1).The difference in the phase structure of $CaCO_3$ is determined by the number of oxygen atoms surrounding each calcium atom, specifically CaO_6 in calcite, CaO_8 in vaterite, and CaO_9 in aragonite (Putri, 2016, p. 6). PCC has a higher price than other calcium carbonate due to its high purity, good chemical absorption performance (Feng et al., 2024, p. 2), and fine micro-scale particle size (Elfina, 2019, p. 2). Additionally, PCC has controllable properties (Maulia, 2020, p. 2) and high particle shape uniformity (Naldi et al., 2023, p. 200). Precipitated Calcium Carbonate (PCC) is often used in various industries such as in the production of paper, paint, plastics, sealants (Ritonga et al., 2022, p. 2327), food, pharmaceutical, and cosmetic industries (Noor et al., 2024, p. 1). In the paper industry, PCC is used as a coating and filler for paper. The presence of PCC enhances the smoothness, brightness, and opacity of paper (Erdogan & Eken, 2017, p. 59).

The demand for paper products increases every year. According to data from Kementerian Perindustrian Republik Indonesia (2024) the paper industry's growth increased by 3.7%. The demand for PCC still relies on natural limestone as the primary raw material. Limiting limestone resources and the increasing industrial demand create a need for sustainable alternative sources of calcium carbonate. Several studies have examined various methods of PCC synthesis, such as carbonation, caustic soda, and precipitation. Some researchers have focused on using organic waste, such as cow bones or eggshells for PCC production. However, no research has discussed the utilization of chicken bone waste as a potential raw material. Chicken bones are waste products available in large quantities from the food industry, but their utilization is still minimal. This results in chicken bone waste because the processing of chicken bones is still not optimal (Jufrinaldi, 2019, p. 70). Chicken bones have a calcium carbonate content reaching up to 85–95%. The high content can make chicken bones a raw material for PCC. The use of chicken bone waste reduces dependence on limestone and offers a solution to environmental problems.

In the study by Wardhani et al. (2021) titled The Effect of PCC Synthesis Temperature with Modifiers on Particle Size and Type, the optimal temperature for obtaining calcite crystals with a size of <10μm is ≤30°C. In the research conducted by Sunardi & Krismawati, 2021titled The Effect of Extraction Time and HCl Concentration on Yield and Calcium Content in the Synthesis of Nano Calcium Oxide from Chicken Eggshells, a hydrochloric acid solution can dissolve calcium ions in calcium carbonate. According to Naldi et al., 2023 in their research titled Removal of Chlorine Content in PCC through Washing and Filtration Processes, PCC with a calcium carbonate content of 98% is required in the paper manufacturing industry. In the study by Laksono et al., 2020 with titled Precipitated Calcium Carbonate from Blood Cockle Shells Using the Double Decomposition Method, the synthesis of PCC using the precipitation method with an agitation speed of 300 rpm resulted in PCC with a calcite crystal structure and a size of 5 μm.

While some researchers have focused on shells, few studies have addressed using chicken bones as raw material in PCC with paper raw material standards. Therefore, this research aims to synthesize PCC from chicken bone waste using the precipitation method by examining the effects of varying reaction times and sodium carbonate concentrations on the crystal structure and the product's compliance with paper raw material standards. This research is to determine the optimal conditions for PCC using the precipitation method resulting in PCC products in the form of calcite polymorphs with particle sizes on the paper industry standards.

METHOD

This research was conducted in 2025 as a laboratory experimental research. The main objective was to synthesize Precipitated Calcium Carbonate (PCC) from chicken bone waste from the frozen food industry. This research was conducted in the materials laboratory of UPN "Veteran" Jawa Timur. The research consists of sample preparation, chemical processing, and product characterization to produce high-quality Precipitated Calcium Carbonate (PCC) with a uniform calcite crystal structure for potential use in the paper industry. The collected data were analyzed using both quantitative and qualitative methods Data analysis combined quantitative methods, including crystal size calculation via the Debye–Scherrer equation on XRD results, and qualitative methods through phase identification and evaluation of synthesis parameters.

