

MUNA NOORI ISMAEL¹, HAZIM FALEH HASSAN¹, HADI SALMAN AL-LAMI^{2*}

¹Middle Technical University, Institute of Technology/Anbar, Al Fallujah, 31002, Anbar, Iraq ²University of Basrah, College of Science, Department of Chemistry, Al Basrah University, 61004, Basrah, Iraq

Abstract: Ceramics hold all traditional, scientific and engineering raw materials or fabricated products used in different applications especially that performed at high temperatures through hightempers. The remarkable progress made recently in the advancement of technology has related a prerequisite for a seriously huge number of parts with controlled porosity. In ceramic manufacturing, acquiring a porous item of such parameters and at the same time demonstrating appropriate mechanical strength isn't a simple issue. The powder-mixing system was utilized to fabricate kaolinsilica fired composites. The main purpose of the paper was to prepare ceramic bodies rich in pores with the goal that it will have a lightweight structure. Different wt.% of silica having distinctive twoparticle sizes are blended with kaolin, and afterwards, they shaped by semi-dry pressing in a hardened steel mould under 3-ton loads and sintered at four various temperatures, 900, 1000, 1100 and 1200 °C. Evaluating of composite's physical properties showed that a good level of porosity was obtained, and it was extended; especially in mixtures contain a coarse particle size. These results are enhanced by acceptable evident comes from good apparent solid density and good strength.

Keywords: lightweight, composites, ceramic, impact strength

1. Introduction

Ceramics find applications in various fields. There are applications for natural and synthetic ceramics, and there are traditional and engineering applications. Ceramic products can be categorized into those utilized at ordinary temperatures, and these categories may then be subdivided into those items which have a permeable body after sintering and those which are nonporous. The previous items additionally have a present name as lightweight earthenware production, and as brought up by Ashby [1].

Lightweight ceramics permit the simultaneous optimization of stiffness, strength and overall weight. As these materials represent a rather new class of ceramics, it is reasonable to expect they may have a range of potential applications that are not fully explored [2]. Apart from their traditional use as refractories because of their superior thermal shock resistance, porous ceramics have found wide-spread of applications, such as in the filtration of liquids and gases [3], absorption of shock, catalyst support molten-metal filtration, thermal insulators, high-temperature applications, and environmental protection [4].

The fabrication of lightweight ceramics can be relatively straightforward, for instance by sintering particles together to the desired density or by foaming a constituent slip. The main problem has been to produce materials having both good strength and a high level of porosity [5,6]. As in other brittle materials, the attainable strength is generally determined by the pore sizes and their distributions [7].

In previous works, controlled porous ceramics, based on kaolin, were prepared using different additives as foaming agents. Natural and synthetic polymer powders were added as part of the original mixtures [3], whereas different metals should have been synthetically swollen by a dilute solution of sodium hydroxide to fuzz the blends [8]. In both cases, ceramics with a high level of porosity and good strength were obtained. But, unfortunately, there were some restrictions in using these additives

^{*}email: hadisalman54@yahoo.com



because few of them might have a deleterious effect on the microstructure of the ceramic body as shown by scanning electron microscope.

To maintain avoiding any of those issues, the powder-mixing method was utilized to create claybased composites having a sensible degree of porosity to be called lightweight earthenware production, and simultaneously leaving their surface and microstructure, as we can say, is perfect. We proposed to take a look at the impact of the particle size of strengthening samples during the sintering stage of the dried ceramic bodies. This results in composite bodies should be stacked in pores, with the objective that it will have a lightweight property to suit different applications.

The current attempt in the fabrication of lightweight ceramic composites is not only toward producing a narrow pore-size distribution, as has been attained with other methods, but also to lower their specific densities and accompanying with good mechanical properties.

2. Materials and methods

2.1. Raw materials

Kaolin and silica are abundant materials in Iraq, especially in the western desert with Al-Ratba depression. They were sieved using Podemorse sieving machine No. 15104/ England. For kaolin, the fraction passed through the sieve of 75 µm and retained on the 63 µm sieve, was taken to be used in this work. Its chemical analysis is shown in Table 1.

Table 1. Chemical composition analysis of kaolin.										
Oxide	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	Na ₂ O	K ₂ O	Trace	L.O.I.
						-				
%	47.3	35.1	0.84	1.22	0.71	0.3	0.42	0.51	0.74	12.86

able 1 Chamical composition analysis of leading

Silica powder was also sieved, after washing with water several times. Two fractions were taken and termed as coarse and fine silica particles, i.e., those with particle sizes of 150-180 µm and 63–75 μm, respectively. They were also chemically analyzed and gave the results illustrated in Table 2.

