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Synthesis of precipitated calcium carbonate with the addition of aloe vera extract under different reaction temperatures

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ABSTRACT

The synthesis of precipitated calcium carbonate (PCC) using plant extracts as additives has been developed to modify the morphology, particle size, chemical purity, and stability of PCC to be used for a variety of potential applications. In this study, aloe vera (*Aloe barbadensis Miller*) was used as an additive for the synthesis of PCC. The synthesis of PCC was carried out using a tubular column reactor under the temperatures of 30, 50 and 100°C and was performed without and with aloe vera extract concentrations of 3, 5, 8 and 10 vol%. Recycled carbon dioxide (CO_2) with a flow rate of 1 L/min was used which was obtained from the calcination process. The purity and morphology of synthesized PCC were analyzed using X-ray fluorescence and scanning electron microscope, respectively. The results demonstrated that the increase in the aloe vera concentration from 3 to 10 vol% produced the aragonite crystals at 100°C. The agglomerated cluster was observed with a rhombohedral shape with 3 and 5 vol% of aloe vera extract under the temperature of 30°C. The obtained PCC yield was 91, 72 and 93% at 30, 50 and 100°C, respectively with 10 vol% of aloe vera extract.

Keywords: Aloe vera extract, Aragonite crystal, Morphology, Precipitated calcium carbonate.

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1. INTRODUCTION

Precipitated calcium carbonate (PCC) is a pure form of calcium carbonate formed during a precipitation process in a carbonation reaction which is used as a filler or mixing agent in various industries. Various parameters, including particle size, specific surface area, chemical purity, morphology, and structure are considered before the application of PCC (Abeywardena et al., 2020; Akinola et al., 2022). Morphology and particle size have been emphasized to be considered for the specific application. Calcite and aragonite have caught the attention because of their thermodynamic stability and extensive potential applications (Gopi et al., 2013). Calcite PCC is identified by its rhombohedral crystal structure and is used as a filler for plastics to decrease energy and opacity and to increase the surface gloss in paint. Aragonite PC has a needle-like appearance and is widely used in the paper, rubber, and plastic industries as a filler/additive (Fairchild and Thatcher, 2000; Liu et al., 2016; Jimoh et al., 2017). Improving the quality of PCC can be done with the various modifications process including the addition of organic additives which can influence the precipitation conditions, CO_2 absorption and the shape of the crystal and size.

Several studies have successfully demonstrated that organic additives can improve the quality of nanoparticle products. The addition of saccharide solution in the PCC synthesis affected the rate of CO_2 absorption, which resulted in a change in precipitation rate and the polymorphic composition of calcium carbonate, with the highest concentration of vaterite (90%) was produced from a solution containing sucrose (Konopacka-Łyskawa et al., 2019). Green mussel shells were used as an additive in the synthesis of PCC owing to the high content of calcium. The green mussel shells demonstrated to be potentially recycled the PCC structure from aragonite into vaterite and calcite, which are technically possible for the starting materials in biomedical applications (Ismail et al., 2021). A previous study demonstrated that microalgae could produce cementitious $CaCO_3$ deposits by optimizing the cell growth, pH, and calcium and bicarbonate ions concentration (Arumugam et al., 2021). Plant extracts have been chosen to offer a pile of benefits of eco-friendliness and compatibility for medical applications due to less toxic chemical compounds. Plant extracts were found to influence the morphology of crystals due to their polarity and solubility (Lefebvre et al., 2021). However, the potential of plant extracts as additive materials for the synthesis of PCC is yet to be fully explored.

Another influencing factor that can improve the quality of PCC is temperature. The temperature can decrease the CO_2 solubility but affects the equilibrium of the carbonation system (Yuan et al., 2022). High temperatures can accelerate the higher $Ca(OH)_2$ dissolution and crystallization rates which can affect the final $CaCO_3$ solubility product (Liendo et al., 2022). Furthermore, the rate of nucleation, growth, and agglomeration could be affected by varying temperatures (Liu et al., 2018). The previous finding obtained rhombohedral sharp-edged particles at 45°C while the presence of scalenohedral particles was observed by decreasing the temperature to 25°C (Domingo et al., 2006). Increasing the temperature above 40°C may possibly obtain a mixture of aragonite and calcite without any additive (Santos et al., 2012). Thus, varying temperatures are essential to meet the final PCC product required for specific industrial applications.

