

Processing of porous cordierite via the utilization of two waste metal oxides (Al-slag and silica fumes)

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ABSTRACT

A successful utilization of two waste metal oxides (Al-slag and silica fumes) in preparing valuable cordierite ceramic material and the fabrication of the produced cordierite in a 3D porous structure have been achieved through this investigation. The optimum sintering conditions (in terms of sintering temperature and time) for producing dense mixture compact containing lower amount of undesirable phases beside the main desired cordierite phase were determined. The results revealed that a sintering temperature of 1325°C and a sintering time of 2 h were the most appropriate conditions during cordierite synthesis. However, 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. Herein 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. A 3D porous cordierite structure were prepared via impregnated the, polyurethane foam 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.

Keywords: silica fumes; Al-slag; cordierite; porous structure; rheology; replica method

1. INTRODUCTION

After the finding of C.W. Parmelee and G.H. Baldwin which showed that in the system of clay-feldspar-talc, a ceramic material (cordierite) with excellent properties is produced, open the door for various investigators to prepare and characterize this material (Parmelee

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and Baldwin 1913, Singer 1929, Parmelee and Thurnauer 1935, Rankin and Mervin 1918, Baruschkun *et al.* 1973). The importance of cordierite ceramic arises from its unique properties, such as: low dielectric constant, low coefficient of thermal expansion, high resistance to thermal shock, excellent high temperature properties, good surface properties and good mechanical properties. Accordingly, cordierite ceramic was found to be a good candidate in various industrial applications, such as thermal insulation, gas adsorption, catalytic or biological substrate, water purification, and mass separation. Cordierites occur in three polymorph forms. The high-temperature hexagonal α -cordierite, also known as indialite, is stable above 1450°C (Schreyer and Schairer 1961, Putnis 1980); and low-temperature orthorhombic β -cordierite polymorph is stable between 1450°C and its melting point, 1460°C, (Meagher and Gibbs 1977, Cohen *et al.* 1977); μ -cordierite is also named metastable cordierite (Naskar and Chatterjee 2004, Gonzalez-Velasco *et al.* 1999). Depending on the composition of original mixtures and applied technology, cordierite products of different densities (1.9–2.6 g/cm³) and microstructures are obtained (Pavlovic and Acimovic 1999, Marinkovic *et al.* 2001, Trumbulovic 1997).

Conventional methods for the synthesis of cordierite ceramics include the solid-state sintering of individual oxides of magnesium, aluminum, and silicon of the corresponding chemical composition of cordierite, or sintering of the natural raw materials such as kaolinite and talc with MgO, Al₂O₃, and SiO₂ content. One of the problems arises from using this technique in cordierite synthesis that cordierite mass always has a very narrow sintering intervals (Gibbs 1996). This makes products very sensitive to sintering, due to deformation of products, as well as to properties. If sintering is performed under optimum temperature, enough quantity of cordierite will not be formed and if sintering is performed over optimum temperature a part of the formed cordierite will disintegrate into mullite and metasilicate of magnesium. Consequently, depending on the raw materials used for cordierite synthesis, high precautions during sintering of these materials for producing cordierite ceramics without contaminations (with other phases) should be taken into considerations. On the other hand there are various methods that have been used in synthesizing cordierite ceramics like crystallization from glasses, growing of single crystal, a sol-gel technique, combustion synthesis (Polezhaev and Darienko 1995). However solid state reaction technique is still the most widely process for cordierite synthesis according to its simplicity as well as its low cost of production.

On the other hand cellular ceramics, also named cellular foams or porous ceramics, show series of remarkable properties because of their highly porous cellular skeleton and composition (Ashby

2005). It has been utilized in various fields such as thermal insulation, gas adsorption, catalytic or biological substrate, water purification, and mass separation. Accordingly the production of cordierite ceramic in 3D porous structure will produce materials of interesting properties combining the properties of porous structure with the properties of cordierite itself. One of these applications is utilizing of cordierite ceramic filter in a high-temperature dust collection system (Izuhara 2000). There are processing routes have been applied in manufacturing of porous ceramics and they have recently been reviewed by Studart et al 2006. 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 (Tripkovic 2006). 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 (Negahdaril 2006). However, the rheology of the ceramic suspension has a key point in deciding the success of using this technique in fabricating porous ceramic bodies.

