

DEVELOPMENT OF POROUS CERAMICS AS WALL TILES WITH HUMIDITY CONTROLLING AND ANTIMICROBIAL CHARACTERISTICS FROM MODIFIED DIATOMACEOUS EARTH (DE): POTENTIAL TO IMPROVE INDOOR AIR QUALITY

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ABSTRACT

Background: Indoor humidity is one of the indoor comfort issues in indoor environment in Malaysia. Imbalance of indoor humidity would arise when there is an inefficient use of air-conditioning system which causes overgrowth of microbes. The use of ceramics from Humidity Control Materials (HCM) is one of the solutions to regulate indoor air humidity and reduce bacterial growth and thus preventing problem related to Sick Building Syndrome (SBS).

Objective: The aim of this study is to develop porous ceramics as walling materials in the form of tiles with humidity controlling ability and antimicrobial characteristics from modified Diatomaceous Earth (DE). **Method:** This experimental study consists of the development of porous ceramics as wall tiles from DE, waste glass and oyster shell in several formula (80%, 70%, 60% and 50% DE). The developed porous ceramics were tested for its ability for humidity adsorption and desorption, strength test and anti-microbial assay test. **Result:** The humidity adsorption desorption ability of the tiles was increased as the DE content increased. The humidity adsorption and desorption performance for modified DE was within the range of 3-5%. The inhibition zones against *S. aureus* and *P. aeruginosa* depends on the percentage of DE and it increases as the content increases. On the other hand, the inhibition zones for *S. aureus* depends on the percentage of oyster shell and it increases as the content increases. The average of clear zone against bacterial was 15 mm. The impact test found that the formula of 50% DE has the greatest impact test compared with three other ceramics. **Conclusion:** This study found that a suitable ratio for porous ceramics with humidity controlling and anti-bacteria characteristics could be developed by using 50% modified DE. The modified DE added with local and low-cost materials have the potential to solve the problem of humidity imbalance and microbial growth in office settings can be solved in order to reduce sick building syndrome and ensure productivity of workers is maintained.

Keywords: anti-bacteria, oyster shell, calcium oxide, indoor air quality, relative humidity.

1. Introduction

Sick Building Syndrome (SBS) is a well-known health effects resulting from of poor indoor air quality. It

is one of the major health concerns where building occupants experience acute health effects or thermal comfort problems but where no specific illness or causes can be recognized. This health effects showed a significant relationship with the time spent in a

building (DOSH, 2010). Based on the findings from the survey done, most of occupants (52.8%) categorized their workplace to have the presence of high pollutant level (Ahmad & Hassim, 2015). The result is supported a study done by Fadilah & Juliana., (2012), where occupants showed prevalence of SBS to be between the range of 33.8- 47.4%. Another local study reported SBS problems among workers in wet laboratory setting was 45% compared to 20% among those in dry laboratory setting (Zuliza et al. 2016). Internationally, the American Society of Heating, Refrigerating and Air Conditioning Engineers, (ASHRAE, 2013) suggested that the ideal relative humidity for office building was 20% - 70% meanwhile the Environment Protection Authority, EPA predicted that poor indoor environment quality will lead to lost productivity, higher expenses in medical care and greater number of lost work days (EPA, 1997).

In a tropical country like Malaysia, indoor humidity in offices are often characterized by the use of air-conditioning as one of the main methods to cool the indoor air to ensure thermal comfort (Ahmad, 2018). It was found that most (67.2%) workers in a local study occupies offices fitted with Mechanical Ventilation and Air-Conditioning (MVAC) system which operates on an average of 9 hours per day (Ahmad, 2018). However, the issue with the use of MVAC system is the need to save energy to ensure costs is maintained by reducing ventilation efficiency to minimal. Reduced ventilation means lowering the amount of indoor and outdoor air exchange which causes build-up of humidity in indoor environment. When improper MVAC system is in place, this leads to poor distribution of fresh air to all areas in the office setting. Most of the buildings in Malaysia would be facing similar problem because air-conditioning will not run for 24 hours, instead it is usually switched on during working hours only. Because the high likelihood of humidity problems, ideally during the development of a building, material selection for walls must consider moisture absorption characteristics to ensure humidity can be control and growth of mold or proliferation of bacteria is not encouraged.

