objective of the study, to reduce the fired weight commercial wall tile of YTC and must meet with the standards.

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Local Diatomaceous Earth from Kapatagan, Northern Mindanao Philippines: A Micro/Nanoporous Siliceous Additive for Ceramic Wall Tile Production

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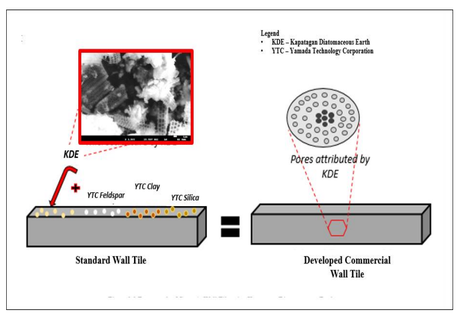
INTRODUCTION & AIM

Advancements in micro/nano mineral-based materials are driving innovations in sustainable ceramics. The Philippine archipelago is rich with inorganic materials like clay and non-clay minerals. Potentially, a diatomite raw material also known as diatomaceous earth is abundant locally in Mindanao Philippines , particularly at Kapatagan Lanao del Norte. Considering as a cheap source of silica, which also composed of siliceous skeletal remains of a fossil-like skeleton called diatoms [2]. The tangled particles of diatoms contain a high concentration of pores that made it lightweight and porous in nature. Also, it has a low density, permeable structure, and good adsorption properties [3,4] which leads towards various industrial applications.

Previous studies show the effect of diatomite to a ceramic body. An addition of raw and calcined diatomite to the art tile bodies leads to the increase of total shrinkage, water absorption, porosity (8.37%), and decreases the density (9.42%) with respect to the reference body [5]. Also, an addition of Spent Diatomaceous earth (waste after filtering process in brewery) as a pore-forming agent or silica carrier, on the properties of clay ceramic bricks was investigated. The result shows that addition of 1 to 5wt.% of SDE to clay matrix lead to the increase of open porosity and reduction of 6-12% of the bulk density compared to the controlled samples which only composed of clay, increase the water absorption up to 4-31%, also decreases the compressive strength ranges between 12-26% [6]. For that reason, this study explores the potential of locally sourced diatomaceous earth from Kapatagan, Lanao del Norte, Philippines, as a micro/nanoporous siliceous additive for improving the performance and sustainability of commercial ceramic wall tiles of Yamada Technology Corporation.

Figure B.1Map of Barangay Cathedral Falls, Kapatagan Lanao Del Norte, Philippines.
(from https://satellites.pro/Philippines_map#7.871321,123.779247,16)

Kapatagan Diatomaceous Earth (KDE)



METHOD

The starting raw materials used in the study includes clay, feldspar, quartz, and Kapatagan diatomaceous earth. The clay (binder), feldspar(flux) , and quartz (filler) were provided by Yamada Technology Corporation (YTC). On the other hand, the raw diatomaceous earth was taken from Barangay Cathedral Falls, Kapatagan Lanao Del Norte, Philippines.

Characterization of Raw Materials / Raw Material Individual Testing (KDE)
The chemical compositions of raw materials are determined using X-ray fluorescence (XRF) spectrometer model "Delta Professional Premium Handheld with silver alloy Anode, Olympus" with different modes. The phase analysis of Kapatagan diatomaceous earth (KDE) was undertaken by X-ray diffractometer (XRD) model "Miniflex 600, Rigaku" with CuKα radiation at 40 kV and 15 mA. The morphology of KDE was examined by scanning electron microscopy (SEM) model "JSM 7600F, JEOL". Also, the average pore size distribution of KDE was determined using Barret- Joyner-Halenda (BJH method), together with its specific surface area using Brunauer Emmet and Teller (BET) via automatic surface area analyzer (Autosorb iQ2 Quantachrome instrument) using nitrogen gas (N_2) adsorption and desorption isotherm. Also, standard procedure of YTC's individual raw material testing were conducted to determine the physical properties of KDE such as percent firing shrinkage, percent water absorption, percent weight loss, bulk density, and apparent porosity.

Preparation of YTC Standard Ceramic Slurry (SCS)
The Clay, Feldspar, and Quartz of Yamada Technology Corporation were dried in the oven for 100℃ removing completely the moisture. After the drying process, it was then weighed at their respective weight percent based on the calculated formulation and placed in a ball mill with the addition of 0.3-3% deflocculant to prevent settling of particles. A 40% water is added on the ball mill based on the desired total batch weight. To facilitate the mixing of the solution, one third alumina balls were used with respect to the volume of the ball mill. Afterward, the milling process then started with a rotation speed of 60%~70% to critical speed to ensure homogenization. After the milling process, the slurry was then screened through ranging from 32 to 28 mesh (500~600 microns) to remove organic matters and stored in a beaker.