The present work demonstrates the synthesis of PCC using aloe vera (*Aloe barbadensis Miller*) as an additive to improve the quality of PCC by adding aloe vera into the reactor. Aloe vera is a cactus-like plant found mainly in a dry and hot climate and contains aloe gel from the parenchyma cells of the plants and has been used for cosmetics and pharmaceutical applications. Aloe vera contains soluble proteins, such as glycine, aspartic acid, and glutamic acid that can form calcium-protein complexes in situ when the protein bonds with Ca^{2+} (Ni et al., 2004; Jimoh et al., 2017). Thus, the use of aloe vera extract can assist to tailor PCC morphology and particle size for the development of a clean PCC production process. However, the application of aloe vera to the synthesis of PCC is still limited and not fully explored. The synthesis of PCC was performed using a tubular reactor with a CO_2 bubbling system with different temperatures (30, 50 and 100°C). The recycled CO_2 was used for the carbonation reaction which was obtained from the calcination process. The effect of aloe vera concentration on the yield and morphology of PCC was investigated.

2. MATERIALS AND METHODS

2.1 Quicklime Used for the Synthesis of PCC

The quicklime for the synthesis of PCC was obtained from the calcination process in a limestone processing system located in Padang Panjang, West Sumatra. The conventional limestone processing system consists of 20 units of calcination chamber. The quicklime after the calcination process was collected and transferred to the laboratory for further investigation. The characteristics of quicklime used in this study are shown in Table 1.

Table 1. Characteristics of quicklime after the calcination process

Components	Unit (%)
CaO	50
Al_2O_3	1.73
Fe_2O_3	0.56
MgO	7.68
SiO_2	2.68
SrO	0.03

2.2 Preparation of Aloe Vera Extract

The collected aloe vera leaves were washed with distilled water and cut into small pieces. 300 grams of aloe vera was boiled with 1000 mL distilled water and filtered through Whatman filter paper and was distinguished at different concentrations of 3, 5, 8 and 10 vol%. The aloe vera extract was stored at 5°C for further experiments.

2.3 Synthesis of PCC

Quicklime was dissolved in water and mixed without and with aloe vera extract of 3, 8, 5 and 10 vol% and stirred using a magnetic stirrer at 300 rpm for 1 h. The obtained slurry was pumped using a peristaltic pump to the tubular reactor under different temperatures of 30, 50 and 100°C. A tubular column reactor with a height-to-diameter ratio of 50 cm/ 5 cm was used for PCC production in a carbonation process system as shown in Fig. 1. The CO_2 gas was supplied and flowed into the near bottom of the reactor with the flow rate of 1 L/min to create turbulence and shearing motion of the suspension. The reaction from the entire system is shown in Equations (1) and (2). The precipitation is considered complete when the PCC suspension reaches near the top of the column. The obtained PCC after carbonation was collected and analyzed for its properties.

$$\mathrm{CaO}_{(s)} + \mathrm{H_2O}_{(l)} \rightarrow \mathrm{Ca(OH)_2}_{(aq)} \quad \Delta H = -65.8\ \mathrm{kJ/mol} \quad (1)$$

$$Ca(OH)_{2(aq)} + CO_{2(g)} \rightarrow CaCO_{3(s)} \quad \Delta H = -112.5 \text{ kJ/mol} \tag{2}$$

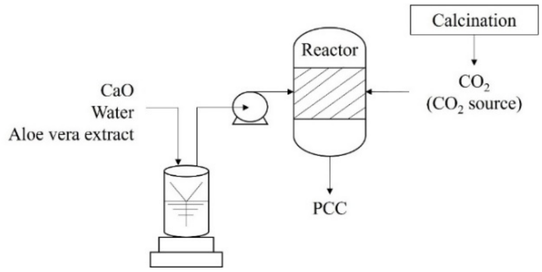

Fig. 1. Experimental setup for the synthesis of PCC with the addition of aloe vera extract in a tubular reactor under different temperatures (30, 50 and 100°C).

2.4 Characterization of PCC

The initial limestone and dried PCC were analyzed for the chemical components using an LR 39487 C X-ray fluorescence (XRF) analysis (PANanalytical, The Netherlands). A Quanta 450 FEG scanning electron microscope (SEM) (FEI, Czech Republic) was used to observe the morphology of the PCC products.