Humidity Control Materials (HCM) in the form of porous ceramics is one of the solutions to regulate indoor ambient air humidity. To control moisture in indoor environment, Diatomaceous Earth (DE) as one of the HCM that has been shown in several studies to be a very good humidity adsorption material (Vu et al., 2013; Hu et al., 2017; Zheng et al., 2017; Ediz, 2008). To add to the ability of HCM in its antimicrobial activi-

ties, the use of additives materials such as oyster shells, cockle shells, date palm seed powder and Arabic gum has been reported in literature (Xing et al. 2013; Hamidi & Zulkifle, 2013; El Hassni et al. 2004; Montenegro et al. 2012). In considering materials being of local origins, oyster shell are primarily found in Malaysia and is considered as waste products. Xing et al, (2013) reported that oyster shell have anti-microbial characteristics at high concentration. Waste glass in powder form has been often added in ceramic products as fillers and as a refractory material to which it can retain a specific physical shape after being applied with heat-treatment process.

Such porous ceramics if able to be developed is hypothesized to help regulate humidity in indoor environment, in which it has an ability to balance indoor humidity by adsorbing the humidity from its surrounding and desorbing it into the air when needed. From the previous studies reported (Ahmad & Hassim, 2015; Rohizan & Abidin, 2015; Abidin et al., 2017) there is the need for the development of porous ceramics to help reduce humidity problems and sick building syndrome in building offices.

This study aims to develop porous ceramics with humidity controlling ability and anti-microbial characteristics from modified Diatomaceous Earth (DE). This study will test and investigate DE and other potential materials including oyster shell powder and waste glass that can be used in the development of porous ceramics. The resultant porous ceramics developed will be subjected to analysis for ability of humidity adsorption desorption, antimicrobial assay and impact strength test. These characteristics is expected to reduce the problems related to high moisture content in indoor environment, together with reducing the problems associated with the growth of microbes indoors.

2. Materials and Method

This study is an experimental study which consists of the development of porous ceramics in the form of wall tiles conducted in the Institute of Tropical Forestry and Forest Products (INTROP), Universiti Putra Malaysia (UPM). The performance of anti-microbial testing was performed in Medical Microbiology and Parasitology Laboratory and impact strength test was done in Environmental Health Laboratory, at the Faculty Medicine and Health Sciences, UPM.

2.1 Experiment Procedures

2.1.1 Materials and Sample Preparation

The main porous material, diatomaceous earth (DE) was bought from Green Integrity Trading (GIT Sdn Bhd, Malaysia), Diatomaceous Earth Food Grade supplier in Kuala Lumpur. This sample was stored in room temperature to preserve the products from interacting with humidity. Waste glass was collected from a recycling company at Machap, Johor. The waste glass was dried at 105°C for 24 hours and was grinded using wet-ball mill machine. The waste glass was then dried again at 105°C. The powdered waste glass was then sieved using a sieve with mesh of 300 µm and were then stored in polyethylene plastic bag for the next process in the development of porous ceramics.

The method for preparation of oyster shell powder was adopted from a reference (Mohamad et al., 2016). Raw oyster shells were washed thoroughly with tap water. The shells were then brushed to clean the impurities found on the shells and to ensure there were no mud left. Next, the cleaned oyster shells were boiled for 15 minutes at 100°C and cooled at room temperature. The shells were then removed and cleaned with distilled water. The shells were then exposed and dried under the sun for 1 day. Drying is important to avoid agglomeration during grinding process. The shells were then crushed into small pieces using a pestle and mortar. The powder obtained was then sieved through a 106 µm stainless steel sieve shaker. The oyster shells powder was then kept in the polyethylene plastic bag for development of porous ceramics.