Table 2. Chemical composition analysis of the and coarse sinca.									
Type	Oxide	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	L.O.I.		
Fine	%	98.37	0.46	0.04	0.23	0.60	0.30		
Coarse	%	98.22	0.52	0.05	0.20	0.71	0.30		

Table 2 Chemical composition analysis of fine and coarse silica

2.2. Preparation of Specimen

Specimen preparation is of great importance in the final properties of the prepared ceramic materials. Many samples composed of a different weight percent of kaolin and silica having two different particle sizes were fabricated using a semi-dry pressing technique with a hydraulic press type Shimadzu Corporation/Japan. All samples were pressed in a cylindrical form having about 30 mm diameter and 5 mm height, using a stainless steel mould. The 3-ton forming load was used because it was found to be the best pressing load for this type of ingredients [3]. The conditions used are shown in Table 3.

2.3. Drying and Firing of the Pressed Specimen

Samples were dried first at room temperature for 48 h, then at circulating oven for at last 10 h. This gave sufficient opportunity to drying of the samples to avoid vapour evolution into the samples due to the moisture content. The dried samples were fired at four several temperatures, 900, 1000, 1100 and 1200 °C in a Carbolite furnace.



		1	1 0			
Sample	Kaolin			Silica	Forming	Firing Temperature
Code	%	Particle Size	% Particle Size		Load (ton)	(°C)
		(µm)		(µm)		
PM1	70	63-75	30	63-75	3	900,1000,1100,1200
PM2	50	63-75	50	63-75	3	900,1000,1100,1200
PM3	30	63-75	70	63-75	3	900,1000,1100,1200
PM4	70	63-75	30	150-180	3	900,1000,1100,1200
PM5	50	63-75	50	150-180	3	900,1000,1100,1200
PM6	30	63-75	70	150-180	3	900,1000,1100,1200

	O 11.1	C 1	•	1 .	C' 1	•1•
Table 3	('onditions	of prepared	ceramic sami	กโคร เมราทด	tine and	coarse silica
Lanc J.	Conditions	or propurou	coranne sam	JICS USING	mic and	course sinca.

2.4. Determination of Physical Properties

2.4.1. Determination of Linear Shrinkage

Linear shrinkage was determined by utilizing a micrometre to quantify the dimensions of each sample (thickness or diameter) before and after firing. It was calculated as %L. Sh.

2.4.2. Measurements of Porosity, Water Absorption, and Apparent Solid Density

Since porosity is the essential physical property which must be controlled during the handling of lightweight composite bodies. The standard test method (ASTM C20-83) has been used for measuring the level of porosity. It was also used to measure water absorption and apparent solid density using boiling water [9].

2.5. Determination of Mechanical Properties

The investigation of the mechanical properties of samples was essential for their use in various applications. The fracture strength in compression and impact are among the primary properties examined.

2.5.1. Brazilian Disc Fracture Test

This sort of test is normally utilized when traditional tension is difficult to carry out due to the brittle nature of the test material [10]. This test was performed on sintered discs having about 29.5 mm diameter and 5 mm height, using compression test device Instron 1195. The samples were fixed between upper and lower plates to start compression at a rate of cross-head speed 5 mm/min until sample failure. By then, composites fracture strengths in MPa were determined.

2.5.2. Impact Strength Test

The impact strength was measured by the Izod system. In this method, a pendulum arm was dropped through a definite arc onto the centre of a specimen to break it. Another test method is to escalate the pendulum strength gradually until the specimen ruptures. This has a benefit that the associated lost strength is diminished, and its remaining energy is estimated by its ascent on the opposite side [11].

3. Results and discussions

Different mixtures of kaolin and silica were prepared. The ratio of the components in the mixtures varies according to the desired properties of the ceramic product. The high content of the clay component in a mixture is necessary for the product which is to have prevalent mechanical quality and all the while great shaping properties [12].

The making of pore framework, porosity control, and consequently different properties was concentrated right now depending on the powder blending method of an alternate weight percent of kaolin and silica, and distinctive particle size of the latter constituent. The mixtures are illustrated in Table 3 and they were referred to as PM1, PM2, and PM3 for fine silica particle (particle size of 63-75 μ m) and as PM4, PM5, and PM6 for coarse silica particles (particle size of 150-180 μ m).



Figure 1 shows the linear shrinkage increases with the increase of sintering temperature. This may be attributed to the effect of high temperature on the volume of material prompting better densification of particles. This figure additionally shows that the shrinkage diminishes with expanding silica content, which known to be the non-plastic material [4,12]. An increase in viscosity of silica liquid and the growing volume of the dense particles led to a decrease in the linear shrinkage. This can be seen very clearly for mixtures PM4, PM5, and PM6 in Figure 2. From these results, we can conclude that an increase in the silica content, from fine particle size to coarse particle size, in ceramic samples, reduces the ability of ceramic body shrinkage. A similar conclusion was made by Al-Uqily [13] in his investigation on the manufacturing of fire-clay refractory brick from flint clay.



