

ADSORPTION OF HEAVY METALS USING BIO-CALCIUM CARBONATE DERIVED FROM A GOLDEN APPLE SNAIL (GAS) SHELL

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Abstract

In this research studied the use of calcium carbonate (CaCO_3) from golden apple snail (GAS) shells for application as an absorbent material to remove heavy metals in water sources, which replaces commercial calcium carbonate (CaCO_3) to reduce production costs and increase waste value. The adsorption of heavy metal contents (such as lead; Pb and Cadmium; Cd), phase formation, and physical characterization of golden apple snail shells were investigated for use as a calcium source in the production of naturally based biomaterials. The samples were calcined between 700°C and 950°C for 5 hr. TG and DTA analysis of samples demonstrated the decomposition of CaCO_3 to CaO . The XRD results demonstrated that natural shell powder has a crystal phase of CaCO_3 with an aragonite structure. Furthermore, the CaCO_3 (calcite phase) was transformed into calcium oxide (CaO) as a component, which showed that the phase transformation depended on the calcination temperature. The adsorption experiments showed good performance at about 99.6%, 99.7%, and 97% removal efficiency in a shorter time for calcined GAS at 700, 800, and 900°C , respectively. This study suggests that the golden apple snail shell could be an effective biomaterial for heavy removal from contaminated water.

Keywords: Adsorbent; Calcium carbonate; Heavy metal ions; Microstructure

Introduction

Golden apple snail (GAS) or *Pomacea canaliculata* (Lamarck) is a mollusk native to tropical and subtropical South America (Halwart, 1994; Naylor, 1996; Ranamukhaarachchi and Wickramasinghe, 2006). It was introduced to Southeast Asian countries in 1980 with the expectation that it could be cultivated as a high-protein source for

domestic consumption and an export commodity for industrialized regions (Naylor, 1996; Ranamukhaarachchi and Wickramasinghe, 2006). Recently, there have been large populations in most of Asia. Invasive GAS has been known as one of the 100 worst invasive alien species by the Invasive Species Specialist Group of the World

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Conservation Union (Lowe *et al.*, 2000; Zhao *et al.*, 2016)

The GAS commonly colonizes rivers, canals, lakes, and rice fields. It can consume up to rice seeding leading to significant economic losses in wetland rice cultivation, threatening biodiversity and impacting human health (Thaewnon-Ngiw *et al.*, 2004; Djeddour *et al.*, 2021) Estimating economic losses in Thailand resulting from GAS is about three billion US dollars per year (Chaichana and Sumpun, 2014). The GAS has increased rapidly and has been spreading in the rice field. Numerous methods, such as chemical, mechanical and biological have been utilized to kill the snail. Subsequently, a large amount of waste GAS shells were directly discarded resulting in serious environmental problems. Therefore strategies to convert low-valued wastes into new materials and products have been getting more interest. The GAS shell is mainly composed of 98-99% calcium carbonate (CaCO_3), of which aragonite and calcite are the common forms, and 1-2% other oxides like SiO_2 , MgO , and SO_3 (Zakaria *et al.*, 2004; Awang-Hazmi *et al.*, 2007). CaCO_3 offers promising advantages and is employed in many fields, including its capability to be incorporated and replace bone tissues (Islam *et al.*, 2011;

Hoque *et al.*, 2013; Mahmood *et al.*, 2017), material to adsorb carbon dioxide (CO_2) (Li *et al.*, 2009; Ma and Teng, 2010), and bio-adsorbent to remove basic fuchsin dye (Flores-Cano *et al.*, 2013). CaCO_3 has been getting much attention as a bio-adsorbent. Comparatively, bio-adsorbents have more advantages than conventional approaches for removing heavy metal ions: chemical precipitation, ion exchange, and membrane method in terms of cheapness, ease of operation, and high removal efficiency (Ahmad and Kibret, 2013). It is also one effective adsorbent abundant in nature and available as different nano/microstructures and composites.

Based on waste-to-wealth exploration and the utilization of waste GAS shells to remove metal heavy in water provides an innovative idea in water treatment and an essential practical method to use bio-waste. Hence this research aims to develop a low-cost bio-adsorbent for heavy metal removal in water from the synthesis and characterization of CaCO_3 from waste GAS shells. The correlation between the crystal structure of GAS shells and the efficiency of the adsorption of heavy metals is also investigated.