2.1.2 Development of Porous Ceramics

The modified DE products mixed with waste glass and oyster powder in different ratio (the percentage ratio of mixed samples shown in Table 1). By using compression molding presses, model CMV100H-20-BCLPX serial #10967, the mixed samples were pressed at 60 mPA for 5 minutes. The pressing allows the initial powder form of humidity control materials to sustain its molded shape. Green compacts with dimension area of 15cm x 15 cm and 5 mm in height were produced from the molding process. The green compacts pieces were the placed on aluminum foil and the weight before sintering were measured and recorded. The green compacts were sintered in "DAIHAN" Wise Therm (R) digital muffle furnace, manufactured by Daihan Scientific, Korea, at 1100°C for 20 minutes. All the green compacts that

have been sintered are dried at 105°C for 24 hours before being analyzed and kept in desiccators. The sintered samples are now sturdy pieces of samples that have a tile-like shape.

Table 1: The percentage ratio of the mixed samples

Materials	DE	Waste Glass Powder	Oyster Shells Powder
50-30-20	50	30	20
60-30-10	60	30	10
70-20-10	70	20	10
80-10-10	80	10	10

2.1.3 Humidity Adsorption-Desorption Test

The specific relative humidity was generated in the form of water vapor from saturated salt solution using a Japanese standard method (JIS, 2017). The sintered samples were wrapped on the sides with aluminum foil and tape, except on the top surface, to allow water vapor to adsorb and desorb through it. The process is as follows: firstly, the sintered samples were kept with magnesium nitrate (53% RH). When the relative humidity was constant at the required value, the weight of the sintered samples was measured. Next, the relative humidity was then changed. This humidity was maintained for 24 hours for it to stabilize in this condition. After completing the adsorption process using sodium chloride 75% RH, the humidity was changed to 53% RH by switching to magnesium nitrate salts.

The reading was immediately recorded 2 hours after the salt solution was changed. Silica gel was placed in a similar desiccator to keep the relative humidity constant at 53% RH. The salt solution (magnesium nitrate) inside the desiccator cannot retain the relative humidity for the expected duration of time as sodium chloride. This problem affects desorption process and the reading of the samples as well. In order to solve this problem, the salt solution from magnesium nitrate needed to be changed every four hours. However, due to budget constraint, silica gel was used to replace the magnesium nitrate.

2.1.4 Antimicrobial assay

i. Sample preparation

For the agar diffusion tests, the sintered samples were prepared in the form of powder. Pestle and mortar was used to crush and grind the ceramics samples into fine powder form. About 1g of each sample was diluted in distilled water and heat up at 100° C for 30 minutes. This step was done to ensure the mixture of powder and distilled water was homogenized. All the samples were kept in universal sterile container for next process. The homogenization process took about 16- 24 hours to allow the adsorption of distilled water into the dissolve powder.

ii. Bacterial culture

Staphylococcus aureus (*S. aureus*) ATCC 12228, which are gram positive bacteria and *Pseudomonas aeruginosa* (*P. aeruginosa*) which are gram-negative bacteria were used for microbial assay testing. *S. aureus* bacteria is the gram-positive bacteria with the highest colony numbers and percentage in indoor air found inside building environment (Hussin et al, 2011). Similarly, *P. aeruginosa* has been characterized and identified bacteria exist in the building (Hussin et al, 2011). The method for the culturing of bacteria are as follows: first, sterilize the laminar air flow with alcohol; second, *S. aureus* was placed inside the ice box to preserve its shelf life; third: before streaking the bacteria onto red blood plate ager, rub the stock tube with fingers to thaw; forth: pipette tip was used to scrape the bacteria and to inoculate the red blood agar plate; fifth: use a sterile loop to streak the bacteria. After completing the five successive steps, bacteria were grown in an incubator in an upside down position at 37° C for 24 hours.

iii. Antibacterial testing

The plate from the bacterial culture was taken out from incubator. A single colony of *S. aureus* was scraped with a sterile loop and the bacteria is inoculated in sterile normal saline, and it is incubated until 0.5 Mac Far land standard turbidity was obtained, then it is used for assay. The concentration was adjusted to 10⁶ CFU/mL to bacterial strains. The test tube with microbial suspension was then prepared by removing the cap and sterilized on a gentle flame. Pipette the bacterial broth from the tube with pipette tips to inoculate on the red blood agar and repeat the flame sterilization to avoid contamination. The red blood plate agar was swabbed in a side-to-side motion with sterile cotton swab. This is to ensure bacteria growth is uniformed. The plate is rotated lightly in different motions.

