

## Use of Calcined Oyster Shell Powders as CO<sub>2</sub> Adsorbents in Algae-Containing Water

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### ABSTRACT

Here, we introduce a means of utilizing waste oyster shells which were obtained from temporary storage near coastal work-places as CO<sub>2</sub> adsorbents. The calcined CaO can be easily dissociated to Ca<sup>2+</sup> cation and CO<sub>3</sub><sup>2-</sup> anion by hydrolysis and gas-liquid carbonation reaction and converted to precipitated calcium carbonate (PCC) in algae-containing water. The calcium hydroxide and carbonation combination in algae-containing water significantly contributed to improving water quality which is very dependent on the addition amount of calcined powders.

**Key words :** Carbon dioxide, Adsorbent, Calcined oyster shell, Carbonation, Algae-containing water

### 1. Introduction

Greenhouse gas emissions have contributed to warming the Earth's surface and atmosphere which has significant implications on rainfall as well as the retreat of glaciers and sea ice. Especially, the increase in tropospheric ozone from air pollution (NO<sub>x</sub>, CO, and others) is an important greenhouse contributor.<sup>1,2)</sup> Among the greenhouse gases, CO<sub>2</sub> contributes more than 60% to global warming due to its huge emission amount. Currently, CO<sub>2</sub> concentrations in the atmosphere are approaching a level higher than 400 ppm, compared with 300 ppm prior to industrial revolution.<sup>3)</sup> For the first time in the past 20 years, more than 190 countries participated in the Paris climate conference (COP21, 2015) where an agreement was made to limit global warming below 2°C.<sup>4)</sup>

Though various CO<sub>2</sub> capture technologies have been proposed, chemical adsorption is currently believed to be the most suitable one for post-combustion power plants.<sup>5,8)</sup> Many porous materials have been developed for the high-efficiency adsorption of various gases in the atmosphere.<sup>9)</sup> Porous granular ceramic containing metal oxides can use as a fluoride adsorbent in aqueous solution.<sup>10)</sup> Waste oyster shells can be recycled as an adsorbent for atmosphere-polluting gas removal such as sulfur dioxide/nitrogen oxide (SO<sub>2</sub>/NO<sub>x</sub>).<sup>11)</sup>

In particular, as shown in Table 1, disposal amounts of the oyster shell waste have shown a steady increase at a scale of 50 ~ 100 thousand tons per year due to high seafood consumption, reaching an amount in excess of 660,000 tons in 2008.<sup>12,13)</sup> As shown in Fig. 1, the land filling of solid wastes (i.e., oyster shell waste) paralyzes many useful areas on earth and pollutes the surrounding environment by releasing hazardous gases. Therefore, the capability of oyster shell waste to be a replacement resource for limestone is an ideal breakthrough to solve the by-products treatment problems.

In this study, we attempted a carbonation process on the hydrated formation of calcined oyster shell powders as a way to mitigate discharge of global climate changing CO<sub>2</sub> in

**Table 1.** Annual Report of Oyster Production Amounts and Estimated Oyster Shell Disposal Amount

Year	Oyster production amount (ton)	Estimated disposal amount of oyster shell (ton)
2000	15,939	239,085
2001	10,056	150,840
2002	7,950	119,250
2003	20,201	303,015
2004	25,690	385,350
2005	27,320	409,800
2006	31,016	465,240
2007	34,240	513,600
2008	44,058	660,870

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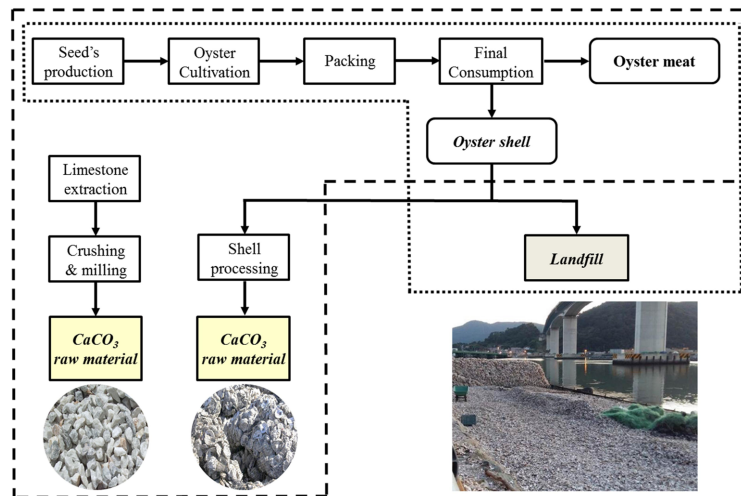


Fig. 1. Two scenarios for treatment of oyster shell waste: replacement resource development and landfill.

green algae-containing water. The  $\text{CO}_2$  adsorbing performance to precipitated calcium carbonate (PCC) by dissolving or injecting  $\text{CO}_2$  in the algae-containing water and the changes of morphology, crystallinity, and chemical composition will be discussed. Simultaneously, we also demonstrate that the hydrate and PCC converted from calcined oyster shell powders plays a key role in improving the quality of the algae-containing water.