3. RESULTS AND DISCUSSION

3.1 Effect of Aloe Vera Extract Addition on the PCC Yield

The yield of PCC was lower at 30°C compared to those at 50 and 100°C without and with aloe vera extract of 3, 5 and 8 vol% which may ascribe to unready CO_2 diffusion and the low solubility of $Ca(OH)_2$ (Fig. 2). A relatively high PCC yield of 93% was obtained at 100°C with 10 vol% of aloe vera extract. High temperature can accelerate the solubility of $Ca(OH)_2$ resulting in more CO_2 dissolved into the mixture solution and more calcium ion dissociation from Ca $(OH)_2$ to be involved in the carbonation process (Bo et al., 2019; Li et al., 2022). Regardless of the different reaction temperatures, the aloe vera extract concentration did not give a significant change to the PCC yield, especially at 100°C. However, the PCC yield increased when the aloe vera extract increased to 10 vol% at 30°C (81.8 to 91.7%), confirming the XRF analysis detected high purity of the synthesized carbonate products. The marginal increase of PCC yield was observed from 3 to 10 vol% aloe vera extract concentration at 30°C as shown in Fig. 2. This could be due to the formation of calcium-protein complexes in situ which is induced by the uniform dispersion of Ca $(OH)_2$–CO_2 solution containing soluble proteins, thus increasing bicarbonate ion formation. Another mechanism was probably the protein content in aloe vera could serve as an intermediator between aloe vera and mineral ions for promoting the nucleation of $CaCO_3$, as the protein content released in the reactor of 30°C was not degraded.

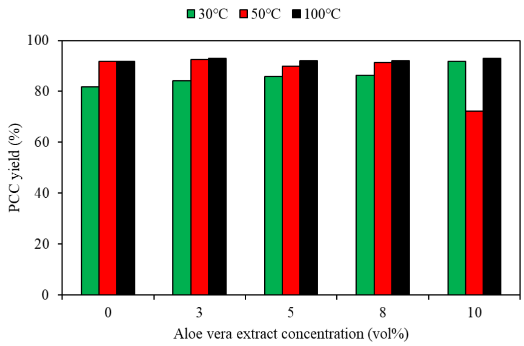

Fig. 2. The yield of PCC obtained after carbonation reaction without and with the addition of aloe vera extract concentrations of 3, 5, 8 and 10 vol% under different temperatures of 30, 50 and 100°C.

Table 2. XRF analysis of PCC

Temperature (°C)	Aloe vera extract concentration (vol%)	PCC (%)					
		Al_2O_3	SiO_2	MgO	Fe_2O_3	SrO	Cl
30	0	2.09	2.51	9.4	0.33	0.02	0.07
	3	1.72	2.34	8.39	0.32	0.03	0.03
	5	2.38	3.23	11.1	0.38	0.03	< 0.02
	8	2.32	2.85	7.04	0.37	0.03	0.04
	10	0.57	2.41	3.01	0.41	0.03	0.03
50	0	1.88	2.59	2.32	0.39	< 0.02	< 0.02
	3	1.97	2.52	1.64	0.33	0	< 0.02
	5	2.15	2.94	3.71	0.42	0.02	0
	8	2.18	2.92	2.34	0.39	0.03	< 0.02
	10	2	2.62	3.23	0.32	0.02	0
100	0	< 0.02	2.68	4.17	0.36	0.03	0.04
	3	< 0.02	2.68	2.94	0.37	0.03	0.03
	5	1.74	2.44	2.55	0.34	0.03	0.03
	8	< 0.02	2.57	4.24	0.34	0.03	0.02
	10	< 0.02	2.47	3.23	0.36	0.03	0.01

As the aloe vera extract concentration showed a low or no change in the PCC yield at 100°C, a small amount in the PCC yield was observed when the solution was added with 10 vol% of aloe vera extract at 50°C (increased from 50% before carbonation). The result demonstrated that only a small CO_2 dissolved into the aloe vera extract in the mixture solution. In addition, the formation rate of bicarbonate was probably lower than the rate of calcium–protein complex decomposition due to low binding energy between Ca^{2+} and protein (Jimoh et al., 2018). The adsorbed CO_2 gas was easily transformed from carbonic acid to bicarbonate ions at high temperatures and the soluble protein in aloe vera extract can easily form calcium–protein complex in situ due to probably high Ca^{2+} dissociation rate (Ham et al., 2005; Jimoh et al., 2018), resulting in the high conversion of CaO into $CaCO_3$ at 100°C. Overall, the recycled CO_2 used in this study can favorably increase the purity of PCC with the aid of temperature. The previous study revealed that recycled CO_2 with a capacity of 1 kg CO_2/h can optimize mineral carbonation with a calcium utilization ratio of 74.8% (Yu et al., 2020).