This investigation dealt with the using of solid state reaction combining with polymeric sponge replica technique in order to produce a 3D porous cordierite body. Usually analytical grade oxide powder or raw materials based on silica, alumina and magnesia was applied in synthesizing cordierite via solid state reaction (Kobayashi *et al.* 2000, Tulyaganov *et al.* 2002, Kumar *et al.* 2000, Tamborenea *et al.* 2004, Yamuna *et al.* 2004, Acimovic *et al.* 2003, Trumbulovic *et al.* 2003). 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 (Zhong 2005). However, there are huge quantities of aluminum slag (dross) were annually produced from exposing of the molten Al surface of primary and secondary process to the atmosphere. In addition considerable amounts of silica fumes (produced as secondary raw materials during ferro-chrome silicon alloy manufacturing) characterized with high silica content and very small grain size is annually produced. The disposal of these materials in the landfill causes severe environmental problems. Although this waste have drastic effect on the environment but it has valuable oxide. Therefore, finding of successful method to utilize this waste in developing products with low cost will significantly contribute in solving the environmental issue and improve the products economy. This investigation is a continuation of our previous two manuscripts confirmed the suitability for utilizing either silica fumes (Ewais *et al.* 2009) or Al-slag (Ahmed *et al.* 2009) in synthesizing 3D porous

cordierite body. However, to the best of our knowledge there is no reports have been mentioned in the literature for using two wastes metal oxide in the course of preparation of cordierite ceramic. Accordingly, the suitability of making combination between these two wastes with studying the effect of this combination on both the reaction progress during cordierite formation as well as the processing of the porous body and characterization of the produced body were carried out.

2. EXPERIMENTAL AND CHARACTERIZATION

2.1 Experimental

Silica fumes (delivered from the Ferro-Chrome Silicon Alloy Company, Egypt), Aluminum slag (delivered from Aluminum industry in Naga Hamdy in 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 Al-slag, silica fumes and talc in proportions equal to the stoichiometric cordierite composition were prepared. In the beginning all the raw materials received 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 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 $^\circ\text{C}$) and different sintering times (2-6 h) at a constant heating rate of 5 $^\circ\text{C}/\text{min}$. 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 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.

2.2 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 (Milosevski *et al.* 1999).

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).

3. RESULTS AND DISCUSSIONS

3.1 Effect of sintering temperature

Fig. 1 illustrates the effect of different sintering temperatures on the phase composition formed during sintering of compacts formed from the mixture containing stoichiometric amount of silica fumes, aluminum slag and talc. The sintering temperature was varied from 1250 to 1350°C with 25°C intervals.

The X-ray diffraction patterns of samples fired at 1250°C indicates the presence of α -cordierite (PDF # 89-1485) as the prevailing phases along with a significant amount of tridymite (PDF # 71-0197), MgAl₂O₄ spinel (PDF # 82-2424), proto-enstatite (PDF # 11-0273) and corundum (PDF # 46-1212). Meanwhile, some of these phases were disappeared with increasing the formation of other ones upon increasing the sintering temperature.

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

- At 1250°C sintering temperature it was found that many phases were present in the produced sample. The cordierite phase is represent the most abundant phase formed during the sintering of the compacts at this temperature. Meanwhile, a noticeable amount of undesired phases (proto-enstatite and tridymite) were detected. However, both of corundum and spinel phases were still present. This result indicates that at this sintering temperature uncompleted solid-solid reaction was attained for developing a complete formation of cordierite. This uncompleted reaction is responsible for the formation of a part of the desired phase (cordierite) with the presence of other ones.

- With increasing sintering temperature at 1275°C it could be noticed that the amount of the proto-enstatite is slightly deceased. Meanwhile, intermediate increasing of spinel amounts as well as increasing in the cordierite phase is clearly observed. This phenomenon was repeated with the increasing in the sintering temperature to 1300°C at which a huge decrease in both of the proto-enstatite phase and tridymite with the formation of more cordierite phase as well as spinel was attained. This behavior is attributed to that with increasing sintering temperature the proto-enstatite phase is decomposed which 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 (Ewais *et al.* 2009).