This research uses chicken bones from the waste of the frozen food industry PT XYZ. The solvents used are HCl (Pudak Scientific, 12%) and aquadest (Smart Lab). In addition, NaOH (Smart Lab, 98%) is used as a pH regulator and Na_2CO_3 (Pudak Scientific, 3%) as a reagent for forming PCC. The cleaned chicken bones were dried in an oven at 100°C until dry, then crushed and sieved to obtain a uniform size of 100 mesh. A 250 mL solution of 2 M HCl was added to the chicken bone powder. The solution was checked for pH, and if it was too acidic, 0.1 M NaOH was added to adjust the solution's pH to 10. The solution was filtered with filter paper until the precipitate and filtrate were separated.

The filtrate is reacted with varying concentrations of sodium carbonate 0.5 M; 1 M; 1.5 M; 2 M; 2.5 M, each 250 mL, and heated at a temperature of 30°C. Then, it was stirred using a magnetic stirrer at 300 rpm for 10, 20, 30, 40, and 50 minutes. The solution is filtered with filter paper to separate the precipitate from the filtrate. The precipitate is washed with aquadest to remove sodium chloride until a neutral pH. Then, it is dried at room temperature for about 24 hours.

Data collection was performed through laboratory testing. The composition of the obtained PCC was observed using X-ray Fluorescence (XRF) and X-ray Diffraction (XRD). In addition, a pH test was conducted in the range of 9-10 on the PCC because it affects the size of the crystals produced. X-ray Fluorescence is used to identify the type of mineral based on the structure and chemical composition of the formed PCC (Meirawaty, 2022, p. 12) The XRF test was conducted at the Laboratory of Minerals and Advanced Materials University of Malang, and the XRD test at the Energy and Environmental Laboratory Institute of Technology Sepuluh Nopember. X-ray diffraction (XRD) to analyze the phase composition of PCC. Calcite is the only carbonate species detected by XRD (Sari et al., 2024, p. 1632). Phuyal et al., 2024found that the high concentration of calcite identified through XRD confirms PCC's calcium carbonate content. The diffraction formed from various angles will indicate the characterization of the sample, so the crystal size analysis can also use the XRD test with the help of the Debye-Scherrer equation. Sumadiyasa, 2018 state the Debye-Scherrer equation:

$$D = \frac{K\lambda}{\beta\ cos\ cos\ \theta}$$

Remaks:

D is the crystal size

K is the Scherrer constant (0.9)

λ represents the wavelength of X-ray (0.15406)

β represents the full width of half maximum value (radians)

Θ represents the fraction angle (radians)

FINDINGS AND DISCUSSION

Synthesis of PCC with standard paper raw materials from chicken bones is carried out by dissolving chicken bones using a hydrochloric acid solution. After that, a precipitation process is carried out with a sodium carbonate solution at varying solution concentrations and precipitation times. The obtained PCC was then analyzed to determine the $CaCO_3$ content, pH, crystal structure, and crystal size. Before the PCC synthesis, chicken bones are tested using XRF to determine the elemental content. Here are the XRF test results for chicken bones:

Table 1. XRF Test Results of Chicken Bones

Component	Concentration (%)
S	0,49
K	0,39
Ca	98,83
Fe	0,052
Cu	0,12
Sr	0,12

In the paper industry, calcite has become an important element, replacing the role of kaolin in the protective coating of paper. Putri et al. (2016) said the most stable calcium carbonate phase is calcite, which is widely used in the paint, paper, textile, detergent, plastic, and cosmetic industries. With the evolution of paper production techniques from acidic to neutral or alkaline conditions, kaolin, which was previously used as a protective coating, has been replaced by calcite. In the paper industry, metastable calcite is used as a coating and filler.