The chemical compositions of PCC generally increased when the aloe vera concentration increased to 5 vol% at all temperature conditions, especially MgO. The high percentage of MgO at 30°C suggested that the addition of aloe vera extract could increase the decomposition rate of MgO at low temperatures (increased 1.5 times from the initial percentage). It is noted that a high residue of MgO from the decomposition process during the carbonation reactions occurred during the low-temperature reaction (Highfield et al., 2016; McQueen et al., 2020). In addition, the soluble protein in the aloe vera extract could promote dehydrogenation processes in the alkaline electrolyte, this behavior can increase and decrease the oxides of alkali and alkaline during the carbonation reactions (Shen et al., 2021). The more porous carbonized particle, the better adsorption of the oxides in the surface layer of samples and, accordingly, high detection of Ca, Mg, Al, and Si was observed in the samples with the addition of 5 and 8 vol% of aloe vera extract at 30 and 50°C. The increase in temperature to 100°C may decrease the oxides elements under all conditions. This suggested that the high temperature promoted high solubility of Ca^{2+} and more CO_2 was adsorbed by the dissolved Ca^{2+} to form $CaCO_3$.

3.2 Effect of the Temperature on the Morphology of PCC without Aloe Vera Extract

Morphology of PCC without aloe vera extract was observed using SEM and is shown in Fig. 3. During the carbonation process, aggregation will be performed, and calcium particles will be agglomerated into different crystal shapes under different temperatures (Song et al., 2020). In this study, the rhombohedral shape was formed under the temperature of 30°C with the fiber-like shape connecting between the crystal and became solid with the increase in the temperature to 50°C with uniform particle size. During the synthesis of PCC, the formation of aragonite crystals required a higher temperature above the ambient temperature, as well as high energy demand (Ševčík et al., 2015; Kim et al., 2017). In this study, the aragonite shape started to form under the temperature of 100°C. The previous study also demonstrated that aragonite is formed above 50°C (Santos et al., 2012). Carbonation at temperatures above 50°C makes it possible to obtain pure aragonite under mechanical stirring without any additives (Santos et al., 2012). High temperatures can create thermal vibrations when Ca^{2+} ions are bonded with the O atom (Chen et al., 2013). In addition, the presence of thermal vibration affects the increase in surface energy and the stronger Brown motion (a movement caused by liquid or gas particles that spread continuously) which form aragonite crystals (Perdikouri et al., 2011). The results showed that rhombohedral crystals can transform into aragonite at temperatures above 50°C.

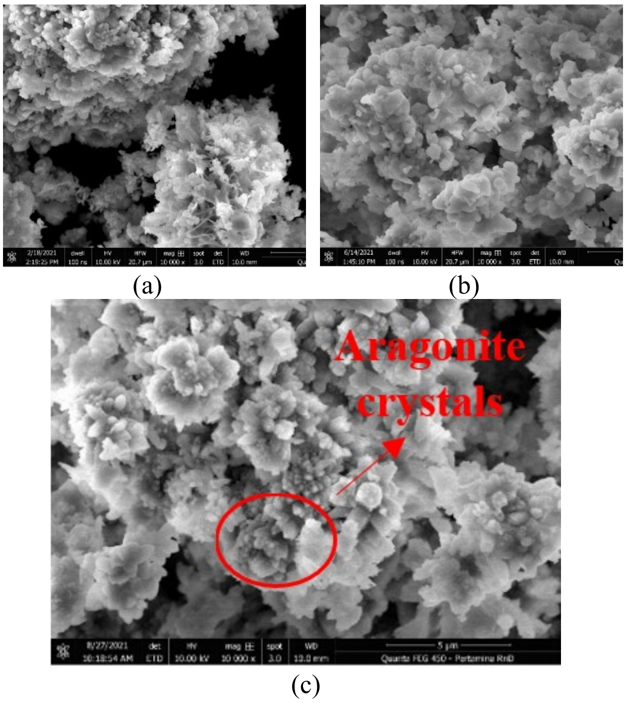

Fig. 3. SEM images at 10,000 x magnifications of synthesized PCC without aloe vera extract and under the reaction temperatures of (a) 30°C, (b) 50°C and (c) 100°C.