- With further increasing in the sintering temperature (at 1325°C) a complete disappearance of all undesired phases (tridymite, proto-enstatite) was clearly observed. However, no corundum phase was observed. The pattern give indications that the phases formed at this sintering temperatures were mainly cordierite and spinel without detection any of other phases.

- The most interesting observation was observed when the sintering temperature is further increased beyond 1325°C. At 1350°C appearance of tridymite, enstatite and corundum phases are clearly detected. The appearance of tridymite phase could be easily notice from the peak characteristic for tridymite ($2\theta = 21.6$) which is largely interfered and overlapped with one of the characteristic peaks of cordierite which interpret the high intensity of this peak than expected for pure cordierite phase at this sintering temperature. The explanation for what happened at 1350°C could attributed to the presence of high alkali contents in the aluminum slag especially Na₂O as indicated in the chemical composition of Al-slag (Table 1). The presence of Na₂O is responsible for increasing the

liquid phase formation, viscosity degradation and cordierite decomposition (Ahmed 2009). Accordingly the optimum sintering temperature for producing compacts mainly composed of cordierite phase without the presence of the undesired ones (tridymite, enstatite and corundum) from a mixture composed of Al-slag, silica fumes and talc is 1325°C.

On the other hand, the effect of different sintering temperature on the physical properties of the produced compacts was shown in Figs. 2 and 3.

In fact the apparent porosity is inversely proportional with the bulk density i.e. with decreasing the apparent porosity, the bulk density increases. In this investigation, this trend is valid in the whole range of temperatures, Fig. 2. This abnormal behavior could be understood in term of the formation of low melting glassy phases in parallel with volume expansion. As it is well-known that silica is accompanied by volume change reaches 14 to 23 % with heating above 1000°C. Therefore, the batch volume containing quartz is expected to be increased. This expectation is true and is matched with the results seen in Fig. 3. Also, it seems that there is a race between the volume expansion of the batch and diffusion of the glass phases formed. The liquid phase fill the voids formed among the batch particles during the expansion and wet the batch constituents making in-situ reaction without shrinkage. This kind of mode leads to decrease the porosity and density in the same time till 1300°C sintering temperature. Also, dissimilar trends in bulk density and apparent porosity beyond 1300°C can be related to the formation of some sealed pores.

3.2 Preparation and characterization of porous cordierite

Fig. 4 shows the variation of zeta potential of the powder mixture as a function of pH without addition and with the addition of 0.1 % sodium silicate and 0.1 % aluminum mono-phosphate. All of the three samples show analogous electrophoretic behavior in aqueous media and gives negative zeta potential over the pH range studied. For a given pH the zeta potential increases (become more negative) from the suspension contains 0.1% aluminum mono-phosphate to without addition and to 0.1% sodium silicate. This result indicates that the using of aluminum mono-phosphate is not recommended to be used during the processing of porous cordierite according to its negative influence on the suspension stability.

On the other hand, the increase of the zeta potential with the using of sodium silicate being attributed to complete adsorption of this electrolyte on the particle surface. However, it was also noticed that at higher pH the zeta potential tends to be less negative. This observation is quite clear in case of suspension contains 0.1% sodium silicate. This behavior was attributed to the fact that the high surface potential in highly acidic or basic regions is usually accompanied by strong ionic strength that could compress the double electric layer and decrease the electrostatic repulsive force (Mei *et al.* 2001). According to this observation the using of sodium silicate as dispersion agent at pH of 8 is the most suitable condition for stabilization the slurry of this mixture.

3.3 Rheology

Based on the results conducted from the zeta potential measurement, the flow behavior of the slip having 40 % solid loading (consisting of a stoichiometric amount of silica fumes, aluminum slag and talc) without and with different content of sodium silicate were investigated.

