

ORIGINAL ARTICLE

Humidity Controlling Material Development from Diatomite, Waste Glass and Cockle Shell Powder: An Experimental Study

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ABSTRACT

Introduction: Innovative ways of balancing indoor relative humidity without compensate for the optimal working environment is crucial as excessive dampness is a risk factor for health symptoms among people and deteriorate building performance. This study examines the potential of fabricating humidity controlling material by sintering a mixture of diatomite, waste glass and cockle shell powder. Moisture sorption isotherms, moisture buffer values, pore structure, surface morphology and flexural strength of the final products were determined. **Methods:** Mixture of diatomite, powdered waste glass and cockle shell powder is mixed at different ratio, pressed and sintered at 1100°C for 20 minutes. The final products were examined using nitrogen gas adsorption-desorption and scanning electron microscopy for mesoporous and morphology properties. The moisture adsorption-desorption performances of the samples were tested using desiccator method while the flexural strength is tested using universal testing machine. **Results:** The sintered products have specific surface area from 5.744 m²/g to 14.765 m²/g and have pore size from 39.5-67.7 nm. The best product, manufactured by mixing 60% diatomite, 30% waste glass and 10% cockle shell powder, showed a good moisture buffer value (MBV: 1.3 g/m² %RH) and flexural strength of 8.23 ± 1.8 MPa satisfy the standard of those commercial porous ceramics. **Conclusion:** Usage of waste glass and cockle shell powder in development of humidity control material helped in the waste reduction. The humidity control material produced can regulate indoor humidity without additional energy consumption. The superior products show excellent characteristics and highly promising for various construction applications.

Keywords: Indoor relative humidity, Humidity control material, Recycle, Malaysia

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INTRODUCTION

There are evidences for association between indoor dampness as a risk factor for health effects such as upper respiratory symptoms, asthma and allergy (1,2). Excessive dampness in buildings can cause damages in terms of building materials such as growth of mould on ceiling or walls (3). Air-conditioning has been the common approach for indoor humidity control, but it is costly when operating hours is long (3). In some cases, pressed organic or inorganic compounds have been used as wall materials to regulate the humidity in buildings (4,5). However, these approaches have disadvantages; the organic materials used is flammable, frail and short-lived while inorganic materials demonstrate inferior moisture adsorption capability and may have adverse effects on

human (6). The increasing demand to sustain a healthy living environment and energy saving has encouraged the augmentation of various kind of humidity control materials.

Humidity control materials (HCM) was firstly introduced and developed in Japan where its application on chemistry industry, textiles and cultural heritage preservation (7). The adaptability of HCM is demonstrated by development of various HCM from materials including activated carbon from bamboo (8), sepiolite (9), waste-based geopolymers (10), inorganic salts (11), fibers and bio-composites (12,13). Applications of HCM on the walls of building helps with humidity control without energy consumption. Thus, it has been considered as one of the most inspiring indoor humidity technologies and well-studied by researchers worldwide.

Diatomite is a natural porous nonmetallic mineral containing mainly of the fossilized skeleton remains of diatom (14). It has several attractive characteristics such

as high porosity, unique pore structure, nontoxicity, large surface area and excellent adsorption ability (15). Diatomite has been utilized as interior wall materials with humidity controlling properties with the development of diatom ooze and diatomite composite building materials recently (16, 17, 18).

Glass used for various applications as well as in the traditional manufacturing of glass bottles. However, glass recycling is limited in Malaysia and even drives some glass recycling companies out of business due to unable to secure continuous sources of bottles (19). Wabisabi Concrete Solutions, manufacturer of recycled building materials such as producing stone slabs made from recycled glass has proved the business to convert waste into wealth while reduces waste glass head for landfills (19). In this study, waste glass was used as adhesive agent and to increase the strength of the final products.

Statistics from Department of Fisheries Malaysia shows that production of cockle reached 9,596.6 tonnes in 2016 (20). Cockle shell has been studied for the potential as an alternative for the calcium carbonate sources instead of using the limestone (21). This is because limestone mining in large scale can affects the surrounding environment and high operating cost dealing with environmental compliance. Besides, the shells were deemed as non-valuable and are usually thrown-away. Owing to the availability and inexpensive of these cockle shells, they are highly promising to be utilized as an economical source of calcium carbonate for industrial applications. In this study, the shells of cockle, *Anadara granosa*, were used as filler material.

Until now, there are few reports on the diatomite with recyclable materials such as waste glass and cockle shells composite humidity control material. Thus, this study aims to prepare a novel diatomite, waste glass and cockle shell composite HCM and determine the porous properties, moisture adsorption-desorption performances and flexural strength.