After the entire surface of the plate has been covered, the cotton swab is disposed in biohazard container. The red blood plate agar were then divided into five equal quadrants by used a marker. Wells on the agar plates were made with the help of blue pipette tips. Well sizes were measured and labelled from 1-5 for each type of the samples of porous ceramics prepared at the back of the red blood agar plate. Approximately 100 µl of ceramics samples homogenized in distilled water were transferred into the wells provided. All the plates with ceramics sample were kept in incubator for 24 hour at 37° C with a lid side up position. Three replicates were made to obtain the mean clear zone diameter. Finally, the inhibition zones diameters were measured.

2.1.4 Impact Strength Test

The impact strength test method was adopted from Agbayani & Espinosa (2006). Impact strength test was done to gauge the limit of porous ceramics to withstand different loads. Load (100g, 200g, and 500g) were dropped on porous ceramics in increasing weight with similar height (0.68cm) and marks will be given based on rating scale below,

- 10 – Extensive damage, crushed
- 20 – Broke into fragment
- 30 – More cracks but did not break into fragments
- 30 – More cracks but did not break into fragments
- 40 – Chipped; few cracks
- 50 – No cracks, no damage

3. Results

3.1. Humidity adsorption desorption performance

Figure 1 illustrates the humidity adsorption desorption performance of porous ceramics from modified DE. Four ceramic tiles with different formula were tested for humidity adsorption desorption test. The adsorption process was analyzed 24 hours after the salt solution of magnesium nitrate (53% RH) changes to sodium chloride (73% RH). The results show that the adsorbed moisture measured in weight increase slightly with increasing time. Ceramic tiles with formula 80-10-10 stabilized at the same percentage before gradually increasing. The percentage of adsorbed moisture for formula 80-10-10 was higher compared to others. Meanwhile ceramic tiles with formula 50-30-20 showed least amount of adsorbed moisture. For desorption process which is at 53% RH condition, RH drastically dropped for ceramic tiles with formula

50-30-20 after the salt solution was changed and increased with increasing times up to 48 hours.

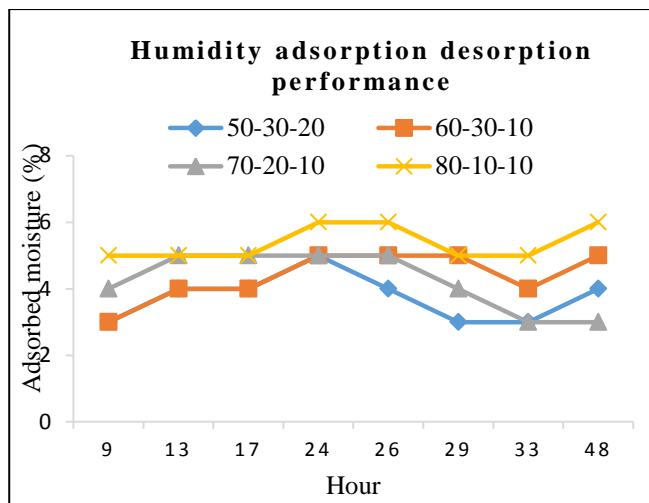


Figure 1: Humidity adsorption desorption performance of porous ceramics from modified DE sintered at 1100° C for 20 minutes

3.2. Antimicrobial assay

Figure 2 shows the diameter of inhibition zone for DE, oyster, DE and oyster, ceramic tiles samples for formula 50-30-20, 60-30-10, 70-20-10 and 80-10-10 against bacterial strain, *S. aureus*. Zone of inhibitions were confirmed when the diameter (mm) of each zone exceeds 8 mm (the size of the pipette tips). Ceramic tile with formula 60-30-10 showed no antimicrobial activity while formula 70-20-10 and 80-10-10 had no clearly significant inhibition zones against the bacterial strains.

