

Preparation of porous cordierite ceramic using a silica secondary resource (silica fumes) for dust filtration purposes

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Cordierite-based porous ceramic samples were successfully prepared using a mixture composed of waste silica fumes, bauxite and talc via a polymeric sponge replica technique. Firstly, the optimum sintering conditions (in terms of sintering temperature and time) for acquiring as much of the cordierite phase with the absence of undesirable ones were determined for a dense mixture compact. The results revealed that a sintering temperature of 1350 °C and a sintering time of 2 h were the most appropriate conditions during cordierite synthesis. Secondly, the key role for the successful fabrication of a porous ceramic body via replica method is to prepare a well-dispersed suspension from the mixture of raw materials. Here the optimum conditions for preparing a well-dispersed suspension in terms of type and concentration of additives as well as suspension pH were evaluated. The using of 0.2% sodium silicate and a suspension pH of 8 were found to be the most favorable conditions for obtaining a well-dispersed suspension. Thirdly, in order to prepare porous cordierite, polyurethane foam was impregnated with a well-dispersed suspension and sintered at the optimum conditions conducted for a dense cordierite sample. The porous cordierite produced was found to possess 46.4% total porosity. The porosity was comprised of large and small interconnected pore surrounded by a web of ceramic. This structure is suitable for applications in filtering purposes.

Key words: Silica fumes, Cordierite, Porous structure, Rheology, Well-dispersed suspension, Replica method.

Introduction

The presence of porosity in a material is often viewed as problematic. However, there are many applications in which the use of porous materials can be advantageous or even necessary, for example in filters, membranes, catalytic substrates, thermal insulation, gas burner media, and as refractory materials. In these applications, materials are chosen for their special functional properties, such as low thermal conductivity, high permeability, high temperature stability, excellent thermal shock resistance, or low dielectric constant.

Generally, porous ceramics are usually understood as a materials having porosity over 30% [1]. They could be considered as systems that consist of a solid substance that forms walls between pores (a skeleton) and air that fills the pores. The composition, pore characteristics, and crystal structure are the main parameters determining the thermophysical properties of porous ceramics [2]. Various processing routes have been applied in manufacturing porous ceramics and they have recently been reviewed by Studart *et al* [3]. These methods include; replica, sacrificial template and direct foaming methods while many other techniques are being developed. In contrast to these various processes, where a change of many parameters leads to different types of product from the same starting

materials, in the polymeric sponge replica method the final product can be affected only by changing the viscosity of the ceramic suspension [4]. When the viscosity is low, the resulting product is a positive replica of the polymeric sponge used and vice versa. This fact makes the polymeric sponge replica method, the most popular and widely-used technique in manufacturing porous ceramics [5]. However, the rheology of the ceramic suspension has a key role in deciding the success using this technique in fabricating porous ceramic bodies.

Porous thermostable ceramic materials with a low temperature coefficient of linear expansion (TCLE) are required to fabricate refractories, thermal insulation, catalyst supports and filters for hot liquid gases used in industry [6]. One such material is a cordierite-based ceramic. Cordierite is known to display very low thermal expansion as an attribute of an excellent thermal shock resistant material when it is subjected to rapid and sever changes in temperature [7, 8]. There is no doubt that this material is one of the potential ceramic filter material in meeting the demand of highly efficient particulate removal from a hot gas stream in a number of industrial process plants. These includes preparation of combustion off gases for reuse across secondary turbines [9], removal of particulates from hydrocarbon processing gas streams above the dew point, filtering of gases from incineration of reuse and disposal facilities [10], removal and/or recovery of catalyst from off gases, etc. Numerous techniques have been mentioned in the literature for the synthesis of cordierite, such as, crystallization from glasses, growing of single crystal, a sol-gel technique, combustion synthesis or a solid

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phase reaction [11]. Although the first four processes permit one to obtain pure cordierite at low temperatures, they can not be applied yet on an industrial scale due to the high cost of the reactants used and the relative complexity of the technological procedures [12]. Sintering of oxide powder through a solid state reaction has been regarded as the most-widely used process in the synthesis of cordierite, especially in large scale production. A large number of investigations have dealt with using of such process in the synthesis of cordierite with the using of various starting materials or even from analytical grade oxide powder [13-20]. Meanwhile, using secondary resources or waste materials can bring a higher output of cordierite at lower costs than for traditional processes, and make a break-through in raw materials as well as in technology [21]. However, various secondary resources and waste materials such as Al slag, Al-rich anodizing sludge, fly ash and rice husk ash were successfully utilized in synthesizing cordierite materials [22-25]. To the best of our knowledge, no reports regarding the using of silica fumes (produced as secondary raw materials during ferro-chrome silicon alloy manufacturing) in synthesizing of cordierite has been reported so far.