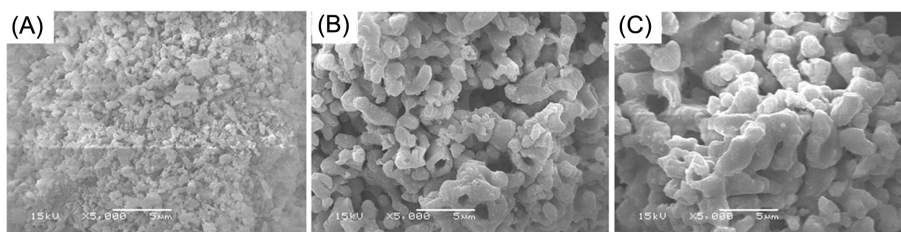
## 2. Experimental Procedure

Oyster shell waste was obtained from temporary storage near the seashore of Gaduk-do in Geoje province, and washed with alcohol and water to remove impurities attached to the surfaces. The shells were dried in a drying oven, crushed using a jawbreaker machine, and ground to fine powder of less than  $100\ \mu\text{m}$  by physical sieving separation to obtain reproducible experimental results. The calcined powder was obtained in ambient laboratory conditions at  $1000^\circ\text{C}$  (or  $800^\circ\text{C}$ ) for 1 h at heating rate of  $10^\circ\text{C}/\text{min}$  inside an electric furnace. The calcined powder was cooled to room temperature and stored in a closed container. The algae-containing water used in the experiments was collected by direct pumping from Daecheong Lake (a lake-type artificial reservoir located in Okcheon Buso-ri, Busodamak) at the end of October, 2015. One gram or two grams of the calcined powders from the heat-treatment at  $1000^\circ\text{C}$  for 1 h were added in 200 mL of the algae-containing water. Visible changes in water clarity and light penetration from initial green algae-containing water were observed after undergoing the processes of agitation (2 min), coagulation (5 min),  $\text{CO}_2$  injection (flow rate of  $0.5\ \text{mL}/\text{min}$ , 5 min), and precipitation (5 min). All of the samples were separated to solid precipitations and solutions by vacuum filtration using a funnel and an aspirator. All of solid samples containing filtered precipitates were dried in a convection oven for 24 h to observe the following surface measurements. The raw

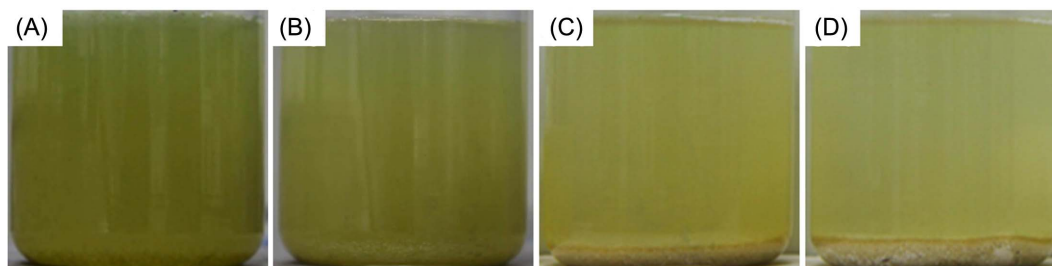
algae-containing solution and five filtered solutions were used for a comparison of the water quality analysis. For the surface morphological and elemental analyses of waste oyster shell surfaces which are converted to the comminuted, calcined, hydrated, and precipitated powders, scanning electron microscopy equipped with energy dispersive x-ray spectroscopy (SEM-EDS, JEOL Co.) was employed and a 10 nm Au coating was conducted to provide surface conductivity on each of the powders. Fourier transform infrared spectroscopy (FT-IR) was performed in the mid infrared region of  $650 - 4000\ \text{cm}^{-1}$  and identified solid structures of the calcined and the hydrated formations and the PCC formation of the oyster shell powder, respectively. X-ray diffraction (XRD) and X-ray fluorescence (XRF) measurements were used to recognize crystallinities and chemical compositions of calcined, hydrated and carbonated formation of the oyster shell powder. All water quality analyses were conducted by well-known conventional methods such as T-N (chromotropic acid method, analyzed wavelength of 410 nm, analysis limit of  $1.0 \sim 50\ \text{mg}/\text{L}$ ), T-P (ascorbic acid method, analyzed wavelength of 880nm, analysis limit of  $0.01 \sim \text{mg}/\text{L}$ ), and COD (reactor digestion method, analyzed wavelength of 540 nm, analysis limit of  $0.6 \sim 20\ \text{mg}/\text{L}$ ) in combination of a UV-Visible spectrophotometer (Humas Co. LTD, HS-3300). In measuring the removal efficiency for algal blooms, each solution was diluted five times to avoid the effects of absorption wavelengths on the green color of the algal solution.