Figure 1. Linear shrinkage of fabricated ceramics fired at different temperatures using fine silica.



Figure 2. Linear shrinkage of fabricated ceramics fired at different temperatures using coarse silica.

Knowing the level of porosity of a fired body is of importance for its processing as an extension of creating and allows the evaluation of an earthenware item for a particular application [3,4]. Utilizing two silica particle sizes in various proportions with kaolin particles was the key point for cumulative the degree of porosity, and this was the situation. This might be attributed to the melting of certain oxides present in the selected raw materials used, Tables 1 and 2, which may act as fluxes and in the early stages of the firing process. This was genuine for its corresponded property, water absorption,

Rev. Chim., 71 (5), 2020, 65-74



Figures 5 and 6. The same conclusion was made by Dobra et al [14] on their study on the effect of the metallic impurity accumulation on the surface of alumina hydrate particles, during the sodium aluminates decomposition in liquid phase in the crystallization stage of aluminium hydroxide in the Bayer technology.



Figure 3. Apparent porosity of fabricated ceramics fired at different temperatures for powder mixed with fine silica



Figure 4. Apparent porosity of fabricated ceramics fired at different temperatures for powder mixed with coarse silica



Figure 5. Water absorption of fabricated ceramics fired at different temperatures using powder mixed with fine silica





Figure 6. Water absorption of fabricated ceramics fired at different temperatures using powder mixed with coarse silica

The enhancement of sintering in the reduction of ceramic body volume causes better densification [15]. This was positively reflected in the apparent solid density of the body. However, the apparent solid density is directly proportional to the firing temperature, in other words, an increase in the firing temperature led to an increase in density, as shown in Figures 7 and 8, and these results in good agreement with the finding of Stanescu et al., on their preparation of the autoclaved aerated concrete (AAC) [16]. Their results showed that there was about a 10% increase in the apparent density of the AACs prepared.



Figure 7. The apparent solid density of fabricated ceramics fired at different temperatures for powder mixed with fine silica





Figure 8. The apparent solid density of fabricated ceramics fired at different temperatures for powder mixed with fine silica

Relating blends assigned as PM1and PM4, one can observe that changing the silica powders from the particle size of $(63-75 \ \mu m)$ to $(150-180 \ \mu m)$ and keeping the weight % amount fixed, directed to an increase in the apparent solid density, even though the clay body had the most significant level of porosity. This can be credited to better compaction was acquired, and that the kaolin fine particles may act as co-binder for the coarse silica particles. Almost the same conclusion was made in the processing of glass filters from crushed soda-lime glass using two different particle sizes of sieved glass powders [3].

The easiest and most advantageous techniques for mass determination of the characteristic of porous or lightweight ceramics are those rely on their density and strength [1,3]. These methods make it possible to control the homogeneity of the end products. The results obtained for the diametrical compression strength with the aid of the Brazilian disc fracture test are shown in Figures 9 and 10 for all-ceramic bodies fabricated in this work.



Figure 9. Compressive strength of fabricated ceramics fired at different temperatures for powder mixed with fine silica





Figure 10. Compressive strength of fabricated ceramics fired at different temperatures for powder mixed with coarse silica

The fracture strength, as expected, was increasing with firing temperature increase. This might be because of that of fluid silica produced during firing and by the liquefying activity of the transitions oxide present act as fluxes in the raw materials. The chemical analysis of the raw materials showed the presence of K_2O , Fe_2O_3 , CaO, MgO, etc. These liquefied fluids may consume the spaces or voids present between ceramic particles in certain phases of the firing procedure. This suggests with rising firing temperature both sinter necks and agglomerated particle contact focuses would be relied upon to be abundant and tougher [14]. However, reduction in low bearing cross-section area and the presence of pores tend to lower the compressive strength. This was also true for impact strength, especially when the silica powder was changed from fine particle size to coarse particle size, as shown in Figure 11.



silica and mix PM4 of coarse silica

Nearly, in the same line, Nergis et al. investigated the effect of mixing aggregates on the fly ashbased geopolymers, and it was analysed from the structure and mechanical properties point of view. They found that fly ash with 70 % by mass aggregates presents the porous structure, and strongly influences the density, compression strength and flexural strength comparing to unmixed fly ash [17].