Here we mainly discuss the possibility of using silica fumes as a silica source during cordierite synthesis. The effect of both sintering temperature and sintering time on the reaction progress between the different constituents of the mixture comprised of silica fumes, bauxite and talc were investigated. Also, a rheological study was conducted in order to determine the optimum conditions to obtain a well-dispersed suspension from this mixture. This study was performed as a function of the type and concentration of the dispersion agent and the suspension pH. Once the sintering conditions and the conditions for obtaining a well-dispersed suspension were optimized, a porous cordierite was prepared by a polymeric sponge replica method. The properties of the porous cordierite produced were evaluated in terms of the total porosity, pore shape and pore size distribution.

Experimental and Characterization

Experimental

Silica fumes (delivered from the Ferro-Chrome Silicon Alloy Company, Egypt), bauxite (supplied by the El-Alamia Company for refractory production, Egypt) and talc (delivered from the Eastern Desert of Egypt (Shalatin area)) were the raw materials used in this investigation. Based on the composition of cordierite ($2\text{MgO} \cdot 2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2$) and the chemical composition of the starting materials (as shown in Table 1), a mixture of bauxite, silica fumes and talc in proportions equal to the stoichiometric cordierite composition were prepared. In the beginning all the raw materials were subjected to grinding to pass through a 74 μm sieve. Then a mixture according to the stoichiometric cordierite composition were prepared and thoroughly mixed before subjecting to compression

Table 1. Total chemical composition of starting materials

Components	Chemical composition, %		
	Silica fumes	Bauxite	Talc
SiO ₂	94.45	6.17	62.7
Al ₂ O ₃	1.46	87.98	0.36
MgO	0.44	0	30.94
Na ₂ O	0.59	0	0.1
K ₂ O	0.66	0	0.04
P ₂ O ₅	0	0	0.044
CaO	0.36	0.19	0.54
Fe ₂ O ₃	0	1.75	0.19
TiO ₂	0	3.52	0
MnO	0	0	0
L.O.I	0	0.26	4.99

under a pressure of 490 MPa via a uni-axial press for green compact formation. The green compacts produced were sintered at different sintering temperatures (1250-1350 °C) and different sintering times (2-6 h) at a constant heating rate of 5 °C/min. The effect of different temperatures and times on the formation of the cordierite phase as well as the physical properties of the sintered compacts was evaluated.

The preparation of a well-dispersed suspension is a key factor in producing porous ceramics using a replica method. Fine ceramic powders suspended in a liquid tend to aggregate due to the attractive Van der Waals forces. These interparticle forces need to be overcome in order to obtain a good dispersion to successfully slip cast. This goal is usually achieved by adding to suspensions a suitable type and amount of a dispersing agent. Surfactants are commonly used as dispersants. Sodium silicate is regarded as the most popular dispersion agent used in slip casting for many years. On the other hand, aluminum mono-phosphate is usually applied in the refractory industries in order to increase the strength of the product. Accordingly, a study concerning optimization of the parameters controlling the suspension rheology, i.e. obtaining a lower viscosity suspension, was made. The parameters studied were the type and concentration of the additives as well as the suspension pH. Two types of additives were examined: as dispersion agent; a local type sodium silicate (56.15% solid content, SiO₂/Na₂O ratio 2.67/1 and 1.6 g/cm³ specific gravity) and as a binder; aluminum mono-phosphate delivered from the El-Naser Company, Egypt. Firstly, homogenization of an aqueous suspension from the mixture in the proportion given above using a high shear mixer for 30 minutes was carried out. Then the rheology of the suspensions was studied for a constant solid loading of 40 wt.% by changing the other dispersion parameters.

For porous cordierite fabrication, a polymeric sponge with an average porosity of 2-5 pores/cm was impregnated with well-dispersed suspension mixtures. The sponge was compressed to remove air, immersed into the suspension

and then allowed to expand. This compression-expansion step was repeated several times. Then the infiltrated sponge was dried at 100 °C to deposit the ceramic particles in the sponge. The dried structure was heated in air at 350-800 °C to volatilize the polymeric sponge. Slow controlled heating was desirable to avoid blowing the ceramic structure apart. Finally the structure was sintered at the optimum conditions stated for dense cordierite.

Characterization

The total chemical compositions of the starting materials were determined using a Philips PW 1480 wavelength dispersive X-ray fluorescence (XRF) spectrometer. Phase identification of samples sintered at different temperatures and time were performed at room temperature by X-ray diffraction (XRD, Bruker axs D8, Germany) with Cu-K α ($\lambda = 1.54 \text{ \AA}$) radiation and a secondary mono chomator in the 2θ range from 10 to 50°. The bulk density and apparent porosity for a dense sintered compact were determined by the Archimedes immersion technique, whereas, the linear change of sintered samples were determined conventionally [26].