All raw samples before heat treatment showed antimicrobial activity exceeding the samples after undergoing heat treatment at 1100° C. Diameter of inhibition zone of raw oyster was higher compared to raw DE and DE with oyster. Samples of 50-30-20 sintered at 1100° C exhibited good bactericidal activity towards *S. aureus*.

By referring to the Figure 3, after heat treatment was applied, all samples showed presence of inhibition zones against *P. aeruginosa*. Ceramic tile with formula 50-30-20 exhibited strong antibacterial activity compared to other samples. Comparing the assay tests between raw DE against gram-positive and gram-negative bacteria, there is a reduction of clear zone.

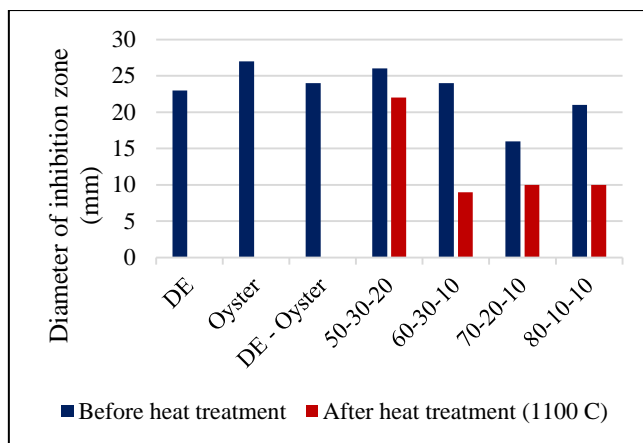


Figure 2: Diameter of inhibition zone across raw materials and samples (non-sintered in blue and sintered in red) against *Staphylococcus aureus* (Gram-positive bacteria)

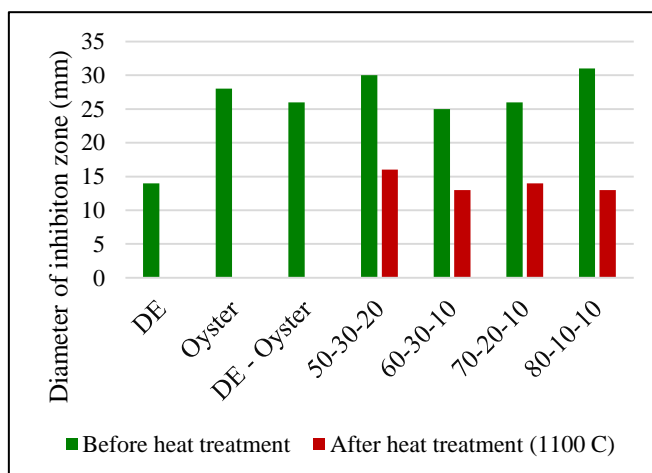


Figure 3: Diameter of inhibition zone across raw materials and samples (non-sintered in green and sintered in red) against *Pseudomonas aeruginosa* (Gram-negative bacteria)

3.6. Impact strength test

Each of the ceramic formulas was tested for impact strength with three different loads at the same height. Referring to the Table 2, the total mean after the impact test was done indicates that ceramic sample with formula 50-30-20 has the greatest impact test compared to the three other ceramics. In contrast, formula 80-10-10 has the least of mean value, illustrating that this ceramic was the weakest one. The results showed that ceramic tile with formula 80-10-10 was not feasible to form ceramics.