Table 2. PCC Raw Material Standards Paper based to International Organization for Standardization

Parameter	Standard
Crystal structure	calcite
Crystal size	2µm
Concentration CaCO3	>98%
pH	9-10

ISO 3262-2:1998

The research used chicken bones as a source of $CaCO_3$ for the synthesis of Precipitated Calcium Carbonate (PCC). The process involved dissolving $CaCO_3$ with HCl to form $CaCl_2$, followed by pH adjustment and precipitation with Na_2CO_3. In this reaction, HCl reacts with $CaCO_3$ to produce $CaCl_2$, H_2O, and CO_2, while Na_2CO_3 supplies carbonate ions that bond with calcium ions to form $CaCO_3$ precipitates, separating as solid sediment from the solution.

In Figure 2 shows the effect of Na_2CO_3 concentration and precipitation time on PCC yield. The highest yield, 99.86%, was obtained at a Na_2CO_3 concentration of 2 M with a precipitation time of 50 minutes. Increasing reaction time generally improved PCC formation, as longer stirring enhanced the crystallization process (Amrullah, 2023, p. 150). However, at 2.5 M Na_2CO_3 the yield decreased because all calcium ions had reacted, leaving excess carbonate ions unutilized. Hgher Na_2CO_3 concentrations increase the availability of carbonate ions to react with calcium, thereby improving PCC formation up to the optimum point (Maulia, 2020, p. 4). The precipitation time affects the increase in the yield of the precipitation results. The longer the reaction time, the longer the reaction between calcium and carbonate ions will take. Precipitation times below 25 minutes, the crystal structure of precipitated calcium carbonate is affected, which is not yet perfect, and the calcium carbonate content is low (Amrullah, 2023, p. 153)

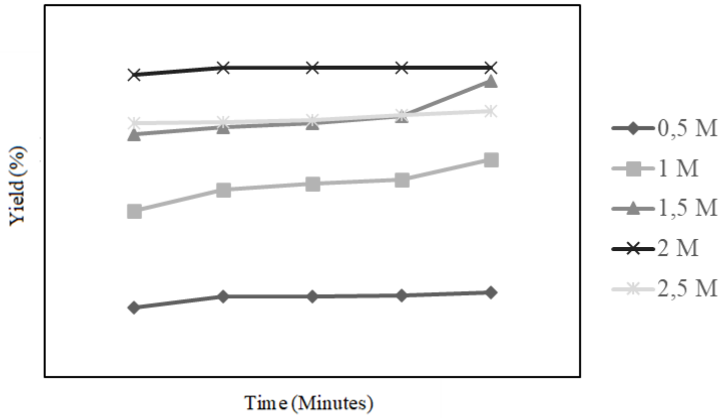

Figure 1. Effect between Na_2CO_3 Concentration and Precipitation Time with PCC Yield

Based on the test results using X-ray Fluorescence (XRF), the data were analyzed using a second-order polynomial regression to determine the optimal conditions for PCC formation. Figure 3 shows that the best condition was achieved at a Na_2CO_3 concentration of 2.5 M with a precipitation time of 40 minutes, producing the highest $CaCO_3$ content of 99.93% with an R^2 value of 0.8275. At lower concentrations, fluctuations in $CaCO_3$ content were observed: 0.5 M showed decreases at 20 and 50 minutes, 1 M at 20 and 30 minutes, 1.5 M at 20 and 40 minutes, and 2 M increased sharply at 20 minutes but decreased at 40 minutes.

Based on figure 3, there is a tendency for the $CaCO_3$ content to increase with the addition of sodium carbonate concentration confirming its critical role in influencing PCC quality and yield. The obtained $CaCO_3$ content exceeded 98%, meeting the ISO 3262-2:1998 standard for paper raw materials. Elfina et al. (2019) who reported that $CaCO_3$ content value is directly proportional to the weight of the obtained PCC the heavier the obtained PCC, the higher the $CaCO_3$ content. The $CaCO_3$ content tends to increase rapidly due to the simultaneous nucleation and crystal growth processes.