Electrophoretic measurements were performed for suspensions with and without dispersion agent at different pHs using a Zeta potentiometer (Coulter Delsa 440 sx, USA) and a solution of 0.001 M KCl as the background electrolyte. The suspension pH was varied from 4 to 10 using 0.01N HCl and 0.01N KOH. The rheological properties of the suspensions were evaluated with a rotational stress controlled rheometer (Bohlin instrument C-VOR, Bohlin Instruments Ltd, UK). The measurements were carried out at room temperature (25 °C) using a cone and plate configuration. A pre-shearing was performed at a higher shear rate (100 s^{-1}) for 1 minute before the measurements, followed by an equilibrium time for 30 s. The flow curve was obtained at a controlled rate condition with a linear increase in the shear rate from 0 to 100 s^{-1} in 200 s. The degree of porosity as well as the pore size distribution of the porous cordierite ceramic (the sintered porous body after the replica technique) was determined by Hg-intrusion porosimetry (Poresizer 9320, Micrometrics, USA). The pore structure was characterized from the polished cross-sections of porous sintered specimens by a scanning electron microscope (JEOL-JSM-5410).

Results and Discussion

Effect of sintering temperatures

X-ray diffraction patterns shown in Fig. 1, illustrate the effect of different sintering temperatures on the phase composition of the sintered compact.

The X-ray diffraction patterns of sample fired at 1250 °C, indicates the presence of α -cordierite (JC PDS # 89-1485) as the main phase along with a significant amount of tridymite (JC PDS # 71-0197), MgAl_2O_4 spinel (JC PDS # 82-2424), proto-enstatite (JC PDS # 11-0273) and corundum (JC PDS # 46-1212). Meanwhile, some of these

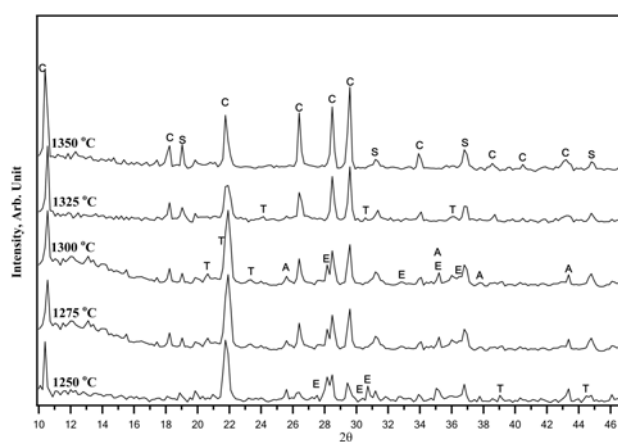


Fig. 1. X-ray diffraction patterns of samples treated at different sintering temperatures.

phases disappeared upon increasing the sintering temperature with an increase in the formation of others.

The detailed analysis of the obtained X-ray patterns has shown the following:

- At a sintering temperature of 1250 °C many phases were detected and the amount of the α -cordierite is still low. On the other hand a larger amount of proto-enstatite with a noticeable amount of tridymite, corundum and spinel was detected. This result indicates that at this sintering temperature the solid state reaction has started with the development of only a fraction of the characteristic peaks of cordierite phase.

- With an increase of the sintering temperature to 1275 °C it could be noticed that the amount of the proto-enstatite is slightly deceased. Meanwhile, a slight increasing of the spinel and tridymite phases is clearly observed. This phenomenon was repeated with an increase in the sintering temperature to 1300 °C. A further decrease in the proto-enstatite phase with the formation of more cordierite phase as well as spinel and tridymite was observed. This behavior is attributed to that with an increase in the sintering temperature the proto-enstatite phase is decomposed. This leads to the formation of Mg-Al species like spinel as there is enough alumina in the mix to allow its formation. Also with the presence of enough silica in the system it leads to the formation of more cordierite phase. Also it could be easily noticed that the peak characteristic for tridymite ($2\theta = 21.6$) is largely interfered and overlapped with one of the characteristic peaks of cordierite which explain the higher intensity of this peak than expected for the pure cordierite phase.

- With a further increase in the sintering temperature (to 1325 °C) the tridymite phase was greatly decreased and the complete disappearance of the proto-enstatite phase was detected.

- At 1350 °C almost all detected peaks are characterized for both cordierite and the spinel without the detection of any of undesired phases (proto-enstatite and tridymite).