Table 2: Impact test with different loads

Ceramics	1 (100g)	2 (200g)	3 (500g)	Mean
50-30-20	40	30	20	30.00
60-30-10	30	20	20	23.33
70-20-10	30	20	10	20.00
80-10-10	20	10	10	13.33

4. Discussion

Relative humidity is one of the factors reported that may influence worker's thermal comfort in office setting. According to a study by Ahmad & Hassim (2015), relative humidity is inversely proportional with temperature. This means that when temperature in indoor environments that is occupied is low for comfort, relative humidity is usually high hence leading to excessive dampness (Ahmad, 2018). It is reported that the average relative humidity in office buildings in a tertiary institution was high (72.1%), exceeding the allowable range when compared with the local Malaysian standard (DOSH, 2010). The complexity of high relative humidity not only causes dampness problem but also triggers the growth of microbes including bacteria and fungi. Othman et al., (2015) reported that there is a positive correlation between relative humidity with bacteria growth. The report further added that high indoor air humidity like Malaysia could be a great reservoir for microbe to grow and proliferate, in some cases, the microbe would manage to spread diseases in such condition.

Conventional solution of using non-energy consuming process presented HCM as one of the alternative ideas to solve the problem related to MVAC system (Hu et al, 2017). As suggested in one study, the concept of cradle to the grave before development of building should be implemented (Ahmad & Hassim et al., 2016). In other words, the selection of building materials must be considered at the earlier stage to ensure indoor humidity will not be a problem to occupants.

Previous study reported that the main factor causing building defects in hospitals was moisture issues (Othman et al., 2015). Microbes may be able to freely live in moisture leaks location such as walls, floors, windows and doors. Moisture issues not only causes defects on the building surfaces, but it can contribute to ill-health problems among the occupants

when exposed to these environment (Othman et al., 2015). Installing tiles to retrofitted buildings to solve indoor air humidity problems is possible. In a previous study (Ahmad, 2018), it was found that 39.1% of respondents strongly agrees for building administrator to install new tiles in their occupied offices if the ceramic tiles can prevent respiratory health problems related with poor indoor air quality. Based on the findings, there is the positive expectation for such tiles to be used in offices after the building have been constructed as an intervention method to reduce problems related to excessive moisture and microbial growths.

In this study, all four samples exhibited humidity adsorption-desorption performance. By comparing samples with the highest and the least percentage of DE, it was obvious that ceramic tiles with formula 80-10-10 which contained 80% DE presented an excellent performance in adsorption. Meanwhile the ceramic tile with formula of 50-10-10 that only had 50% DE had approximately two times lower adsorption performance than the formula with 80% DE. As reported in another study, the ability of moisture adsorption desorption increases as the percentage of DE increased (Vu et al., 2013).

It was found that desorption process took 1 day to reach 75% RH. Yet, for the desorption process, it only took 2 hours for it to stabilize. The reading needs to be recorded immediately before the salt solution is fully saturated. The water vapors that are released by the salt solution were found to evaporate from the modified DE sample to the top of the glass desiccator. This condition was notable as the most suitable time to measure the weight difference of the sample.

This finding was supported by Zheng et al., (2017), that found the adsorption process took longer than desorption process for it to stabilize. This occurrence was caused by pore capillary effects (Zheng et al., 2017). The existence of capillary will promote water vapors to diffuse into the pore of the ceramics thus increasing the adsorption ability. DE has a porous surface and comprised of tiny capillary that has an ability to adsorb water vapor from atmosphere, thereby worthy for water vapor adsorption (Al-Ghouti, 2003). The study that was conducted by Qian et al., (2016) showed that DE powder has a cylindrical shape and numerous pores which categorized DE as material with high surface area and high porosity. This phenomenon revealed that DE has a characteristic of Humidity Control Materials (HCM).

S. aureus commonly exists in the highest number in indoor settings (Hussin et al., 2011). *S. aureus* is a Gram-positive bacteria that grow in 'bunch of grapes' cluster, pairs and also can be found in form of clump. Normally, staphylococcal bacteria cause deep-seated infection, skin infections including boils, impetigo, pimples, hence it could be infection to eyelid or known as styes (Foster et al., 2015). *S. aureus* not only can cause infections to skin but people who get infected with the bacteria may get severe invasive infections including pneumonia, septic arthritis and osteomyelitis (Rao et al., 2018). *S. aureus* has been reported to cause nosocomial (originating in a hospitals) and community-associated (CA) bacterial infections in humans.