Materials and Methods

CaCO_3 powder preparation from GAS shells

The GAS shells were collected from rice fields in Phitsanulok, Thailand. The sample was washed several times with deionized water to remove additional residues from their surface. Then the GAS shells were boiled with sodium hydroxide (NaOH) for 1 hour to remove any connective tissue and contaminated protein. After that, the cleaned shells were dried at 80 °C for 48 hrs. Subsequently, the dried GAS shells were subjected to crushing and grinding and passed through a stainless-steel sieve with 100 mesh (0.15 mm). Next, the GAS shell powder was calcined at 700, 750, 800, 850, 900, and 950°C in an air atmosphere with a heating rate of 5°C/min for 3 hrs as shown in Figure 1. The non-calcined and calcined powders were characterized by XRD, FESEM, and heavy metal absorption.

GAS powder characterization

1. Thermogravimetric analysis (TG) and differential thermal analysis (DTA)

For investigating the thermal decomposition of shells components during calcination the TG and DTA analysis were conducted on the none-calcined (natural) powder from the GAS shells using a thermal analyzer ((NETZSCH STA 449 F3 Jupiter, NETZSCH, Gerätebau GmbH, Germany).

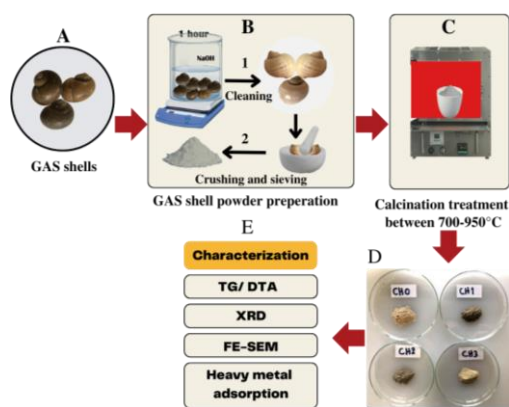


Figure 1. Schematic representation of the experimental approach: (A) The waste GAS shells were taken from rice fields (B) Schematic representation of GAS shell powder preparation; 1) cleaning for eliminating any connective tissue; 2) drying in the oven before crushing and grinding (C) Schematic representation of calcination treatment at 700, 750, 800, 850, 900, and 950°C with a heating rate of 5°C/min for 3 hrs (D) The calcined powder from the GAS shells after heating (E) The calcined powder was characterized by XRD, FESEM, and the heavy metal absorption

Approximately 10 mg samples were heated from 30 to 1100°C at a heating rate of 20°C/min under an N₂ atmosphere.

2. The X-ray diffraction (XRD) characterization

The crystal structure and phase of the non-calcined and calcined GAS powder were identified using the X-ray diffraction (XRD) measurement (XRD, Philip PW 3040/60 X'Pert Pro, Eindhoven, Netherlands) and fitted with the Rietveld refinement procedure employing the FullProf program. The diffraction patterns were recorded in the 2θ values in the region of 10° to 80°.

3. The surface morphology

The surface morphology of non-calcined and calcined GAS shell powder was analyzed through a field emission scanning electron microscope (FESEM) (FESEM Apreo S, Thermo Fisher Scientific Inc., Waltham, MA, USA). The sample was mounted on a metal stub using silver tape. The microscope was set at 2 kV in high-vacuum (HV) mode and 0.05 nA.

4. The heavy metal adsorption study

The heavy metal adsorption experiments were run in the 100-mL Erlenmeyer flasks containing the Cd²⁺ and Pb²⁺ solution at a concentration of 3 ppm for simulating heavy metals contained in wastewater. The Cd²⁺ and Pb²⁺ solution at a concentration of 3 ppm were investigated separately with 1 g of GAS powder for 100 min at room temperature with 200 rpm shaking condition. The mixture was filtered through filter paper and the heavy metal's solution was measured by Atomic Absorption Spectrophotometry (AAS) (Perkin Elmer: PinAAcle 900F AA Spectrometer, USA). The results were expressed as the removal efficiency (E) of the adsorbent on Cd and Pb, which was defined as:

$$\%E = \frac{(c_i - c_e)}{c_i} \times 100 \quad (1)$$

Where c_i and c_e are the initial and equilibrium concentration of Pb and Cd (mgL⁻¹), respectively (Dubey and Gopal, 2007; Dou *et al.*, 2022; Fernández Pérez, 2022).