This study revealed that a sintering temperature of 1350 °C is necessary to achieve a high percentage of the

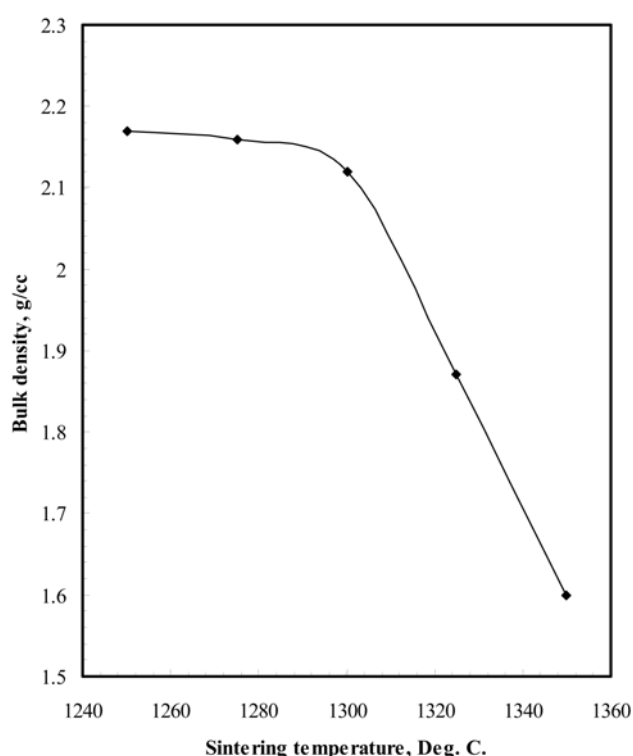


Fig. 2. Effect of sintering temperature on the bulk density of the compacts.

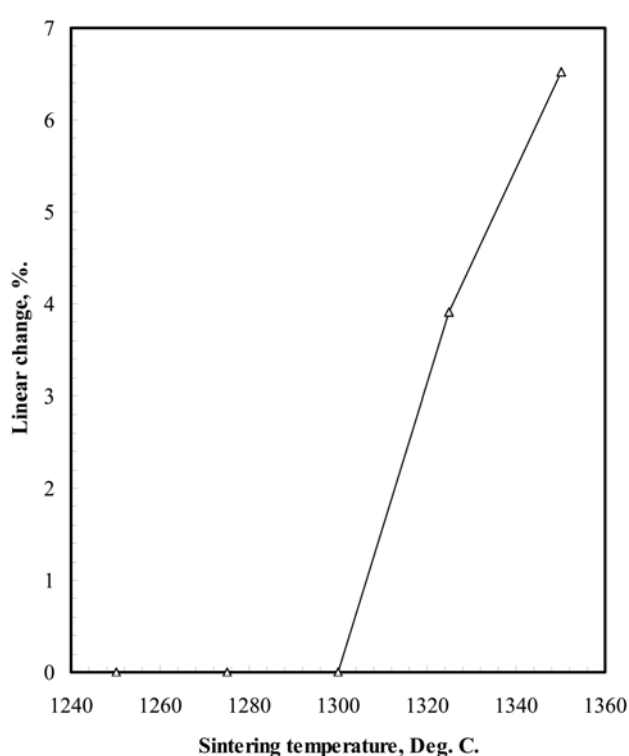


Fig. 4. Effect of sintering temperature on the linear change of the compacts.

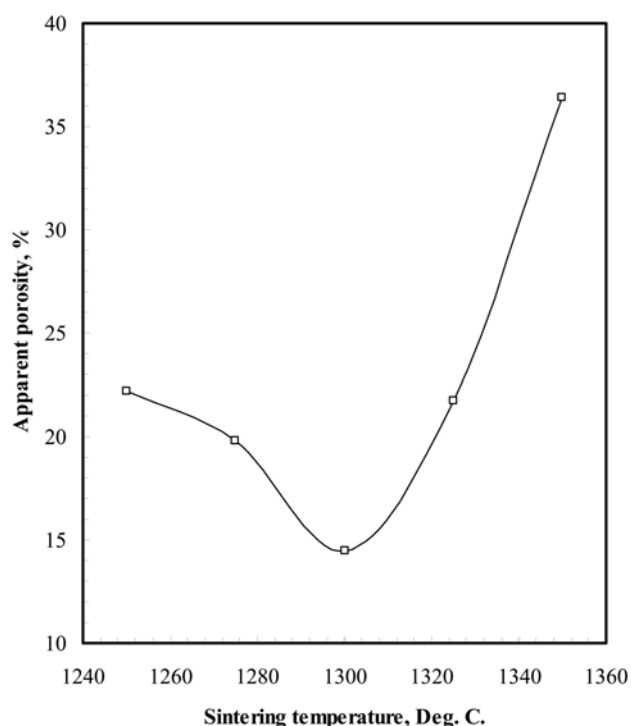


Fig. 3. Effect of sintering temperature on the total porosity of the compacts.

cordierite phase without the formation of the undesired phases (tridymite and proto-enstatite).