3.3 Effect of the Temperature on the Morphology of PCC with Aloe Vera Extract

The SEM images of the synthesized PCC at 30°C show different particles with various concentrations of aloe extracts (3, 5, 8 and 10 vol%) (Fig. 4). The agglomerated cluster was observed with a rhombohedral shape with 3 and 5 vol% of aloe vera extract under the temperature of 30°C. Aragonite crystals started to form under the temperature of 50°C, and the reduction in the particle size of aragonite crystals was clearly observed when the temperature was increased to 100°C. The cube-like calcite was formed at the temperature of the aloe vera extract of 8 and 10 vol%. The cubic-like calcite started to change into a flower-like shape when the temperature was increased to 100°C for the aloe extract of 8 vol%. However, the aragonite crystal began to form in the agglomerated cluster and continue to form when the temperature was increased to 100°C for the aloe vera extract of 10 vol%. This study demonstrated that by increasing the aloe vera extract concentration to 8 vol%, the calcite PCC particle was formed, and by increasing the temperature to 100°C, aragonite crystal increased for all aloe vera extract except for 8 vol%. It is demonstrated that the temperature has a decisive effect on the nucleation rate, the growing grain, and the final grain morphology (Abidi et al., 2022). This finding is concordant with Hariharan et al. (2014), by using N-acetyl glucosamine biopolymer and hydrogen ions can cause the phase transformation from calcite to aragonite.

The bonding effect of calcium-protein complex decomposition induced the soluble protein in the aloe vera extract to inhibit the calcite crystallization and caused the transition to aragonite crystals by increasing the aloe vera extract concentration. The increase in aloe vera extract concentration resulted in the increase of needle-like crystals of agglomerated $CaCO_3$ as well as the formation of the polycrystalline dumbbell-like structure (Greer et al., 2015; Jimoh et al., 2017). Fig. 4 obviously shows that the changes in the crystal structure from calcite to aragonite are an integrated effect of increasing the aloe vera extract concentration (from 8 to 10 vol%) and temperature (from 30°C to 100°C). However, the low concentration of aloe vera extract (3 and 5 vol%) did not give significant changes in the PCC structure, probably due to the low bonding energy between Ca^{2+} and protein.