5. Statistical analysis

Statistical analysis was carried out using OriginPro version 9 (OriginLab, Northampton, MA, USA). All the experiments were performed in triplicate wells for each condition and experimental errors were estimated and expressed as the mean±standard deviation.

Results and Discussion

Thermal behaviors of GAS shell

The thermogravimetric analysis (TG) and differential thermal analysis (DTA) of GAS shells with non-calcined samples are shown in Figure 2. TG and DTA analysis were carried out to determine the temperature at which CaCO₃ decomposes to CaO. Results showed the four stages of metamorphosis. In the first state, 1-2% of weight loss occurred below 200°C related to a slight change of exothermic peak, which is the evaporation of water (Halwart, 1994). In the second state, weight loss of about 2% is occurred around 200-600°C due to the decomposition of organic material (Mohamed *et al.*, 2012; Kamba *et al.*, 2013; Asmi and Zulfia, 2017). The third state occurred around 600-860°C with a weight loss of 42.95%, corresponding to the exothermic peak of about 824.0°C. This state indicated the decomposition of CaCO₃ to CaO (Mohamed *et al.*, 2012; Kamba *et al.*, 2013). In the last state, no weight loss above the temperature of 870°C. The TG and DTA result of the sample obtained from this study also agrees with the results found by previous research (Mohamed *et al.*, 2012; Laonapakula *et al.*, 2019; Vu *et al.*, 2019). Thus CaCO₃ might have decomposed to CaO at a temperature of 824°C.

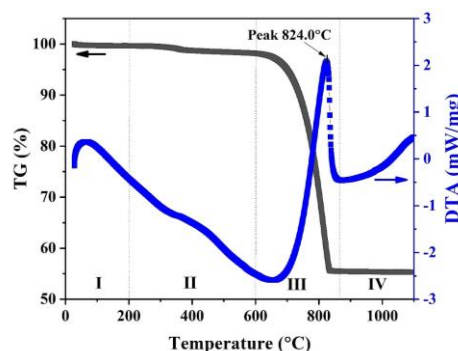


Figure 2. TG and DTA of GAS shells with non-calcined samples

XRD and Rietveld refinement result

The X-ray diffraction pattern of GAS shells with non-calcined and calcined between 700 and 950°C are shown in Figure 3. For the sample with non-calcined powder, the structure was matched with an aragonite structure and no other crystalline phases were detected. After calcination at 700°C, the structure transforms aragonite to calcite structure as a major property and presents a small peak of Ca(OH)₂ at the 2θ range of 28.5° and 34.0°. At 750°C, the triple phase of CaCO₃ with calcite structure, a small peak of Ca(OH)₂ and CaO at the

2θ range of 32.1° , 37.2° , 53.7° and 67.3° was observed. With calcination temperature increased from 800 to 950°C , the disappearance of the calcite peak and second phase of CaO and Ca(OH)_2 was presented. These indicated that the structure of GAS shells transforms aragonite (CaCO_3) to calcite after heating and then transforms into CaO at the high calcination temperature. This result was confirmed by the Rietveld refinement technique to analyze the phase ratio of the samples with the Fullprof program

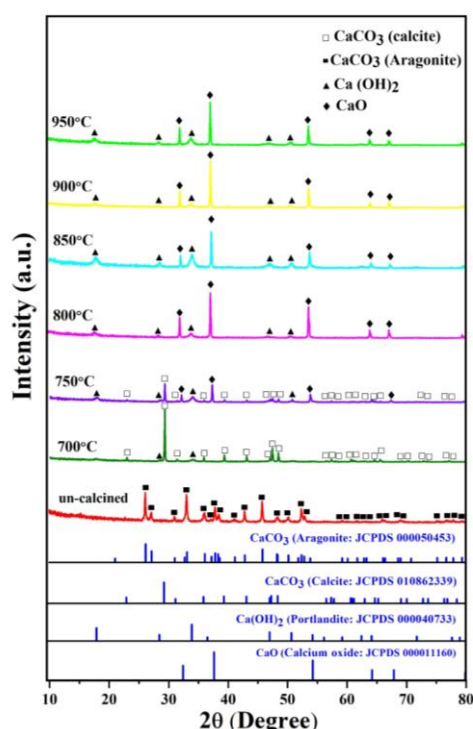


Figure 3. X-ray diffraction pattern of GAS shells with non-calcined and calcined between 700 and 950°C