The physical properties of compacts sintered at different temperatures are shown in Figs 2-4. It was found that

the bulk density of the compacts is slightly decreased with an increase of the sintering temperature up to 1300 °C. Beyond this temperature a sharp decrease in the bulk density was clearly observed. Meanwhile, the total porosity was found to decrease upon increasing the sintering temperatures until it reaches a minimum at 1300 °C, then it increased thereafter. On the other hand, the linear change was found to be unchanged with an increase of the sintering temperature up to 1300 °C. Beyond this temperature a sharp increase in the linear change was observed. The slight decrease in the bulk density with increasing temperature up to 1300 °C was attributed to the formation of low density phases (cordierite, spinel) [27]. However, the sharp decrease in the bulk density beyond 1300 °C was attributed to the sharp increase in the formation of cordierite and spinel phases associated with an increase in both the total porosity and linear change as shown in Figs. 3 and 4. However, this study of the physical properties of the compacts could confirm the results of the phase analysis conducted from the XRD patterns. The slight decrease in the bulk density of the compacts with an increase in the sintering temperature up to 1300 °C with a zero linear change, gives information about the extent of the reaction between the different constituents and types of crystal structure. As mentioned from the XRD patterns, up to approximately 1300 °C no abrupt changes in the phases formed were recognized, it seems that some of the phases were decreased and the others were increased while all phases were present

until 1300 °C and no phase was disappeared completely. Upon increasing the temperature to 1325 °C a complete disappearance of proto-enstatite was observed with the formation of large amount of both the spinel and cordierite. This behavior is repeated at a sintering temperature of 1350 °C, more cordierite is formed which causes a sharp decrease in the bulk density.

Effect of sintering time

From the previous study it could be concluded that a sintering temperature of 1350 °C is the most suitable temperature to achieve the highest content of cordierite without the formation of undesired phases (tridymite and proto-enstatite). Meanwhile a study of the effect of different sintering times at a lower temperature (1300 °C) in order to clarify the actual effect of the different sintering times on the phase composition was performed. The sintering time was varied from 2 to 6 h with 2 h intervals and its effect on the both phase composition formed during the sintering and the physical properties of the compacts were investigated.

The effect of different sintering times on the phase formation in a compact containing silica fumes sintered at 1300 °C are shown in Fig. 5. It was found that the effect of increasing the sintering time is almost similar to the effect of the increasing sintering temperature beyond 1300 °C (as shown in Fig. 5). With an increase of the sintering time from 2 to 4 h, the amount of cordierite phase is sharply increased on the expense of the complete disappearance of proto-enstatite. Meanwhile some of the undesired phase tridymite is still present at this sintering time. This pattern looks very similar to the pattern obtained for a sintering temperature of 1325 °C (shown in Fig. 1). With a further increase in the sintering time, the tridymite phase is almost consumed by the formation of cordierite leading to the presence of only cordierite and spinel at this sintering time. Again this pattern at a sintering time of 6 h looks very similar to the pattern obtained for the sintering temperature of 1350 °C (shown in Fig. 1). This observation of the effect

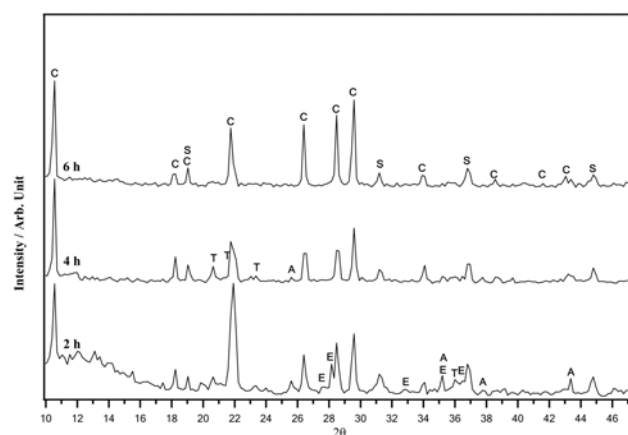


Fig. 5. X-ray diffraction patterns of samples treated for different sintering times at 1300 °C.