Fig. 5 shows the viscosity against shear rate for the suspension without addition of 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 highly decrease in the suspension viscosity. However, suspension without dispersant shows the highest viscosity values at all ranges of shear rates; this is attributed to the high effect of the Van Der Waal's forces. The Van Der Waal's forces are always presents in the colloidal systems, and at microscopic scale, they occur because the interaction of instantaneous dipoles produced in atoms of each particle which leading to the formation of more flocs and floc network and consequently increasing the slip viscosity (Zibouche and Kerdjoudj 2006). 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 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 surface, 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. While all of the silicates exhibit deflocculation capabilities, is frequently the most efficient (Web site). This figure also illustrated that all suspensions exhibit a typical shear-thinning behavior with increasing shear rate. This shear-thinning behavior can be noticed at low shear rates where the surface forces between particles dominate the rheological behavior (Mei *et al.* 2001). Shear thinning behavior is usually associated with the slurry structure. At low shear rates, liquid is immobilized in empty spaces within flocs and floc network. With increasing shear rate, the flocs and floc network break down, the entrapped water is released and a more ordered structure is formed in the flow direction (Sacks 1984). For higher shear rates, where hydrodynamic interaction plays a dominant role, a near Newtonian behavior was observed for all suspensions.

Based on the results conducted from the study concerning zeta potential and suspension rheology, the using of 0.2 mass-% sodium silicate (based on dry solids) at pH of 8 are considered the most appropriate conditions for obtaining a well dispersed suspension of this powder mixture.

3.4 Porous cordierite body

Once the slip was optimized from rheological perspective and the thermal treatment necessary to promote the formation of cordierite was established, the porous bodies were produced by the replica method.

The microstructure of the sintered porous body was examined by the scanning electron microscope and shown in Fig. 6a & b. As can be shown in Fig. 6a, the produced porous structure consists of large number of large pores having spherical as well as irregular shapes interconnected through challenging. It is worth to mention that the wall separate the large interconnected pores are highly porous. Accordingly the observed struts between large pores were not highly dense as well as not highly porous. On the other hand, it appears that dense struts of solid materials are present in the area between these spherical pores. Meanwhile Fig. 6b shows a higher magnification of struts between large pores, from which it could noticed that these struts contains a small spherical and irregular pores. This structure gives the structure the ability for its utilization in dust filtration purposes. The well developed channels by connected open pore structure give porous ceramics filtration ability (Park *et al.* 2002). Filters have been conventionally used in the shape of long hollow cylinder "candle" with one closed end and with the flow inwards during filtration so that dust is collected on the external surface (Seville 1993). This filters structure is completely fit the structure obtained for the produced porous cordierite body. In addition, the produced porous structure having the advantageous of filtrating large dust particle size through the large spherical pores as well the small one through the irregular small pores present in the interior of the large one.

The measuring of the total porosity using mercury porosimeter revealed that the total porosity of the porous body was found to be equal 50%. While, the cumulative pore diameter distribution of the porous cordierite specimen was shown in Fig 7. From this figure a table identifies the relationship between the different cumulative volume percentage of pores and its diameters could be constructed and given in Table 2. This table indicates that about 10% of the total pore volume having the diameter of $\geq 98 \mu\text{m}$ and the mean pore diameter (d_{50}) is $\geq 14 \mu\text{m}$. This indicates that this porous cordierite having a suitable percentage of large pore diameter $\geq 98 \mu\text{m}$ with higher percentage of small ones. This indicates the suitability of using such porous body in the application concerning dust filtration.

4. CONCLUSIONS

It is possible to use two wastes metal oxides (silica fumes and aluminum slag) in preparing 3D porous cordierite body via applying sponge replica technique. The sintering temperature is for the raw mix is highly critical in order to produce final compacts containing mainly cordierite phase without the presence of undesired phases like silica, corundum and enstatite. The 1325°C for 2 hr sintering conditions is enough to obtain

compacts composed mainly of cordierite phase from this raw mix. The using of 0.2 wt% sodium silicate at pH 8 is the most suitable conditions for producing slip of the raw mix with optimum rheology for being applied in the replica technique. The sintered body produced from the replica technique was found to be highly porous and the pore size distribution revealed that it contains both large (>98 μm) and small pores with the presence of porous struts between the large pores. These properties give the suitability for applying the produced body in the field of dust filtration.