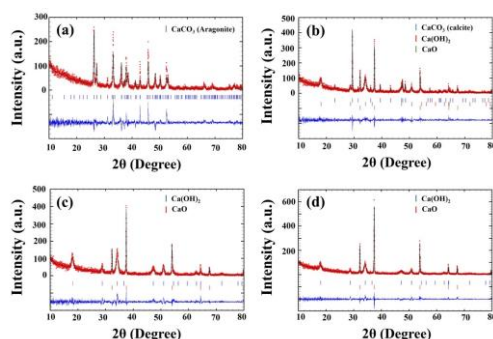


Figure 4. Fitted XRD patterns of the GAS shells for the sample (a) non-calcined and calcined at (b) 700°C , (c) 800°C and (d) 900°C by the Rietveld refinement technique

(Figure 4). The aragonite (CaCO_3) with space group P1, calcite (CaCO_3) with space group R3C, calcium hydroxide (Ca(OH)_2) with space group P3m1 and calcium oxide (CaO) with space group Fm3m were used as initial models for the refinement. In this result, for samples of non-calcined powder, the percentage of phase with aragonite (CaCO_3) is 100, as shown in Table 1. After calcination at 700°C , the phase of aragonite (CaCO_3) disappeared while calcite (CaCO_3) and Ca(OH)_2 occurred. The percentage of phase with calcite (CaCO_3) and Ca(OH)_2 are 91.10 and 8.90, respectively. At 750°C , the percentage of phase with CaCO_3 (calcite), Ca(OH)_2 , and CaO is 51.28, 21.08, and 27.64, respectively. When increased of calcination temperature from 800 to 950°C , the phase ratio between Ca(OH)_2 and CaO was found to be 10.61 and 89.39 for 800°C , 37.86 and 62.14 for 850°C , 13.71 and 86.29 for 900°C and 15.18 and 84.82 for 950°C , respectively, as seen in Table 1.

The surface morphology of GAS shells powder

The surface morphology of GAS shells at different calcination temperatures, as revealed by FE-SEM is presented in Figure 5. For non-calcined powder, the sample possessed small fine particles and plate shapes with parallel cleavages as seen in Figure 5(a). After calcined between 700 and 800°C (Figure 4(b) and (c)), the particle sizes exhibited agglomeration form as a matrix phase behavior in liquid phase formation which is attributed to the removal of water and heat dissolving the organic substance in the raw shell (Laonapakula *et al.*, 2019). The surface morphology of GAS powder at 900°C is illustrated in the split line which can be attributed to the structural transformation of calcium carbonate into calcium oxide (Figure 4(d)) (Mohamed and Maitra, 2012).

Adsorption Efficiency

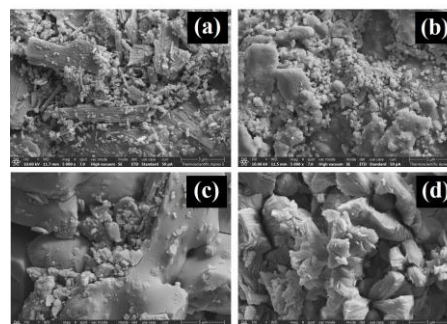
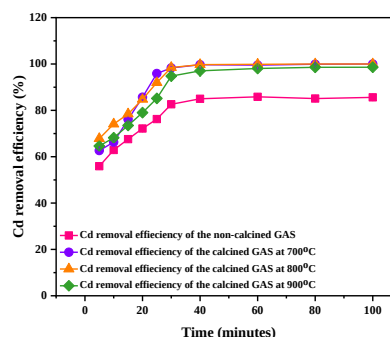
The effect of contact time on the Cd and Pb adsorption of GAS was carried out with varying contact times from 5 to 100 min at a concentration of 3 ppm. All heavy metal adsorption studies were conducted at room temperature with 200 rpm shaking conditions. As shown in Figure 6, in the beginning, the Cd adsorbed onto GAS significantly increased until almost 30 min. The Cd removal percentage increased from $55.9 \pm 0.8\%$ to $84.9 \pm 0.7\%$, $62.6 \pm 0.5\%$ to $99.6 \pm 0.5\%$, $67.8 \pm 0.9\%$ to $99.7 \pm 0.4\%$ and $64.6 \pm 1.2\%$ to $97.0 \pm 2.2\%$ for non-calcined GAS, calcined GAS at 700 , 800 , 900°C , respectively. With increasing the contact time, the Cd absorption increased slowly and remained stable after 30 minutes at all studied non-calcined and calcined temperatures. The trends are also in