MATERIALS AND METHODS

Materials

The diatomite used in this study is Perma-Guard Diatomaceous Earth from USA where it is repackaged by local supplier, Green Integrity Trading Sdn. Bhd. The main chemical compositions of diatomite included: SiO₂ 89.78%, Al₂O₃ 2.74% and Na₂O 1.65%. Waste glass were collected from a glass manufacturer in Kuala Lumpur and cockle shell were collected from seafood restaurant around Kuala Lumpur. Waste glass were washed and dried at 100°C for 24 hours. It was grinded using a planetary ball-mill machine then sieved through a stainless-steel sieve with 300µm to get the fine waste glass powder. Cockle shell collected were washed thoroughly with detergent and tap water. The cleaned

cockle shells were boiled for 15 minutes at 100°C and left to cool at room temperature. The shells were then exposed and dried under the sun till completely dry. Then, it was crushed into small pieces using pestle and mortar before being grind into fine powder. The resultant cockle shell powder is passed through a stainless-steel sieve of 106µm.

Methods

Sample Fabrication

Diatomite, waste glass powder and cockle shell powder were mixed at different ratio as stated in Table I and labeled accordingly. The mixed powder was pressed at 32MPa using compression moulding presses, model CMV100H-20-BCLPX serial #10967 for five minutes. The pressing moulded the initial powder into a green compact with dimension of 15cm x 15cm x 5 mm. The green compacts were then sintered in “DAIHAN” Wise Therm® digital muffle furnace at 1100°C for 20 minutes. All the sintered samples were stored in desiccator and were dried at 100°C for 24 hours prior being analyzed.

Table I: The percentage ratio of the mixed powders (wt %)

Green compact	Diatomite	Waste glass powder	Cockle shell powder
D5W3C2	50	30	20
D6W3C1	60	30	10
D7W2C1	70	20	10
D8W1C1	80	10	10

Abbreviations:
 D5W3C2, Diatomite 50% + Waste glass powder 30% + Cockle shell powder 20%;
 D6W3C1, Diatomite 60% + Waste glass powder 30% + Cockle shell powder 10%;
 D7W2C1, Diatomite 70% + Waste glass powder 20% + Cockle shell powder 10%;
 D8W1C1, Diatomite 80% + Waste glass powder 10% + Cockle shell powder 10%

Determination of moisture adsorption-desorption ability of the sintered samples

Moisture adsorption-desorption isotherm were determined according to method JIS A 1475-2004 (22). The sintered products of dimensions 12 cm in diameter and thickness of 0.5 cm were kept in a desiccator and saturated salt solution such as magnesium nitrate (53% RH) and sodium chloride (75% RH) were used to regulate the relative humidity. The desiccator is placed in an air-conditioned room where temperature is maintained at 23°C ± 2. The samples were wrapped with aluminum foil except for the top surfaces, to allow the water vapor adsorption-desorption through the top surface only. Humidity inside desiccator was changed from 53% to 75% after the weight of samples stabilized at a constant value. Samples were weighted at four hours interval until the change rate of the sample was less than 1%. After that, humidity inside desiccator was decreased to 53% and the samples were weighed until equilibrium was reached. The amount of moisture adsorbed by samples was calculated by the following equation:

$$M = \frac{m_t - m_0}{m_0}$$

where M is moisture adsorbed, m₀ is the initial weight of the dried sample, and m_t is the sample weight at time t. Moisture buffer value (MBV) of the samples were

tested according with the Japan industry standard. The samples were first conditioned at 23°C and 53% relative humidity until the weight changes of the samples was less than 1%. Next, the samples were exposed to a series of step changes in relative humidity between two levels within 24 hours i.e 12 hours at 53% RH and 12 hours at 75% RH at 23°C. Three cycle were carried out with a total test time of 72 hours. The MBV was calculated using following formula:

$$MBV = \frac{\Delta m}{A(\Delta RH)}$$

where Δm is the average between the adsorbed and desorbed mass (g), A is the exposed surface (m²) and ΔRH is the difference in relative humidity (%).

Based on the Nordtest project, the MBVs of specimens were then classified into five categories: negligible (MBV: 0.0-0.2), limited (MBV: 0.2-0.5), moderate (MBV: 0.5-1.0), good (MBV: 1.0-2.0) and excellent (MBV: 2.0 or more).