Preparation of Kapatagan Diatomaceous Earth Slurry (KDES)
The KDE was dried in the oven for 100° C for 4 hours, removing the moisture completely. Crushing and pulverizing was no longer employed to preserve the microstructure of KDE. The slaking process was employed to improve the purity of KDE and to separate organic materials. The weight ratio between water and KDE is 1:1.5, respectively. Then the addition of 0.3-3% deflocculant is followed, preventing the particles to settle. The KDES was then homogenized by modified blunging machine for 4 to 6 hours. After homogenizing the slurry, it undergoes screening using 32~28 mesh (500 ~ 600 microns) and aged for 24 hours. The specific gravity of KDES was then targeted using pycnometer and hydrometer, ranging from 1.30-1.33 and 32-33 Baume respectively.

Slurry to Slurry Mixing
The addition of KDES to the SCS is performed by the following process. The moisture content of both KDES and the SCS was obtained using moisture content analyser of R&D laboratory, Yamada Technology Corporation. The dry weight of SCS is obtained and used as a basis for the amount of the dry weight of KDES that will be added in SCS. The obtained dried weight of KDES will be converted to wet weight of KDES. The slurries are then weighed at their respective mass ratio and mixed using a blunging machine to homogenize the admixture.

$$\text{Dry wt. of SCS} = \text{Wet wt. of SCS} \times \frac{(100 - \%\text{M. C. (SCS)})}{100}$$

$$\text{Dry wt. of Diatomite} = \text{Dry wt. of SCS} \times \%\text{Diatomite added}$$

$$\text{Wet wt. of Diatomite Slurry} = \frac{\text{Dry wt. of Diatomite}}{100 - \%\text{M. C. (D)}} \times 100$$

Where:
SCS – Standard Ceramic Slurry
Dry wt. of SCS - Dry weight of Standard Ceramic Slurry
Dry wt. of Diatomite- Dry weight of Diatomite
Wet wt. of SCS- Weight of Standard Ceramic Slurry
Wet wt. of Diatomite Slurry- Weight of Diatomite Slurry
%M.C (SCS)- Percent Moisture Content of Standard SCS
%M.C (D)- Percent Moisture Content of Diatomite

Dry Pressing and Sintering process
After homogenizing the admixture, the slurry was then placed on the pan and dried on the low-fired gas stove. The dried ceramic slip was cooled in room temperature and then transferred in polyethylene cellophane, kneaded with a little amount of water (7-10%), and underwent screening using 20 mesh to 14 mesh (1190 ~ 841 microns). Each of the prepared batch formulations (200 g) were then filled up to the brim of the metal mold and then pressed by a uniaxial hydraulic pressing machine available at Research and Development of Yamada Technology Corporation. After pressing the sample, it was then undergoing a fast-firing process in a furnace at 1235° C for about 50 ~ 60 minutes.

Physical Characterization
Firing Shrinkage and Weight Loss
Dimensions of the sample prior to firing are measured using calibrated Vernier caliper and analytical weighing balance and recorded. After sintering, dimensions of the samples were measured again. Then, the Percent firing shrinkage (% FS) and percent weight loss (%W.L.).

Water Absorption
The samples were suspended in a thermostatic water bath was boiled for about 5h ± 5min. The samples were then soaked for 24 hours and was wiped off lightly by a damp cloth and then weighed (M) [ASTM C373-14A].