## 3. Results and Discussion

SEM images in Fig. 2 are shown with distinct morphological differences of comminuted (Fig. 2(a)), calcined (Fig. 2(b),  $800^\circ\text{C}$ ) and another calcined (Fig. 2(c),  $1,000^\circ\text{C}$ ) powder of oyster shell waste. The comminuted powder of oyster shell waste had a surface structure of densely attached fine grains in  $1 - 2\ \mu\text{m}$  size. After undergoing the calcination pro-



**Fig. 2.** SEM images of (a) raw comminuted powder and calcined oyster shell powders at temperature of (b) 800°C and (c) 1000°C.



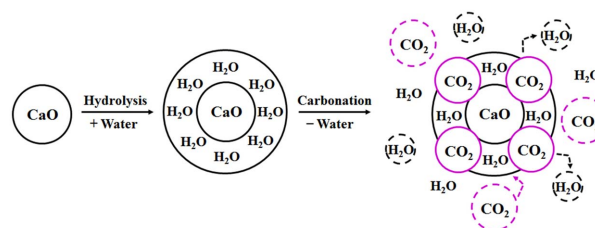
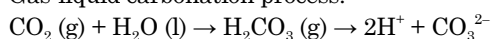
**Fig. 3.** Visible color change by oyster shell powders as CO<sub>2</sub> adsorbent in 200 ml volume algae-containing water: (a) w/o treatment and, (b) 1 g hydrated powder, and (c) 1 g carbonated and (d) 2 g carbonated powder.

cess at 800 °C, it was changed to a structure of connected peanut-shaped grains (width 1 - 1.5 μm /length 3 - 4 μm) and after the continuous release of CO<sub>2</sub> in the calcination process up to 1,000°C, it was changed to a structure of connected larger peanut-shaped grains (width 2 ~ 2.5 μm/ length 3 - 4 μm) compared with those treated at 800°C.

A visible color change shown in Fig. 3 makes it possible to compare the algae-containing water (Fig. 3(a)), the treated water with a hydrated form converted from 1 gram calcined powder (Fig. 3(b)), and the treated water with a PCC converted from 1 gram and 2 gram calcined powders (Figs. 3(c) and 3(d)), respectively. Although a color change and an improvement in water clarity and light penetration of the treated water with a hydrated form was observed, remarkable increases were accomplished after undergoing the carbonation reaction to the PCC powder. When the amounts were increased from 1 gram to 2 grams per equivalent 0.2 L of the algae-containing water, the color distinction from a green to pale green color through a gradual improvement in water clarity and light penetration was demonstrated.

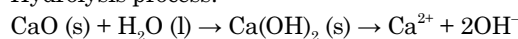
In the algae-containing water at ambient room temperature, the gas-solid carbonation reaction was schematically illustrated in Fig. 4. The carbonation reaction involves gas, liquid, and solid phases; Ca(OH)<sub>2</sub> has a finite solubility in water and the reaction occurs between dissolved CO<sub>2</sub> and OH<sup>-</sup> ions. The calcined powder (CaO) was converted to calcium hydroxide (Ca(OH)<sub>2</sub>) by the hydrolysis reaction of neighboring water molecules and then gradually converted to PCC (CaCO<sub>3</sub>) by carbonation reaction of CO<sub>2</sub> injection. The overall process of the hydrolysis and carbonation of lime consists of the following three steps.<sup>14-17)</sup>

Gas-liquid carbonation process:

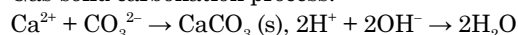


**Fig. 4.** Schematics of the carbonation process of hydrated oyster shell powders; CaO → Ca(OH)<sub>2</sub> → partial CaCO<sub>3</sub>.