4. Conclusions

Method for mixing and characterizing the ceramic composites has been presented. Powder-mixing technique appears to be the most versatile method of fabrication of lightweight ceramics because no other additive was needed to create pores in the fired ceramic body. The porosity was controlled by changing the silica powders from the fine particle size of $(63-75 \ \mu\text{m})$ to coarse particle size (150-180 $\ \mu\text{m}$), led to an increase in the level of porosity reach about 40% accompanying with a good apparent solid density 2.13 g cm⁻¹ at higher firing temperature (i.e. 1200°C), and this is positively reflected on their compressive and impact strength and impact. This certainly leads to an increase in the field of fabrications and the functional utilization of the lightweight earthenware production since there is no additional foaming mediators are used.

Powder mixing technique is found to be capable of distinguishing between a different number of mixtures having various particles size of kaolin and silica. This appears to be the most convenient technique for generating pores in the ceramic body without adding foaming agent. The most important results demonstrated in this work that ceramic composites having both a good level of porosity and good strength, compressive and impact, were fabricated.

References

1. ASHBY, M.F., The Mechanical Properties of Cellular Solids, *Metal. Trans.* A, **14**(9), 1983, 1755-1769.

2. MOHAMMAD, M.S., AGHDAM, K.H., MAHMOOD, M.J., Effects of Nano-Sized Ceramic Particles on the Coefficients of Thermal Expansion of Short SiC Fiber-Aluminum Hybrid Composites, *J. Alloys Compd.*, **803**(30), 2019, 554-564.

3. AL-LAMI, H.S., SAKHARIA, SH.A. 2002, Iraqi Patent No. 2966.

4. HUA, K., A. SHUI, L. XU, K. ZHAO, Q. ZHOU, XI, X. Fabrication and Characterization of Anorthite–Mullite–Corundum Porous Ceramics from Construction Waste, *Ceram. Int.*, **42**(5), 2016, 6080–6087.

5. OBADA, D., DODOO-ARHIN, D., DAUDA, M., ANAFI, F., AHMED A.S., AJAYI, O.A., The Impact of Kaolin Dehydroxylation on the Porosity and Mechanical Integrity of Kaolin Based Ceramics Using Different Pore Formers, *Results in Physics*, **7**, 2017, 2718-2727.

6. SUCHORAB, Z., BARNAT-HUNEK, D., FRANUS, M., ŁAGÓD, G. Mechanical and Physical Properties of Hydrophobized Lightweight Aggregate Concrete with Sewage Sludge, *Materials (Basel)*, **9**(5), 2016, 317-334.

7. KUROVICS, E., GOMZE, L.A., Development of High-Tech Ceramic Composites from Conventional Kaolinite Minerals and IG-017 Additives, Multi-Science-XXXI. Micro CAD International Multidisciplinary Scientific Conference, University of Miskolc, Hungary, April 2017.

8. AL-LAMI, H.S., Chemical Swelling of Aluminium Metal Powder in Processing of Controlled Porous Ceramics, *J. Sci. & Eng.*, 3, 2005.105-115.

9. ASTM Annual Book of Standards, 15(01), 1989.

10. TVERGAARD, V., NEEDLEMAN, A.. An Analysis of Thickness Effects in the Izod Test, *Int. J. Solid and Struct.*, **45**(14-15), 2008, 3951-3966.

11. PERRAS, M.A., DIEDERICHS, M.S., A Review of the Tensile Strength of Rock: Concepts and Testing, *Geotechn. Geolog. Eng.*, **32**(2), 2014, 525-546.

12. AHMAD, R., MOHD A., ALBAKRI, M., Review on Development of Clay Based Geopolymer Ceramic Composites, *Mater. Sci. Forum*, **803**, 2015, 37-43.

13. Al-Uqily, R.A., Industry of Fire-Clay refractory Bricks from Flint Clay, M.Sc. Thesis, University of Technology, Department of Chemical Engineering, 1997, Iraq.

14. DOBRA, G., ILIEV, S., ANGHELOVICI, N., COTET, L., FILIPESCU, L., Impurities Accumulation on the Surface of Alumina Hydrate Particles in Bayer Technology, *Rev. Chim.*, **70**(2), 2019, 355-360.



15.KINGRY, W.D.; BOWEN, H.K. AND UHLMANN, D.R. 1978. Introduction to Ceramic, 2nd Edition, John Wiley & Sons, New York.

16.STANESCU, G., BADANOIU, A., NICOARA, A., VOICU, G., Brick and Glass Waste Valorisation in the Manufacture of Aerated Autoclaved Concrete, *Rev. Chim.*, **70**(3), 2019, 828-834. 17.NERGIS, D., BURDUHOS, D., VIZUREANU, P., CORBU, O., Synthesis and Characteristics of Local Fly Ash Based Geopolymers Mixed with Natural Aggregates, *Rev. Chim.*, **70**(4), 2019, 1262-1267.

Manuscript received: 01.12.2019