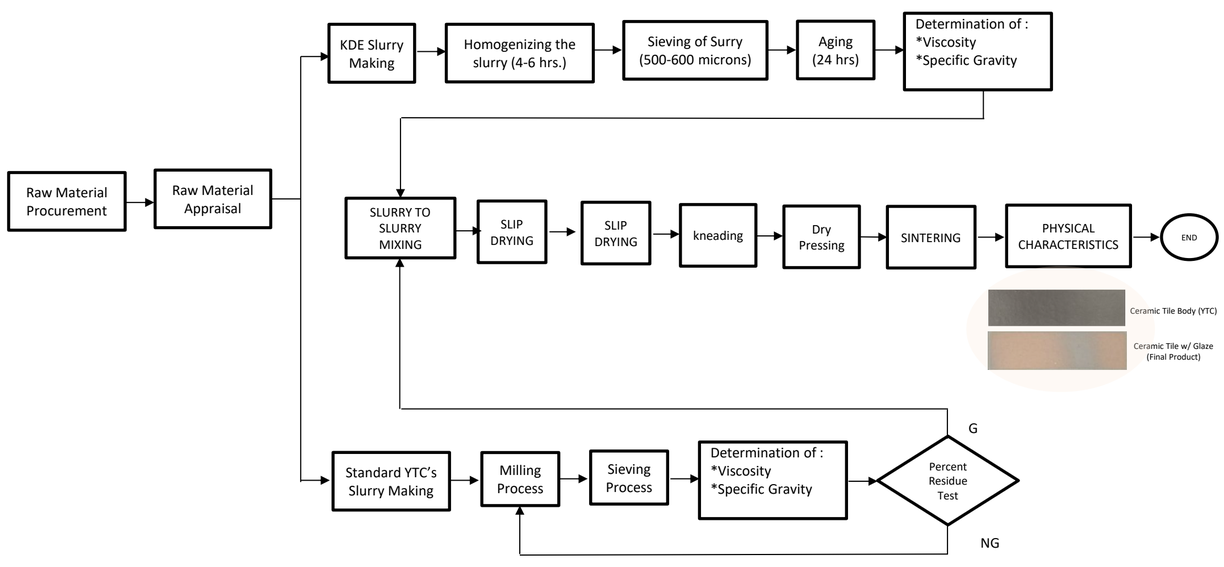
Apparent Porosity
The test bars were suspended in water (Archimedes Principle) and then weighed. The test bars were removed and carefully wiped off from their surface with a damped cloth and weighed [ASTM C373-14A].

Bulk Density
After boiling the sample for 5h±5min and determining the suspended weight as stated above the bulk density is then calculated [ASTMC373-14A].

Modulus of Rupture
Determination of compressive strength is essential as it can be used to assure the engineering quality in the application of building materials. The compressive strength of each fired samples from each batch was carried out in accordance with ASTM C648-04 using compression machine readily available in the R&D laboratory, Yamada Technology Corporation. The determined compressive strength of the fired samples was recorded for sample characterization.

Characterization of the fired sample
After testing the compressive strength of the fired samples, the broken pieces were collected and analysed by using Scanning Electron Microscopes (SEM) model "SU-8230, Hitachi" equipped with energy-dispersive X-ray spectroscopy (EDX). To identify the phase present in the sintered sample, the X-ray diffraction (XRD) model "Miniflex 600, Rigaku" with 40 kV CuKα radiation and 15 mA were used. Also, Thermo-Gravimetric and Differential Thermal Analysis (TG/DTA) model "TGA 60H, Shimatzu" used to determine the thermal behaviour of the sample specifically to identify the physical and chemical changes of the sample respectively. The samples were heated up at 1100 ℃ for 10° C per minute in an air environment.

PROCESS FLOW



RESULTS & DISCUSSION

After conducting the experiments, the gathered data were analysed, evaluated and discussed.

Physical Properties of the Kapatagan Diatomaceous Earth (KDE)
The Diatomaceous Earth of Kapatagan is being tested individually to know its physical properties.

Material	% Firing Shrinkage	Bulk Density	% Weight Loss	% Water Absorption	% Apparent Porosity
KDE	19.28%	0.92	33.84%	52.59%	48.79%

> As observed, KDE can absorb water half by its weight after the sample is fired at 1235° C and has high firing shrinkage. KDE also has a small bulk density (0.92), and apparent porosity of 48.79% with a weight loss of 33.84%.

Microstructure of Kapatagan Diatomaceous Earth (KDE)

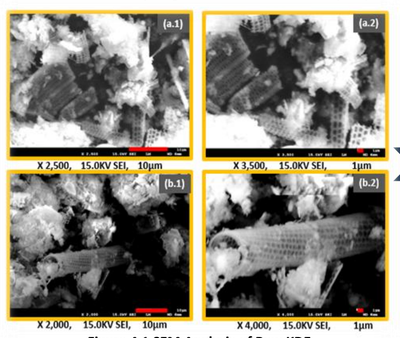


Figure 4.1 SEM Analysis of Raw KDE (Before Firing Process)

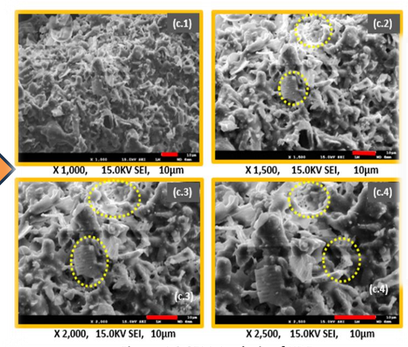


Figure 4.2 SEM Analysis of KDE (After Firing Process at 1150 ℃)