Table 2. Physical properties of sample sintered at different times

Time, h	Bulk density, g/cc	Porosity, %	Linear change, %
2	2.12	14.5	0
4	1.94	22.8	3.05
6	1.90	27.1	4.0

of sintering time on the phase composition is consistent with the observation of Goren *et al.* [28]. They mentioned in their study that a thermal treatment positively affects interactions to the formation of cordierite upon increasing the soaking time. The samples sintered at a lower sintering temperature with a larger sintering time exhibit analogous XRD patterns compared with the sample sintered at a higher sintering temperature with a lower sintering time.

The effects of different sintering times on the physical properties of the compacts are shown in Table 2. It was noticed that the bulk density of the compacts decreased upon increasing the sintering time. However, both the total porosity and linear change were increased. This situation is similar to that of increasing sintering temperature. The larger formation of the cordierite phase with increasing sintering time is responsible for the decrease in the bulk density.

From this study it could be demonstrated that the sintering conditions to obtain the highest cordierite content with the absence of undesired phases for compacts containing silica fumes, bauxite and talk could be one of the following:

1. A sintering temperature of 1350 °C with a sintering time 2 h
2. A sintering temperature of 1300 °C with a sintering time of 6 h

Preparation and characterization of porous cordierite

Zeta potential

The effect of different pHs on the zeta potential of suspensions containing 0.1% sodium silicate, 0.1% aluminum mono phosphate and without any addition are shown in Fig. 6.

It was clear that all the suspensions have analogous electrophoretic behavior in an aqueous media and give negative zeta potentials at any pH. For a given pH the zeta potential increases (becomes more negative) from the suspension containing 0.1% aluminum mono-phosphate to one without an addition and to one containing 0.1% sodium silicate. This indicates that the use of aluminum mono-phosphate is not recommended during the processing of porous cordierite from a mixture containing silica fumes as a result of its negative influence on the suspension stability.

On the other hand it was noticed that with a higher basic media (beyond a pH of 8) the zeta potential of the samples without an addition and with an addition of 0.1% sodium silicate tends to decrease (become more positive). This observation is quite clear in the case of the suspension containing 0.1% sodium silicate. This

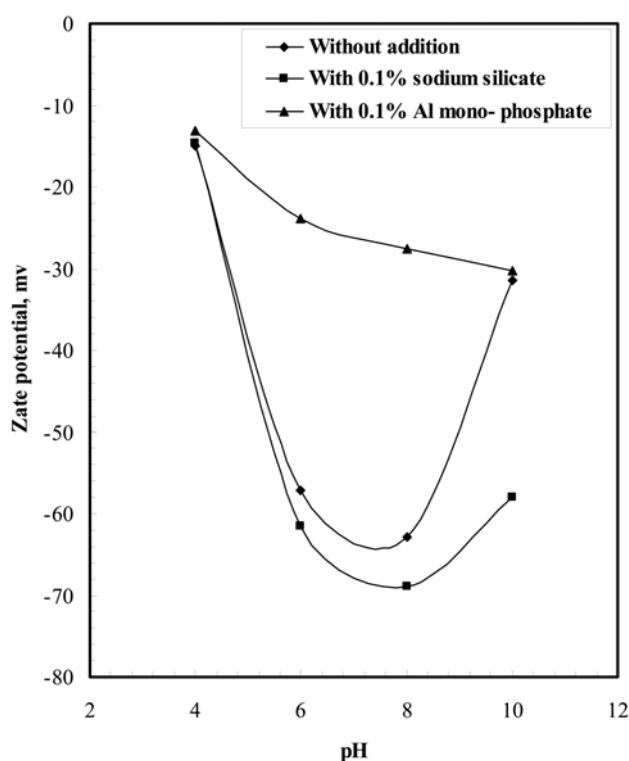


Fig. 6. Effect of pH change on the zeta potential of mixtures containing silica fumes with and without additions.

behavior was attributed to the fact that the high surface potential in highly acidic or basic regions is usually accompanied by a strong ionic force that could compress the double electric layer and decrease the electrostatic repulsive force [29]. The lowest zeta potential was attained for a suspension containing 0.1% sodium silicate at a pH of 8.

Rheology

The flow behavior of a slip having 40% solid loading (consisting of a stoichiometric cordierite from a mixture of silica fumes, bauxite and talc) without and with different percentages of sodium silicate was investigated.