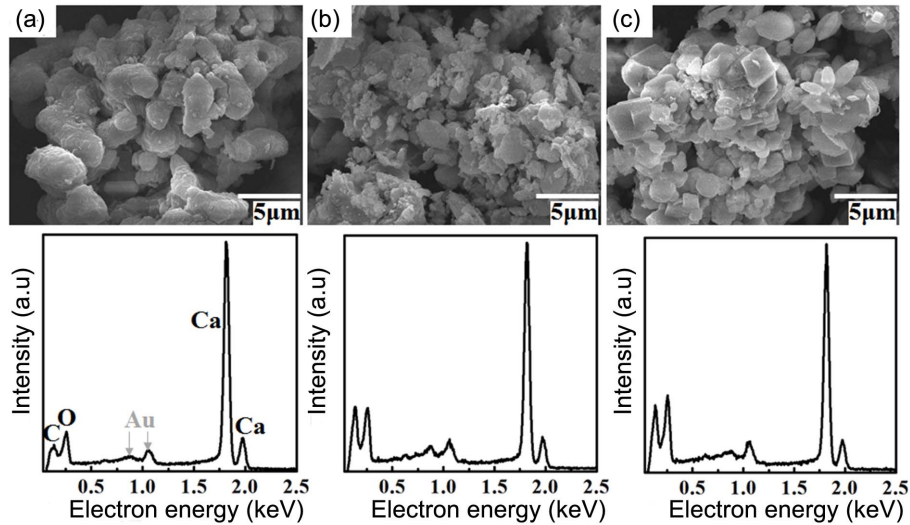
Hydrolysis process:



Gas-solid carbonation process:



The SEM images in Fig. 5 focus on the transformation to the morphology and microstructure of PCC during the carbonation of calcium hydrate. The hydrated morphology (Fig. 5(a)) appears in a peanut shape with larger volumes than that of calcined powder due to clustering between neighboring water molecules in the algae-containing water. Due to CO<sub>2</sub> dissolved in the water, the intensity of carbon (C) and oxygen (O) was stronger than for the calcined powder. Carbonation process (Fig. 5(b) and 5(c)) on the hydrated powder led to clear transformation to PCC with small angular grains in all of 1 gram and 2 gram precipitates, respectively. The intensity increase of C and O element can be reasonably correlated with decreasing Ca(OH)<sub>2</sub> particles and increasing crystal featured CaCO<sub>3</sub> particles. The increase of hydroxide dissolution amounts led to slightly higher crystallinity in calcite PCC. However, the excess of hydroxide dissolution through the continuous addition of calcined powder reacti-



**Fig. 5.** SEM/EDS analysis of (a) 1 g hydrated, and (b) 1 g carbonated and (c) 2 g carbonated surfaces of calcined oyster shell powders; all of the samples were placed in a drying oven for 1 day at 80°C to remove the moisture.

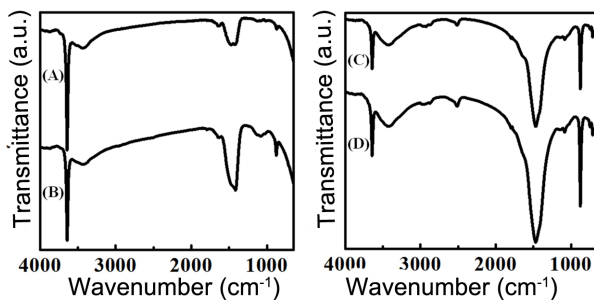
hydrated hydrate reaction after completely finishing carbonation reaction under constant amount of CO<sub>2</sub> gas injection.<sup>18)</sup>

In Fig. 6(a) - (d), FT-IR analyses show the characteristic bands of calcined, hydrated, and precipitation formation (1 gram and 2 gram) converted from waste oyster powders, respectively. In Table 2, the characteristic absorption peaks at the wavenumbers of functional groups were assigned to stretching and bending vibration peaks of hydrated and carbonated formation. The strong O-H stretching band at ~3,643 cm<sup>-1</sup> and the weak H-O-H bending vibration at ~1,418 cm<sup>-1</sup> identify the presence of bulk OH groups of Ca(OH)<sub>2</sub>.<sup>19)</sup> The strong band at ~1,433 cm<sup>-1</sup> (asymmetric stretch) and the weak band at ~2,518 cm<sup>-1</sup> correspond to CO<sub>3</sub><sup>2-</sup> ions. The bands of calcite structure of CaCO<sub>3</sub> (the comminuted shell powders) show the 711 cm<sup>-1</sup> (in-plane bending) and 873 cm<sup>-1</sup> (O-C-O out-of-plane bending). Interestingly, the hydrated precipitate in algae-containing water is observed with partial carbonation bands subjected to CO<sub>2</sub> contents dissolved from the atmosphere