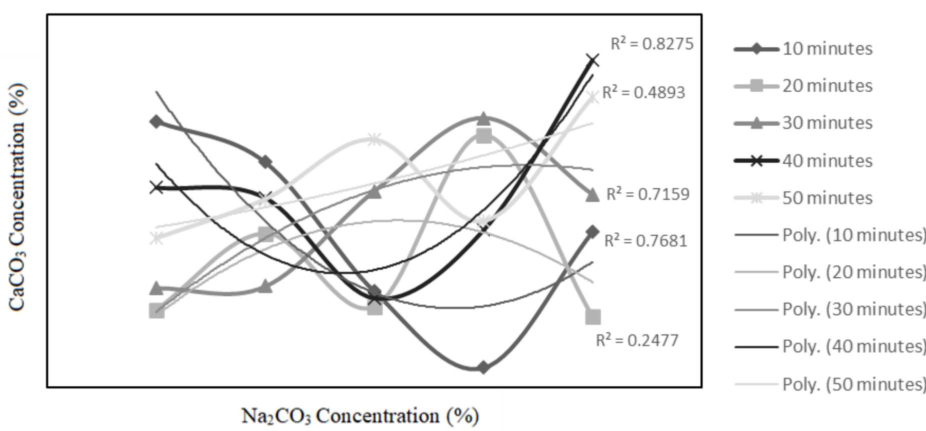

Figure 2. Effect between Na_2CO_3 Concentration and $CaCO_3$ in PCC

In the synthesis process of precipitated calcium carbonate (PCC) as a paper raw material, the pH standard is an important parameter. pH in the PCC synthesis process plays a role in controlling the size and morphology of the formed particles. pH conditions optimize nucleation and crystal growth resulting in PCC with characteristics suitable for paper applications. The optimum pH for the formation of PCC occurs at a pH of 10. According to the International Organization for Standardization, 3262-2:1998 standard within the pH range of 9–10, PCC on the requirements for paper raw materials. pH measurements with pH meter and best conditions using Na_2CO_3 2.5 M 40 minutes. Higher pH resulted in smaller PCC crystal sizes, making the product more suitable for industrial use.

Based on the XRF, the highest $CaCO_3$ content and pH standards is PCC with Na_2CO_3 2.5 M and a precipitation time of 40 minutes. To further confirm the crystal characteristics, an XRD test was conducted to determine the polymorphic phase and crystal size of PCC. XRD analysis provides information on atomic arrangement, symmetry, and mineral composition. In figure 4, the maximum diffraction peak is at 29.470°. with additional peaks at 30.1°, 39.49°, 48.55°, 57.49°, and 64.77°. These diffraction patterns confirm that the crystals formed are calcite. The XRD results also demonstrate that $CaCO_3$ is the dominant component, consistent with PCC standards for paper raw materials. This is PCC standards for paper raw materials.