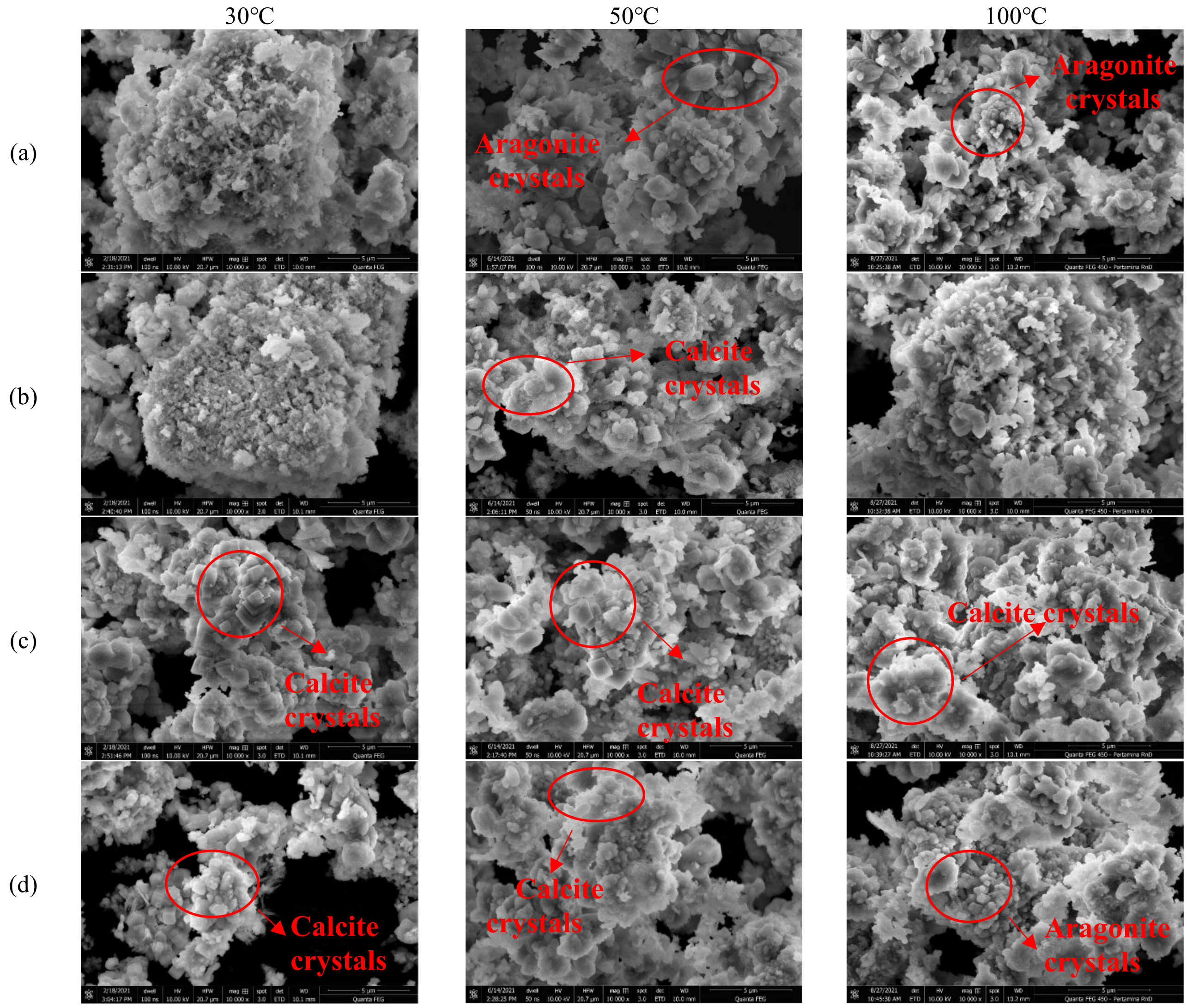

Fig. 4. SEM images at 10,000 x magnifications of synthesized PCC with aloe vera concentration (vol%): (a) 3, (b) 5, (c) 8 and (d) 10 under the reaction temperatures of 30, 50 and 100°C.

In addition, the particle size of the synthesized PCC is getting more minor as the concentration of aloe vera extracts increases, especially under 30°C as well as in the increasing temperature to 100°C. The reduction in particle size confirms the addition of aloe vera and temperatures suggest the possible versatility of the synthesized PCC for a variety of applications (Tippayawat et al., 2016). PCC products with small particle sizes can be used as osteoporosis treatment to increase bone density because the small particle size will increase calcium absorption in the body (Sunyecz et al., 2008). The synthesis of PCC using aloe vera extract resulted in the variation of morphological shapes and promoted the stability of synthesized PCC for specific applications from natural materials that can produce aragonite crystal types.

4. CONCLUSIONS

PCC with aragonite crystals was synthesized by adding the aloe vera extract under different temperature reactions. Different morphology of PCC was observed under different temperatures without the addition of aloe vera extract; rhombohedral, the mixture of calcite and aragonite, and aragonite crystals were observed at 30, 50 and 100°C. When varying the aloe vera extract from 3 to 10 vol%, the calcite crystals were formed at 30 and 50°C samples with an aloe extract concentration of 8 vol%. It is legitimate to conclude, that the morphology of the particles is dependent on the amount of extract and temperature that were used. A high yield of 93% was achieved when the sample was added by 10 vol% of aloe vera extract at 100°C, suggesting that the reactor had ensured efficient contact with reacting species to promote high purity of PCC. The synthesized PCC with the shape of aragonite is beneficial for numerous potential applications, such as fillers for paints, plastic, and papers. However, some limitations in this study appeared to challenge more information on the effect of temperature and additives to produce high purity of PCC. The information on the particle size, chemical stability, and pore size is required to provide the efficiency of aloe vera and the temperature used in this study.

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