condition. The representative feature of carbonated precipitates shows that the relative intensity of the O-H stretching band at ~3,640 cm<sup>-1</sup> of precipitation was decreased and the relative intensity at ~1,433 cm<sup>-1</sup> of CO<sub>3</sub><sup>2-</sup> ion of precipitation was increased.<sup>20,21)</sup>

Crystalline XRD patterns of CaO, Ca(OH)<sub>2</sub> and CaCO<sub>3</sub> reagents and the two precipitation powders are shown in Fig. 7 (a) and (b). Marked major peaks on each powder correspond to characteristic diffraction peaks of CaO at 2θ = 32, 37.2 and 53.7, Ca(OH)<sub>2</sub> at 2θ = 17.9, 34.1, and CaCO<sub>3</sub> at 2θ = 29.4. In agreement with FTIR analyses, all of precipitated powders are composed of a combination of the hydrated and carbonated formation, indicating that Ca(OH)<sub>2</sub> tends to be carbonized to CaCO<sub>3</sub> according to increase of addition amounts.

The chemical components of five samples converted from waste oyster powders with comminuted, calcined(800°C, 1000°C), and precipitation formations (1 gram, 2 gram) were shown in Table 3(a) - (f), respectively. The XRF result shows the major components of the waste oyster shell pow-



**Fig. 6.** FT-IR spectra of hydrated and the carbonated phases of calcined oyster shell powders (1,000°C, 1 h): (a) the calcined powders, (b) spontaneously carbonated powders during the hydrated process, and (c) 1 gram precipitates and (d) 2 gram precipitates.

**Table 2.** The Assignment of Characteristic FT-IR Absorption Bands and Vibrations for the Hydration and Carbonation Process of Calcined Oyster Shell Powders

Functional groups	Wavenumbers (cm <sup>-1</sup> )	Vibrations
CO <sub>3</sub>	711	symmetric
CO <sub>3</sub>	875	asymmetric
CO <sub>3</sub>	1,084	symmetric
CO <sub>3</sub>	1,469	asymmetric
CO <sub>3</sub>	1,805	symmetric
O-H	1,418	bending
O-H	1,636	symmetric
O-H	3,428	symmetric
O-H	3,643	stretching

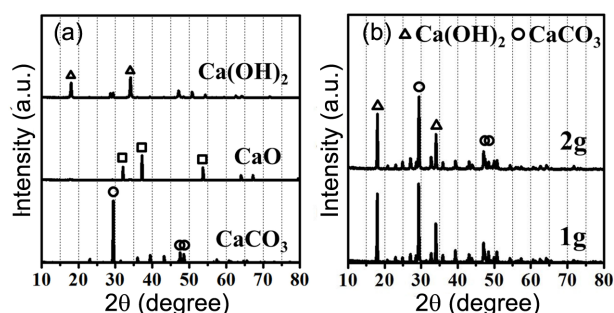


Fig. 7. Crystalline XRD patterns: (a) CaO, Ca(OH)<sub>2</sub> and CaCO<sub>3</sub> reagents and (b) 1 and 2 gram precipitates.

der are 53.29% CaO, ~44.47% CO<sub>2</sub>, and approximately 2.24% of small impurities (containing 0.94% SiO<sub>2</sub>, 0.51% Na<sub>2</sub>O, 0.26% Al<sub>2</sub>O<sub>3</sub> etc.). The content of CaCO<sub>3</sub> in waste oyster shell powders of Table 3(a) reached 97.76%, which is a higher than 97.63% for Canadian limestone and 92.32% for UK limestone.<sup>22)</sup> The purity of CaO was improved to 79.77% at 800°C (Table 3(b)) and 91.2% at 1,000°C (Table 3(c)) by a 1 h calcination process, respectively. In three of the precipitates after the hydrated and carbonated process, CaO and CO<sub>2</sub> contents are almost similar while the content of CaO also showed a tendency of reduction from 62.94% to 66.03%, and the content of CO<sub>2</sub> a tendency to increase from 34.96 to 32.40. The dominant CO<sub>2</sub> content in the hydrated formation suggests that the CO<sub>2</sub> adsorption reaction should occur under exposure process of dry oven or under the ambient atmosphere of hydrated lime as well as by dissolved CO<sub>2</sub> adsorbing and the injected CO<sub>2</sub> bubbling in the algae-containing water. Moreover, it should be noted that these evolutions of the calcium hydroxide and carbonation combination significantly contributed to improving water quality in algae-containing water. These studies, however, did not ascertain complete physical characteristics regarding the presence of eutrophic elements such as phosphorus and nitrogen in algae-containing water.