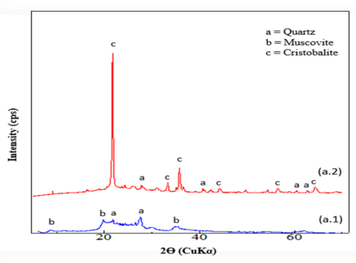


Figure 4.3 XRD Analysis of KDE (After Firing Process at 1150 ℃)

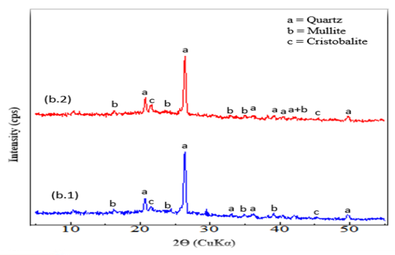
The porous tangled particles of raw KDE called diatoms are well preserved which composed of various shapes like ladders, and capsule-shaped frustules. On the other hand, the coalescence of KDE was observed as shown in Figure 4.2 after sintering at 1150° C. The porous diatom frustule tends to integrate , and various small pores of diatoms tend close [11]. This coalescence behavior of KDE particles after sintering at 1150° C, greatly affects its surface area and porosity. This was proven base on the Brunauer, Emmet, and Teller (BET) analysis using nitrogen sorption. The result shows that the surface area of KDE before the firing process was 5.878 m^2/g, with a major pore diameter of 1.9212 nm and below based on Barret-Joyner Halenda (BJH) analysis. However, after firing at 1150° C, the surface area of KDE decreases valued at around 0.3908 m^2/g with the major pore diameter ranges of 1.1262 nm and below.

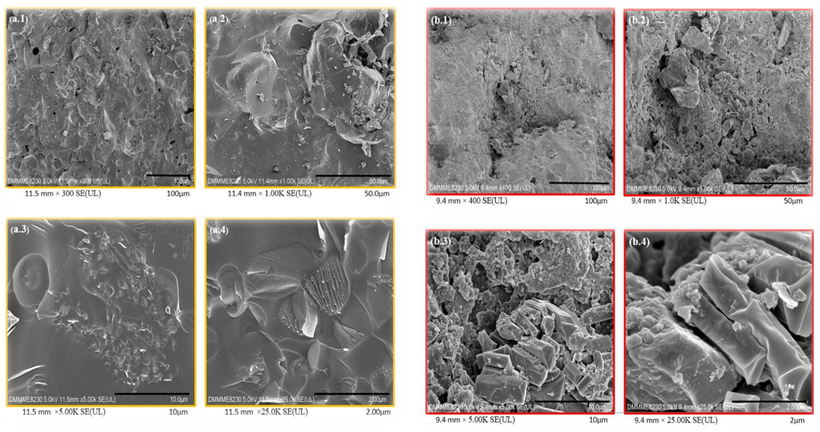
The X-ray pattern of KDE before and after the firing process (1150 ℃). The minerals that present on raw KDE are Muscovite-2M1 (96-110-0009) and Quartz (96-901-2603), the same phases found based on other studies [9]. Muscovite-2M1 ($Al_{1.32}Fe_{0.24}H_{1.28}K_4Mg_{0.32}Na_{0.32}O_{48}Si_{2.16}Ti_{0.08}$) having an amount of 79.0% and Quartz (SiO_2) having an amount of 21%, with unidentified peak areas of 3.4%. On the other hand, the crystalline Quartz (SiO_2) peaks increase up to 74.5% after the KDE was fired at 1150° C. Also, new peaks appear indicating the formation of cristobalite (96-101-0955) with an amount of 25.5% [10], with unidentified peak areas of 4.5%. The appearance of cristobalite phase occurs at a higher temperature (1470 ℃~1500 ℃). However, due to the eutectic reactions at 1150 ℃, which promotes the formation of the liquid phase in the presence of admixture of Al_2O_3, B_2O_3, and alkali earth oxides [9] in the KDE, the appearance of cristobalite at a lower temperature is possible.