Table 1. Rietveld structural refinement results of GAS shells with non-calcined and calcined between 700 and 950°C

Temp.	Goodness of fit	Profile parameters				The percentage of phase		
		CaCO ₃ (Aragonite) (P1)	CaCO ₃ (calcite) (R3C)	Ca(OH) ₂ (P3m1)	CaO (Fm3m)	CaCO ₃ (Aragonite) (P1)	CaCO ₃ (calcite) (R3C)	Ca(OH) ₂ (P3m1)
non-calcined	$\chi^2=1.68$ $R_p=60.9$ $R_{wp}=52.6$ $R_{exp}=40.58$	a=4.962 Å b=5.746 Å c=7.979 Å V=227.488 Å ³				100		
700	$\chi^2=2.11$ $R_p=62.7$ $R_{wp}=53.7$ $R_{exp}=32.92$		a=4.990 Å c=7.073 Å V=368.177 Å ³	a=4.994 Å c=17.080 Å V=368.885 Å ³	a=3.687 Å c=4.808 Å V=56.615 Å ³		91.10	
750	$\chi^2=1.31$ $R_p=58.8$ $R_{wp}=48.6$ $R_{ex}=42.51$		a=4.990 Å c=7.073 Å V=368.177 Å ³	a=3.594 Å c=4.918 Å V=55.014 Å ³	a=4.813 Å V=111.480 Å ³		8.90	
800	$\chi^2=1.35$ $R_p=67.6$ $R_{wp}=48.8$ $R_{exp}=42.01$			a=3.598 Å c=4.920 Å V=55.151 Å ³	a=4.818 Å V=111.840 Å ³			
850	$\chi^2=1.51$ $R_p=90.3$ $R_{wp}=54.0$ $R_{exp}=43.87$			a=3.585 Å c=4.908 Å V=54.62 Å ³	a=4.801 Å V=110.658 Å ³		51.28	21.08
900	$\chi^2=1.39$ $R_p=65.2$ $R_{wp}=47.5$ $R_{exp}=40.39$			a=3.587 Å c=4.905 Å V=54.709 Å ³	a=4.803 Å V=110.784 Å ³			
950	$\chi^2=1.31$ $R_p=75.2$ $R_{wp}=49.6$ $R_{exp}=43.33$			a=3.591 Å c=4.914 Å V=54.861 Å ³	a=4.809 Å V=111.238 Å ³			10.61
								89.39

agreement with another metal absorption study of the freshwater snail shell which showed that the metal absorption rate was lower when equilibrium was reached (Zhao *et al.*, 2016; Va *et al.*, 2019). Similar trend of Cd and Pb adsorption has also been presented previously for other shells such as the cockle shell (Panpho *et al.*, 2023) and Solamen Vaillant seashells (Esmaeili *et al.*, 2020).