Fig. 7 shows the viscosity against shear rate for suspensions without the addition of a dispersing agent and that dispersed with different dispersant concentrations. It is clear that at a given shear rate, increasing the dispersant concentration leads to a large decrease in the suspension viscosity. However, a suspension without dispersant shows the highest viscosity values in all ranges of shear rates; this is attributed to the large effect of the Van der Waals forces. The Van Der Waals forces are always presents in colloidal systems, and at a microscopic scale, they occur because the interaction of instantaneous dipoles produced in atoms of each particle leads to the formation of more flocs and floc networks and consequently increases the slip viscosity [30]. Meanwhile, the addition of sodium silicate leads to a considerable decrease in the slip viscosity. When water is added to various types of clay, the water becomes oriented between the clay

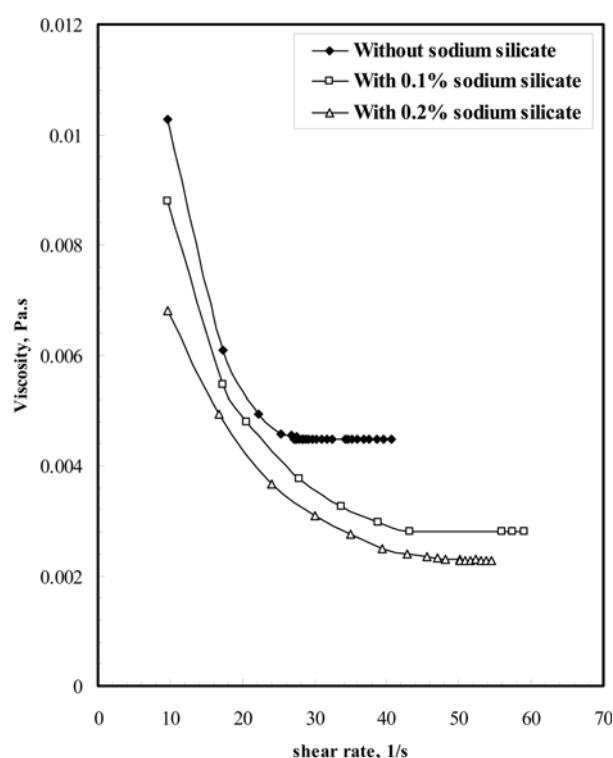


Fig. 7. Shear rate/viscosity curves of mixtures containing silica fumes with bauxite and talc at different dispersion agent concentrations.

particles to form a strong bond. When a polyelectrolyte such as sodium silicate is introduced, ion exchange or ion adsorption occurs on the clay particle surfaces, creating like electrical charges between particles. This charge effect causes the particles to repel one another, thus fluidizing the system and allowing it to flow more freely.

This Fig. (7) also illustrates that all suspensions exhibit a typical shear-thinning behavior with a decreasing viscosity with an increase in the shear rate. This shear-thinning behavior can be noticed at low shear rates where the surface forces between particles dominate the rheological behavior [29]. Shear thinning behavior is usually associated with the structure of the slurry. At low shear rates, liquid is immobilized in empty spaces within flocs and floc networks. With an increase in the shear rate, the flocs and floc networks break down, the entrapped water is released and a more ordered structure is formed in the flow direction [31]. For higher shear rates, where hydrodynamic interaction plays a dominant role, a near Newtonian behavior was observed for all suspensions.

According to the results obtained from the study concerning the zeta potential and suspension rheology, using of 0.2 wt.% sodium silicate (based on the total wt.% of the suspension solid loading) at a pH of 8 are considered the most appropriate conditions for obtaining a well-dispersed suspension of this powder mixture.

Porous cordierite

From the previous study once the slip was optimized from a rheological perspective and the thermal treatment necessary

to promote the formation of cordierite was established, porous bodies were produced by a replica method.

The microstructure of the porous bodies obtained were examined by a scanning electron microscope and are shown in Fig. 8(a) and (b). Fig. 8(a) shows that the porous structure consists of a large number of interconnected spherical pores. On the other hand, it appears that dense struts of solid material are present in the area between these spherical pores. Meanwhile Fig. 8(b) shows a higher magnification of the pore interior. It was noticed that even the pore interiors consists of large numbers of small pores having an irregular shape. The well-developed channels of the connected open pore structure give a filtration ability of these porous ceramics [32]. Filters have been conventionally used in the shape of a long hollow cylinder “candle” with one closed end and with the flow inwards during filtration so that dust is collected on the external surface [10]. This structure of a filter completely matches the structure obtained for the porous cordierite body produced. In addition, the porous structure produced has the advantage of filtrating large size dust particle though the large spherical pores as well small ones through the irregular small pores present in the interior of the large pores.

The measurement of the total porosity using mercury

porosimeter revealed that the total porosity of the porous body was found to be equal 46.4%.

The cumulative pore diameter distribution of the porous cordierite specimen was shown in Fig. 9. From this figure a table which identifies the relationship between the different cumulative volume percentage of pores and their diameters could be constructed and is given in Table 3. This table indicates that about 10% of the total pore volume has diameters of $\geq 70 \mu\text{m}$ and the mean pore diameter (d_{50}) is $\geq 10 \mu\text{m}$. However, it may be noticed from Table 3 that the porous cordierite structure obtained has a suitable percentage of large pore with diameters $> 70 \mu\text{m}$ with a high percentage of small ones. This indicates the suitability of using such a porous body in an application concerning dust filtration.