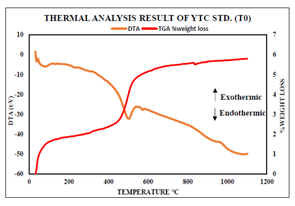
Actual Body Formulation
From the prepared raw materials, three formulations were made based on the standard body formulation of YTC. Formulation T3, T5, and T7 vary on the amount of Kapatagan Diatomaceous Earth Material (KDE) incorporated to the standard formulation of YTC, 3 wt%, 5 wt%, 7 wt% respectively based on the total solid loading. Considering the chemical analysis of the raw materials used, the actual formulations for each body have the proportion of RO/R_2O, R_2O_3, and RO_2 shown in Table 4.2

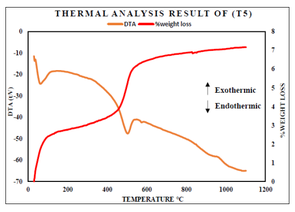
Raw Materials	T3 (+3 wt% KDE)	T5 (+5 wt% KDE)	T7 (+7 wt% KDE)
YTC Clay	30~50 %	30~50 %	30~50 %
YTC Feldspar	20~40%	20~40 %	20~40 %
YTC Silica	30~40 %	30~40 %	30~40 %
RO/R_2O, R_2O_3 and RO_2 ratio	6.01:22.95:66.22	6.09:22.78:66.33	6.16:22.62:66.43

Physical Properties	YTC std. (T0)	T3	T5	T7	Accepted Std.
%A.P.	0.61%	0.75%	0.80%	0.95%	–
%FS	9.23%	9.44	9.57	10.05	8.71- 9.96%
%W.A.	0.36%	0.45%	0.48%	0.57%	0-3 %
%WL	9.81%	12.20%	12.92%	13.19%	–
F.W. (grams)	72.06	71.26	66.92	64.55	–
MOR	6.76	6.55	6.41	6.03	>5.36 MPa
ρ	1.693	1.672	1.670	1.668	–









As observed in Table, the addition of KDE to the standard formulation of YTC tend to increase the firing shrinkage of each formulation. This may be attributed to coalescence behavior of KDE that was incorporated to the standard formulation of YTC, which the porous diatom frustule tends to integrate, and small pores of diatoms tend to close after sintering at 1235 ℃. Among the three formulations, T7 exhibits the highest percent firing shrinkage for about 10.05% and does not lie within the range of the accepted standard value of YTC, considering formulation T7 critical during production. Moreover, KDE may also consist of organic matter and some carbonates material, thus adding KDE to each formulation tends to increase the ceramic bodies total weight loss percentage. The low values of water absorption for all formulations were evident. Each formulation has a percent water absorption value of less than or around 0.5%, indicating a porcelain stoneware body [12,13,14]. Accordingly, at a temperature above 1050℃, water absorption is associated with liquid formation wherein it penetrates the pores, closing them and isolating neighboring pores. The liquid surface tension and capillarity help to bring pores close together and reduce porosity. These particles undergo a rearrangement that promotes densification and contraction of the internal structure, thus explains the low value of water absorption [15]. However, as the amount of KDE increases to each formulation, an increasing trend of percent water absorption and apparent porosity was evident. This may be the effect of KDE as a pore former [6]. Moreover, porosity is also correlated to bulk density and breaking strength of a ceramic material. Accordingly, the high sintering temperatures yield higher bulk density and is correlated to the low porosity obtained. During sintering, bulk transport will create a change in the interparticle spacing as neck growth takes place. This result in shrinkage, increased density, and higher strength for the entire powder compact [16]. However, as the amount of additives increases to each formulation, this leads to a slight decrease in bulk density as well as the breaking strength, which correlates to the increase of porosity. Among the three formulations, T3 and T5 are within the accepted standards physical properties of YTC. Although both formulations exhibit promising results, T5 shows slightly lower bulk density and does have lower fired weight compared to T3. For that reason, T5 considered as the optimum formulation among the three considering the main

CONCLUSION

The study shows that KDE is an effective additive to the YTC standard formulation which yields promising results. The following conclusions are drawn:

1. The addition of KDE tends to decrease the fired weight (F.W.), bulk density (ρ) and the modulus of rupture (MOR) of the ceramic wall tile. On the other hand, the firing shrinkage (%FS), water absorption (%WA), weight loss (%W.L.), and apparent Porosity (%AP) increase.

2. T5 is considered as the optimum batch formulation for ceramic wall tiles fired at 1235 ℃ firing temperature, which is 5% of KDE is added on the standard formulation of Yamada Technology Corporation.

3. The SEM images of formulation T5 revealed the formation of irregular pores such as interparticle porosity which affect the physical properties of the wall tile. The XRD analysis revealed quartz, mullite, and cristobalite as the main phase for both YTC std. and T5 formulation. TGA shows the total weight loss of T5 and YTC std. (T0). The formulation T5 (6.946%) has a higher total weight loss than YTC std. T0 (5.5955) due to the volatile matters that present on KDE which contributes to a total weight loss of ceramic wall tile.

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