Similarly, the removal percentage for Pb absorption increased with an increase in time before equilibrium was reached (Figure 7). It can be seen that as time increment (Figure 7), the Pb removal rate increased from 45.4±3.2% to 88.6±1.2%, 52.9±0.8% to 97.9±0.2%, 55.3±0.8% to 98.7±0.2% and 55.9±0.3% to 97.2±0.4% for non-calcined GAS, calcined GAS at 700, 800, and 900°C, respectively. The behaviors can be explained that all the active sites on the surface of the bio-sorbent are initially vacant due to an interactive force between solute molecules in the solid phase and bulk phase. With increasing contact time, the bonds between the active sites and the Pb and Cd increase and the adsorption process becomes less effective (Heidari *et al.*, 2013; Ahmadi and Esmaeili, 2018; Vu *et al.*, 2019; Esmaeili *et al.*, 2020). A comparison of the Cd and Pb removal efficiency between non-calcined and calcined GAS is shown in Figures 8 and 9, respectively. There were statistically significant differences for both Cd and Pb removal efficiency when comparing the non-calcined and calcined at different temperatures. Before calcination, the structure of GAS is an aragonite crystal. The GAS

**Figure 5. Surface morphology of GAS shells with (a) non-calcined and calcined at (b) 700°C, (c) 800°C and (d) 900°C****Figure 6. Effect of time on the removal of Cd on GAS from aqueous media using the GAS at various calcined temperatures (3 ppm of Cd; 1 g of GAS, mixture rate: 200 rpm)**

powder exhibited lamellar aragonite structures as shown in Figure 5(a) (Gopi *et al.*, 2013). While after heating, the structure transforms aragonite to calcite structure and almost all CaCO_3 was converted into CaO . The surface of the GAS powder became rough

and a large number of the split line appeared (Figure 5(d)). It can be explained that the phase changes at high temperatures because hydrogen bonds are broken. As a result water vapor unavoidably occurs and the porosity increases to release CO_2 (Irfan *et al.*, 2021). A porous surface structure can assist the progress of heavy metal ions (Papadopoulos and Rowell, 1988).

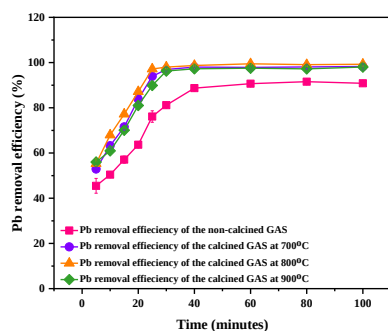


Figure 7. Effect of time on the removal of Pb on GAS from aqueous media using the GAS at various calcined temperatures (3 ppm of Pb; 1 g of GAS, mixture rate: 200 rpm)

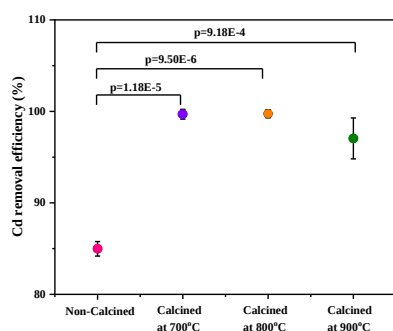


Figure 8. Cd removal efficiency (%) of non-calcined and calcined GAS at at 700, 800, and 900°C. Bars represent the mean values and error bars represent the standard deviation

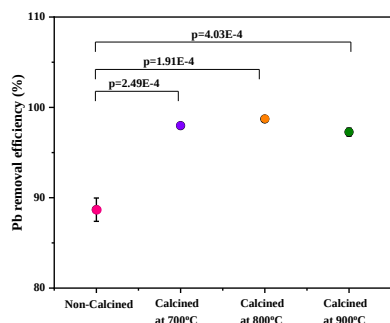


Figure 9. Pb removal efficiency (%) of non-calcined and calcined GAS at at 700, 800, and 900°C. Bars represent the mean values and error bars represent the standard deviation

Conclusions

This study synthesized and investigated CaCO_3 from waste GAS shells as a low-cost bio-adsorbent for heavy metal removal in wastewater. Based on XRD data, our work presents that the crystal structure of GAS before the calcination process is the aragonite phase for the first time. The crystal transformation from the original aragonite phase to CaO during the calcination process has influenced the porosity on the GAS shell surface. The scanning electron microscopy confirmed that the surface of the GAS shell became rough and split at calcination temperature. The rough and porous surface can assist the heavy metal ions' adsorption. The adsorption experiments showed good performance at about 99.6 %, 99.7% and 97% removal efficiency in a shorter time for calcined GAS at 700, 800, and 900°C, respectively. Our results can support that the CaCO_3 from GAS is effective and suitable for being used as a low-cost adsorbent to remove heavy metal ions in wastewater.

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