Conclusions

In conclusion, this paper describes the possibility of

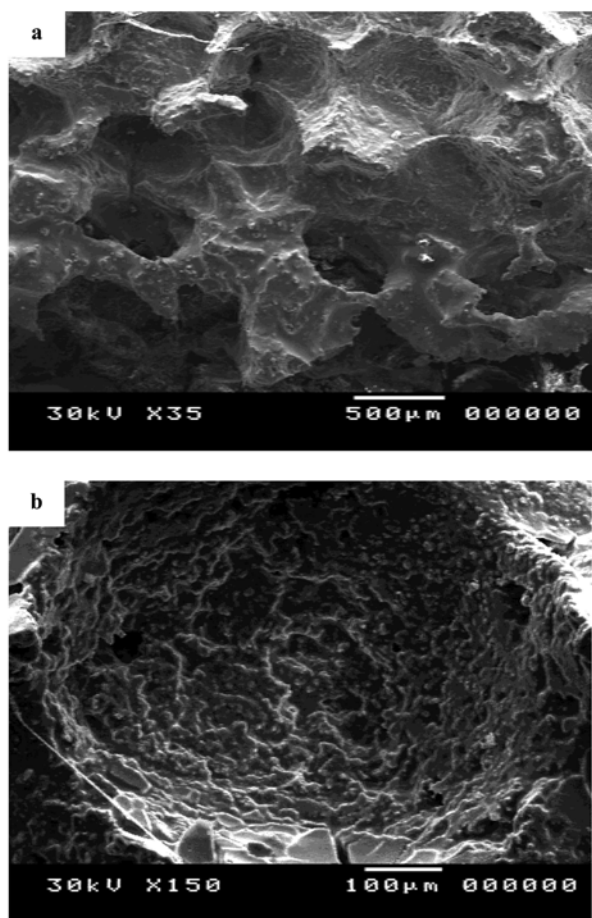


Fig. 8. SEM micrograph of a porous cordierite body, (a) lower magnification, (b) higher magnification.

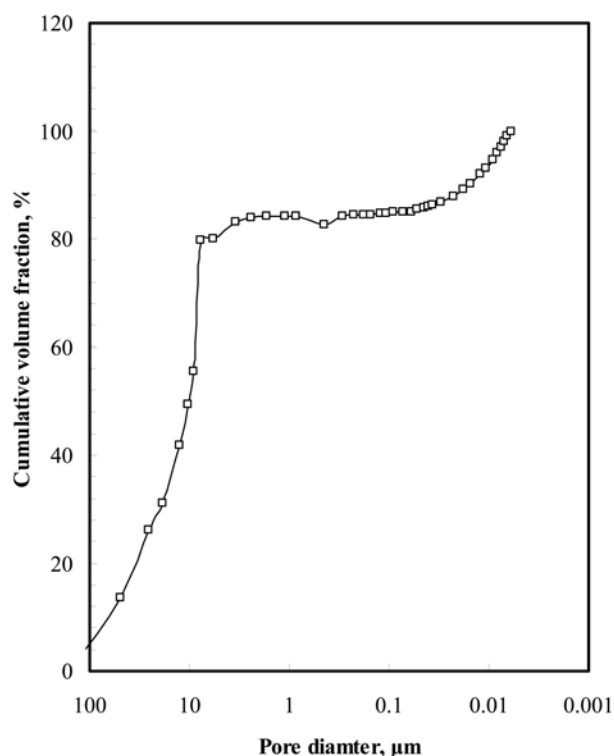


Fig. 9. Cumulative pore diameter distribution by volume.

Table 3. Relationship between cumulative volume percentage and pore diameters

Cumulative pore volume, %	Pore diameter, μm
10	70
20	34
30	19
50	10
70	8
90	0.015
100	0.006

manufacturing porous cordierite bodies from a silica secondary source (silica fumes) using the replica technique in fabricating the porous structure. The sintering temperature as well as the sintering time is the key in synthesizing the cordierite phase without the inclusion of undesired phases such as enstatite and tridymite. However, the suspension pH as well as the type and concentration of additives are important in controlling the suspension rheology. 0.2% sodium silicate with a suspension pH of 8 proved to be the most suitable conditions in obtaining a well-dispersed suspension from the batch mixture. A porous body with a total porosity of 46.4% was successfully obtained from sintering a structure of polyurethane foam impregnated with well-dispersed suspension at the optimum conditions stated for dense body.

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