Morphology analysis of green compact and sintered samples

The sintered sample of D5W3C2, D6W3C1, D7W2C1 and D8W1C1 cut into small pieces and green compact in powder form were stuck onto the stub using double sided tape. Stub containing the samples were then placed in oven at least 24 hours prior to gold coating in the sputter coater. The surface microstructure of green compact and sintered products was observed using LEO 1455 variable pressure scanning electron microscope (SEM) under magnification of 3.0 k at 15.00 kV. Three spectrums of each samples were selected for the energy dispersive x-ray (EDX) analysis to identify the elemental composition of the samples being analyzed.

Determination of surface area, porous properties and flexural strength of the sintered samples

Nitrogen gas adsorption-desorption measurements were carried out at 77 K by a specific surface area analyzer (BELSORP-mini II, Japan). 0.5 gram of each sintered samples were outgassed at 200°C for 24 hours prior to the exposure to nitrogen gas. The surface area was measured using Brunauer-Emmett-Teller (BET) method. The BET equation is applied in linear form:

$$\frac{p/p_0}{n(1-p/p_0)} = \frac{1}{n_m C} + \frac{C-1}{n_m C} (p/p_0)$$

where n is the specific amount adsorbed at the relative pressure p/p_0 , n_m is the specific monolayer capacity and C is the BET constant. Results on BET surface area, total pore volume and pore diameter obtained were calculated by BELSORP analysis program installed in the computer.

The flexural strength was determined using Universal Testing Machine according to ASTM C1161-13 method (23). Sintered samples were cut into length 90 mm, width 8.0 mm and 6.0 mm depth. Each sample with three test specimens were measured via a three-point bending test

with a span of 40 mm and a crosshead speed of 1.0 mm/min. Testing was carried out at ambient temperature at $23 \pm 2^\circ\text{C}$ and relative humidity of $50 \pm 10\%$.

RESULTS

Moisture adsorption-desorption ability of the sintered samples

Figure 1 shows the moisture adsorption-desorption isotherms of the sintered samples. All the tested samples showed a similar trend for moisture sorption. When the relative humidity increased, the samples adsorbed moisture while desorbed water vapour when the humidity decreased. The increased in mass of samples during adsorption is higher than the decreased in mass of samples during desorption similar with other studies (8, 10, 24). The rate of desorption was higher at the beginning of the exposure to lower relative humidity compared to the rate of adsorption when the samples is placed in higher relative humidity. The water vapour was released rapidly and the desorption process slowed down after a period of rapid release. The calculated moisture buffer value (MBV) of the samples were tabulated in Table II. The MBV for the samples range from 1.2-1.9 g/m² %RH shows that the tested samples are good humidity control composite material (12, 13, 25).

Morphology of green compact and sintered samples

Figure 2 shows the SEM images of D5W3C2 (Fig. 2A), D6W3C1 (Fig. 2B), D7W2C1 (Fig. 2C), D8W1C1 (Fig. 2D), mixture of diatomite, waste glass and cockle shell powder before sintering (Fig. 2E). As seen from Figure 2,

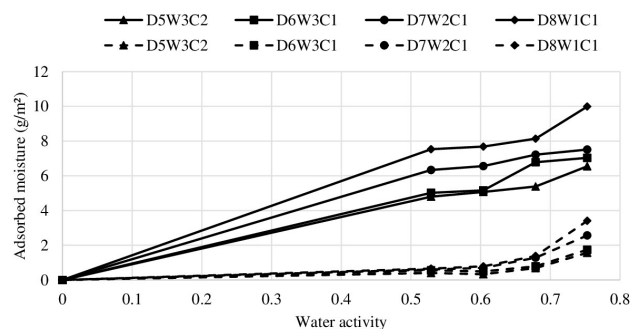


Figure 1: Moisture adsorption-desorption isotherms of the sintered samples. (solid lines: adsorption; dashed lines: desorption)

Table II: Moisture buffer values, surface area, porous properties and flexural strength of D5W3C2, D6W3C1, D7W2C1 and D8W1C1

Samples	Moisture buffer values (g/m ² %RH)	BET surface area (m ² /g)	Total pore volume (cm ³ /g)	Average pore diameter (nm)	Flexural strength (MPa)
D5W3C2	1.2	5.744	0.097	67.752	9.76
D6W3C1	1.3	9.197	0.102	44.41	8.23
D7W2C1	1.6	12.765	0.133	41.633	5.11
D8W1C1	1.9	14.984	0.146	39.506	1.95

Abbreviations:

D5W3C2, Diatomite 50% + Waste glass powder 30% + Cockle shell powder 20%;
D6W3C1, Diatomite 60% + Waste glass powder 30% + Cockle shell powder 10%;
D7W2C1, Diatomite 70% + Waste glass powder 20% + Cockle shell powder 10%;
D8W1C1, Diatomite 80% + Waste glass powder 10% + Cockle shell powder 10%