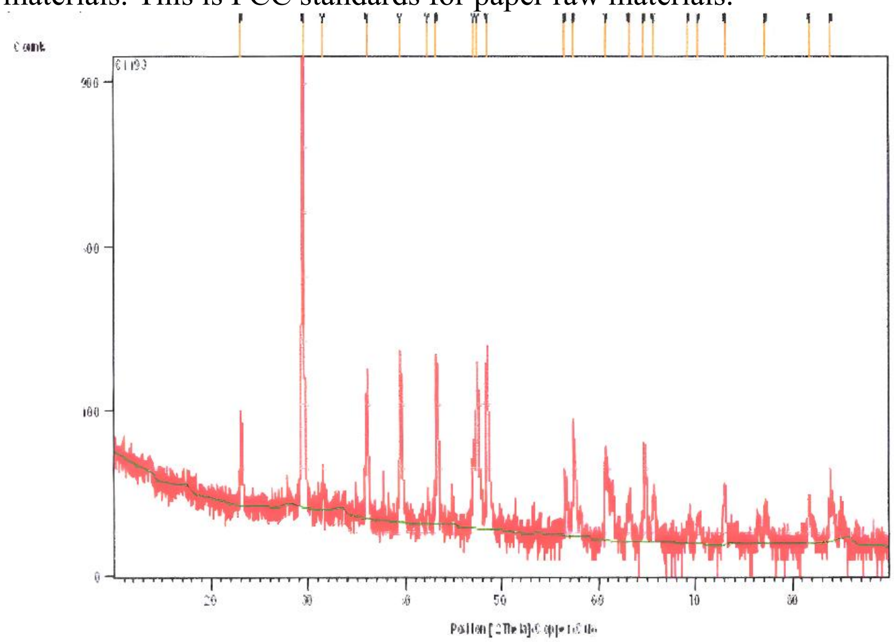

Figure 3. Pattern of PCC with Na_2CO_3 2.5 M and precipitation time of 40 minutes

Meanwhile, Figure 4 shows that each diffraction peak corresponds to same particle sizes in PCC synthesized with Na_2CO_3 2.5 M and a precipitation time of 40 minutes. Particle size analysis revealed that the resulting calcite polymorph had an average size of 2.14752 µm, which meets the ISO 3262-2:1998 standard for paper-grade PCC requiring 2 µm. In the paper industry, particle size strongly influences paper strength and flexibility, with smaller particles forming stronger interparticle bonds. The results indicate that PCC synthesized from chicken bones is suitable as a raw material for paper. The higher the concentration of Na_2CO_3 the smaller the particle size will be, but the $CaCO_3$ content will be higher (Wardhani, 2021, p. 5). The effect between precipitation time and particle size is inversely proportional. Longer precipitation times produce smaller particles, while faster precipitation times result in larger particles.

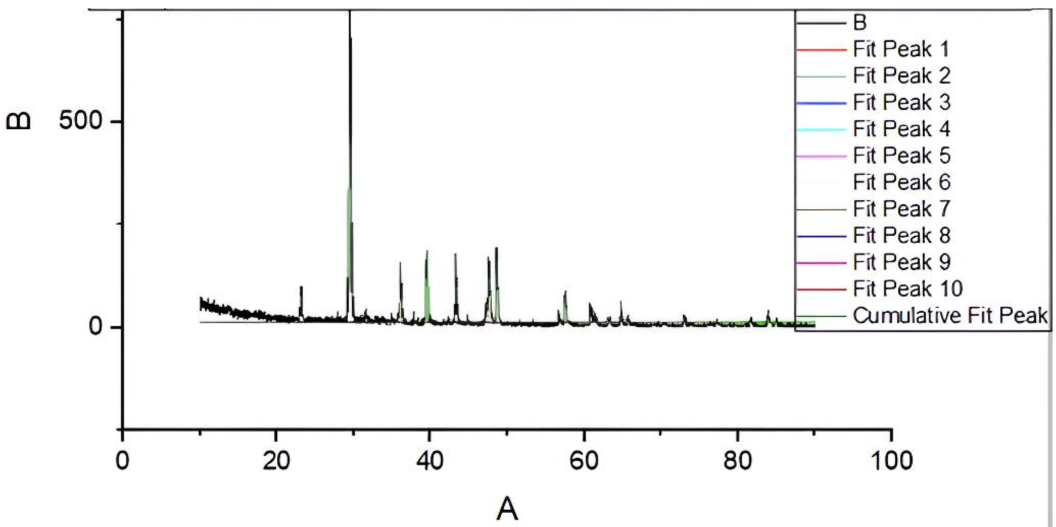

Figure 4. Data Peak Particle Size XRD Pattern PCC Na_2CO_3 Concentration 2.5 M 40 Minutes

CONCLUSION

In the research of synthesis of precipitated calcium carbonate (PCC) using chicken bones as a raw material for paper, the best results were obtained Na_2CO_3 2.5 M and a precipitation time of 40 minutes. It was found that the time and concentration of Na2CO3 affect the results of the PCC research. The precipitation time and Na_2CO_3 concentration are directly proportional to the weight of the obtained PCC. Based on the results of XRF and XRD tests, it was found that chicken bones can be used as raw material for producing PCC with papergrade standards because they are in the form of calcite polymorph with a particle size of 2.14752 µm.

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