Therefore, to assess water quality improvement performance of calcined oyster shell powders, total phosphorus (T-P), total nitrogen (T-N), and chemical oxygen demand (COD) were quantitatively determined as shown in Table 4. The T-P concentration was reduced to 0.2 mg/L in 1 gram addition and 0.1 mg/L in 2 gram addition while the value of

Table 4. Water Quality Improvement of Algal Containing Water Using Recycled Oyster Shell Powders as CO<sub>2</sub> Adsorbent: T-P, T-N, and COD

	T-P (%)	T-N (%)	COD (%)
Algal Solution	4.25	7.38	20.33
1g	0.2 (95.3%)	1.14 (84.6%)	13.75 (32.4%)
2g	0.1 (97.6%)	0.66 (91.1%)	13.86 (31.8%)

the raw algal containing water was 4.25 mg/L. The removal efficiency was increased from 95.3% to 97.6%. The T-N concentration was reduced to 1.14 mg/L in 1 gram addition and 0.66 mg/L in 2 gram addition while the value of the raw algae containing water was 7.38 mg/L. The removal efficiency was increased from 84.6% to 91.1%. The COD concentration was also reduced to 13.75 mg/L in 1 gram addition and 13.86 mg/L in 2 gram addition while the value of the raw algal containing water was 20.33 mg/L. The removal efficiency was very similar as 32.4% and 31.8%. Note that the water quality improvement of the algal containing water is very dependent on the amount of powders which consists of calcium hydrates and carbonates formation.

#### 4. Conclusions

The present study was conducted to develop a means of converting waste oyster shells into useful CO<sub>2</sub> adsorbents in algae-containing water. Comminuted and calcined powders of waste oyster shells formed porous surface structures susceptible to hydration and carbonation reactions with water and CO<sub>2</sub>. The hydrated morphology appeared in a peanut shape with larger volumes than that of calcined powder due to clustering between neighboring water molecules in the algal-containing water. Through the gas-liquid carbonation reaction process, hydrolysis between CaO and water molecules resulted in a carbonation reaction with injected CO<sub>2</sub> gas. At this time, the PCC can be formed as the produced Ca<sup>2+</sup> and CO<sub>3</sub><sup>2-</sup> ions are combined. The morphology and microstructure of PCC evolved to calcite crystals which are easily distinguished from the hydrated morphology. After the hydration and carbonation reactions take place, the visible color, water clarity, and light penetration were gradually changed to a pale green from the dark green of algae-

Table 3. X-ray Fluorescence (XRF) Spectroscopy of Comminuted, Calcined, Hydrated and Precipitated Powders of Oyster Shell Waste; (a) Comminution, (b) 800°C Calcination, (c) 1,000°C Calcination, (d) 1 Gram Hydration (e) 1 Gram Precipitation, and (f) 2 Gram Precipitation

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	TiO <sub>2</sub>	MnO	P <sub>2</sub> O <sub>5</sub>	Igloss
(a)	0.94	0.26	0.14	53.29	0.35	0.07	0.51	0.01	0.02	0.21	44.47
(b)	0.48	0.13	0.08	79.77	0.67	0.06	<0.02	0.01	0.03	0.22	17.20
(c)	0.60	0.06	0.05	91.20	0.64	0.06	<0.02	0.01	0.03	0.21	6.09
(d)	0.74	0.18	0.13	62.94	0.57	0.02	<0.02	0.02	0.04	0.21	34.96
(e)	0.81	0.18	0.12	64.40	0.52	0.03	<0.02	0.02	0.04	0.20	33.63
(f)	0.64	0.16	0.11	66.03	0.53	0.02	<0.02	0.02	0.04	0.20	32.4

containing water. Note that the calcined oyster shell powder plays a key role as CO<sub>2</sub> adsorbent and as a recycled lime resource for water quality improvement in response to serious algal bloom occurrence in summertime.

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