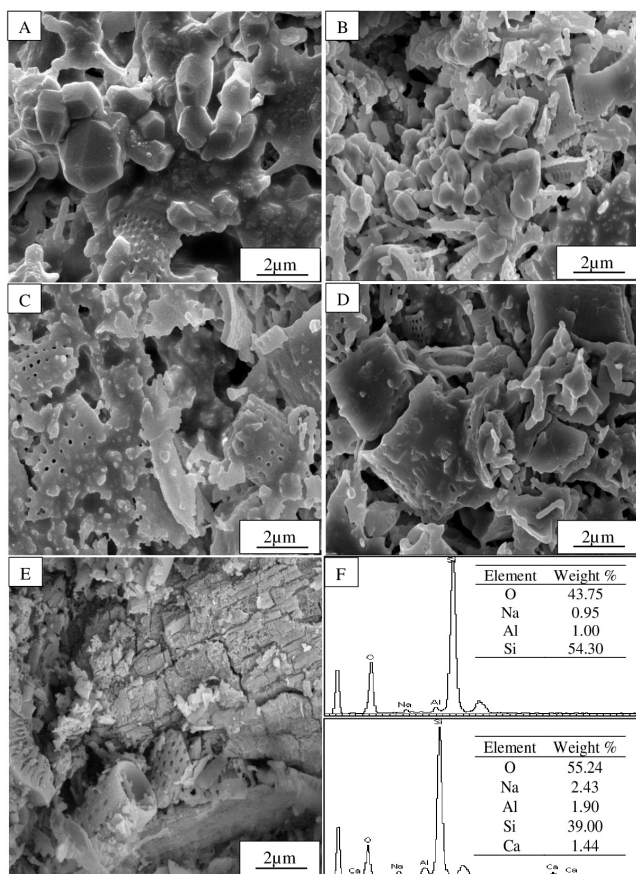


Figure 2: SEM images of D5W3C2 (A), D6W3C1 (B), D7W2C1 (C), D8W1C1 (D), green compact (E), and EDX images of green compact and D6W3C1 (F)

diatomite is cylindrical, hollow with special poriferous structure and porosity (27). A lot of diatom fragments can also be found on the surface of diatomite. Cockle shell powder is not porous material, as illustrated in Fig. 2E. Natural cockle shell displays layers structure known as aragonite (21). The structures of the cockle shell are observed to changed drastically when calcined at 1100°C. The surface of calcined waste cockle shell has irregular shape with some of them bonded together to form large aggregates (21). Cockle shell particles are bonded with diatomite by calcination as Fig. 2A and Fig. 2B shown. Figure 2F shows the EDX images of green compact and D6W3C1. From the results of EDX analysis, traces of calcium element can be found on the surface of sintered products. This proves that a novel composite material is fabricated successfully.

Surface area, porous properties and flexural strength of the sintered samples

Figure 3 illustrates the nitrogen adsorption-desorption isotherms of the sample products at 77K. According to the IUPAC classification system, all the isotherms are in conformity with the type IV curves with hysteresis loops. This confirms the mesoporous structure within these products (21). The nitrogen sorption volume of tested samples range from 75.016 cm³/g (D5W3C2) to 99.521 cm³/g (D8W1C1) at a relative pressure closes to one. The surface area, porous properties and flexural strength

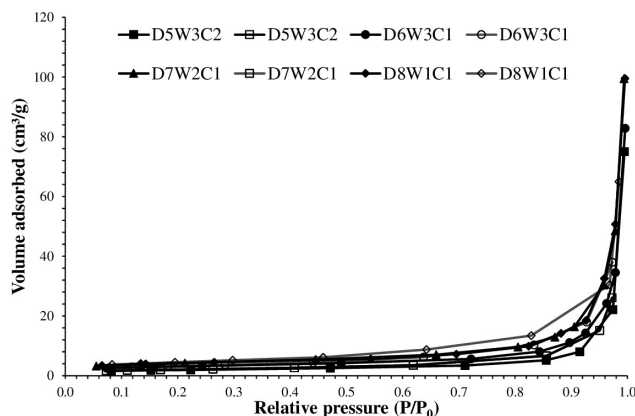


Figure 3: Nitrogen adsorption-desorption isotherms for D5W3C2, D6W3C1, D7W2C1, and D8W1C1 at liquid nitrogen gas temperature (77K)

of all sintered products are summarized in Table II. D5W3C2 has the lowest specific surface area and total pore volume with 5.744 m²/g and 0.097 cm³/g respectively. Conversely, D8W1C1 has the largest specific surface area of 14.984 m²/g and highest total pore volume of 0.146 cm³/g. The average pore diameter of D8W1C1 is the smallest with 39.506 nm while D5W3C2 has the largest average pore diameter of 67.752 nm. D5W3C2 has the highest flexural strength with 9.76 MPa while D8W1C1 has the least flexural strength with 1.95 MPa.