Oyster shells consist of 97.5% of CaCO_3 (Agbayani & Espisosa, 2006). After heat treatment was applied onto this powder, CaCO_3 is converted into CaO. According to Xing et al., (2013), CaO, MgO and ZnO exhibited strong antimicrobial activity. The study further reports that nano particles of CaO showed excellent anti-bactericidal activity towards *Staphylococcus epidermidis*, *P. aeruginosa* and *Candida tropicalis* (Xing et al., 2013) which are aligned with the results of this study. The authors illustrated that high shell powder concentration will significantly exhibits excellent antimicrobial behavior.

By referring the results of this study, CaO powder in oyster shells revealed that it could be used as anti-bacterial agents to kill gram-positive bacteria in indoor setting. However, heat treatment suppressed the ability of bactericidal activity of the ceramics, hence, reducing the efficiency of this material to kill the Gram-positive bacteria. Nevertheless, this study found that the inhibition zone was 22 mm, which shows that at the 50-30-20 ratio, the ability of the ceramics to kill gram-positive bacteria is existent. This makes it a suitable as ceramics with antibacterial characteristics.

Another study reported the antibacterial mechanism of CaO powder involved alkaline effects (Sawai et al. 2001). Thin water layer that forms around the oyster shell powder could be the reason for the antibacterial mechanism. Water may be adsorbed onto the oyster powder unintentionally during the grinding process or when exposed to environment. The pH of oyster shell powder (after heat treatment) was higher than its equilibrium state in solution. When the CaO are in contact with bacteria, the high pH in the thin surface water layer could damage the membrane, resulting in cell death.

For the strength test, three different loads were dropped on each ceramic at similar height and the tiles with formula 50-30-20 showed the greatest impact test when compared with the other three samples. The tiles with formula 80-10-10 illustrated as the weakest ceramics after three loads was applied. This can be seen when the waste glass and oyster powder was reduced to 10%. Meanwhile for formula 50-30-20, it contains 30% of waste glass and 20% of oyster powder. Waste glass powder was added as a refractory material. Refractory material has an ability to retain its physical shape after in high temperatures which is 1100°C is applied. In addition, silicon, sodium and calcium oxides that are known as soda-lime glass, was a main composition in most waste glass. This composition will be softened at 650°C to 750°C enabling the ceramic to form from the waste glass powder to vitrify at viscous-phase at sintering temperature (Haun et al., 2005). At this condition, the melted waste glass will bind with other components from DE and oyster shell powder.

In addition, the transformation of calcium carbonate in oyster shell powder to calcium oxide after heat treatment was applied will strengthen the ceramics. The structure of shell powder will be increased drastically when sintering temperature increases (Mohamad et al., 2016). Moreover, the irregular shape of calcined shell powder will bond with other components to form great aggregate, thus formed much immaculate surface (Mohamad et al., 2016).

This study had some limitations that needs to be considered. This study used salt solution in glass desiccator to obtain the specific relative humidity required. However, relative humidity recorded cannot be maintained for the expected duration of time as explained in methodology. As a suggestion, humidity chamber is the best instrument that can be used to replace this method. This instrument could give constant reading of relative humidity for a longer period and is a more physiologically ideal environment for simulation of real conditions as in indoor environment.

5. Conclusion

There is the need for the development of porous ceramics to help reduce relative humidity problems in Malaysian indoor spaces. This study found a suitable ratio for porous ceramics with humidity controlling ability and anti-bacteria characteristics which could be developed by using 50% modified DE. The modified

DE added with locally sourced and low-cost materials have the potential to solve the problem of humidity imbalance and microbial growth in indoor settings which can be used to solve problems related to sick building syndrome and ensuring productivity of occupants can be maintained.

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