DISCUSSION

Moisture adsorption-desorption ability of sintered samples

The adsorbed moisture contents of D8W1C1 is much higher than those of D5W3C2. These results proved that the final properties of the composite HCM is affected by the initial material (3, 9, 12, 13). Diatomite used in this study which is a natural porous material has much higher porosity than waste glass and cockle shell powder do, and the formation of small crystalline SiO₂ in sintered samples is enhanced as the diatomite content increased. Therefore, D8W1C1 with more diatomite in percentage than D5W3C2 can adsorbed more moisture.

The MBV of all the samples is within 1.0-2.0 thus considered the samples were characterized by a good predisposition to moisture buffering. The MBV supported the hygroscopic characterization of the sample’s responses to environmental conditions in realistic indoor application of these HCM in air-conditioned buildings in tropical climate (25).

Morphology of green compact and sintered samples

The SEM analysis shows the changes of pore diameters and grain shape in the sintered samples because of sintering process. Sintering theory (28) can be used to explain the observed morphology and changes of these materials. During sintering of a compacted powder, materials were transported through a film of

thin liquid from the connection area between grains to the off-contact neck region resulted in densification and shrinkage. This process causes pore size to reduce and grain shape changes continuously thus become gradually anhedral until there is no more pore in the material. However, this sintering theory have some intrinsic flaws. As sintering proceeds, pore size reduces continuously where large pore collapses and maximum pore size should lessen when sintering temperature increases. But, the above-mentioned changes in the pore size were not observed in this study after the sintering process. Samples D5W3C2 has macropores while D6W3C1, D7W2C1 and D8W1C1 have mesopores in structure (29). The pores in the final products is not totally eradicated.

Cockle shells is an alternative calcium carbonate sources instead of limestone. When it is sintered at 1100°C, transformation of CaCO₃ to CaO happened. Findings illustrated in Figure 2 were found like the calcined limestones studied by Sun and colleagues (30) where the grain shapes with some grain-neck growth were resulted from the sintering.

Waste glass powders are interlaced in the samples to form a network structure, increasing the mechanical properties of the composite materials with the diatomite and cockle shell powder enclosed therein (31).

Surface area, porous properties and flexural strength of the sintered samples

Nitrogen gas adsorption-desorption measurement of the sintered products generated important information on the porous properties including surface area, pore size and pore volume which are crucial for HCM. Most pores of D5W3C2 are macropores with average pore diameter of 67.752 nm. According to the Kelvin capillary condensation theory, pore diameter affected the relative pressure of adsorbate during capillary condensation. Occurrence of capillary condensation required greater relative pressure when the pore size is larger. The presence of macropores in D5W3C2 made it required more relative pressure for the water vapor condensed within the pores and constrain the diffusion of water vapor (32, 33).

D8W1C1 has the largest BET surface area which is almost three times larger than that of D5W3C2, the largest total pore volume and average pore diameter less than 40 nm, showing a good mesoporous structure (32). When comparing D8W1C1, D7W2C1 and D6W3C1, the specific surface area and total pore volume increase with the increase amount of porous materials, while average pore diameter decreases (34). This improves the moisture adsorptive and desorptive performances of the sintered samples. All the pore structure parameters confirm that the final products have unique porous structure that can adsorb and desorb water vapor. On the other hand, D5W3C2 and D6W3C1 have

flexural strength more than 6 MPa which satisfy the Japanese Industrial Standard (JIS) for the flexural strength test. These results are in conjunction with (31,35) which revealed that addition of waste glass powder in the composite increases the flexural strength. The values of bending strength of the final HCM increases with the percentage ratio of waste glass.

CONCLUSION

Four kinds of diatomite, waste glass and cockle shell powder composite humidity control materials were fabricated by sintering mixture of the three raw materials. The moisture adsorption-desorption abilities, surface morphology and pore structure as well as flexural strength of the final products were examined. D8W1C1 showed the superior moisture adsorption performances. The pore structure and morphology analysis reveal that cockle shell powder particle coats on the diatomite surface and complete transformation of CaCO₃ to CaO. With the increasing content of waste glass, the flexural strength is improved obviously. With moisture adsorption-desorption ability, pore structure and flexural strength in concerns, the best product, manufactured by mixing 60% diatomite, 30% waste glass and 10% of cockle shell powder, sintered at 1100°C for 20 minutes.

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