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Table 1

Components	Chemical composition, %		
	Silica fumes	Al-slag	Talc
SiO ₂	94.45	2.77	62.7
Al ₂ O ₃	1.46	87.57	0.36
MgO	0.44	4.2	30.94
Na ₂ O	0.59	1.86	0.1
K ₂ O	0.66	0.26	0.04
P ₂ O ₅	0	0	0.044
CaO	0.36	0.85	0.54
Fe ₂ O ₃	0	0.25	0.19
TiO ₂	0	0.13	0
MnO	0	0.11	0
L.O.I	0	0	4.99

Table 2 Relationship between cumulative volume percentage and pore diameters

Cumulative pore volume, %	Pore diameter, μm
10	98
20	48
30	30
50	14
70	10
90	1.076
100	0.006

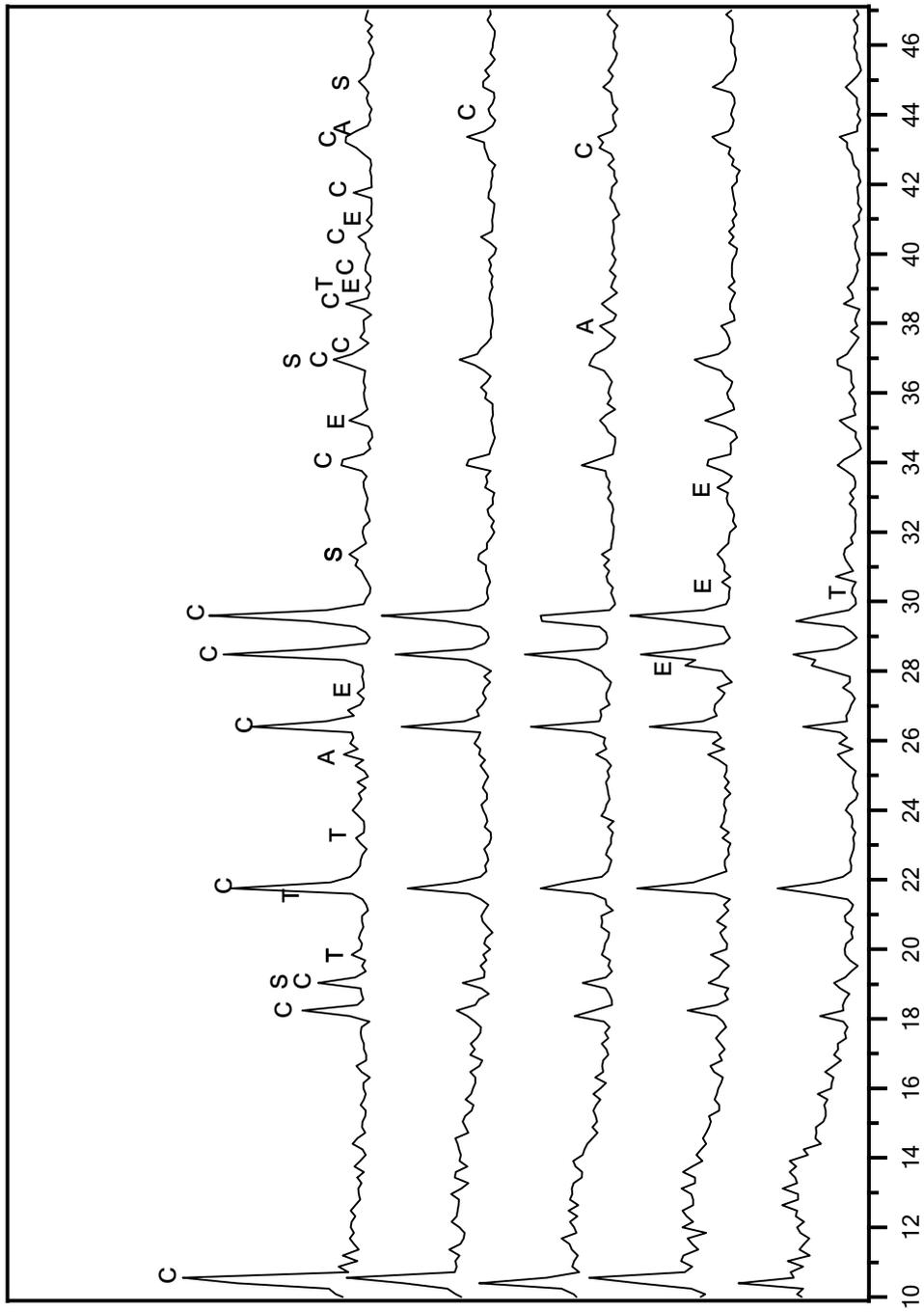


Fig. 1 X-ray diffraction pattern of sample treated at different sintering temperature

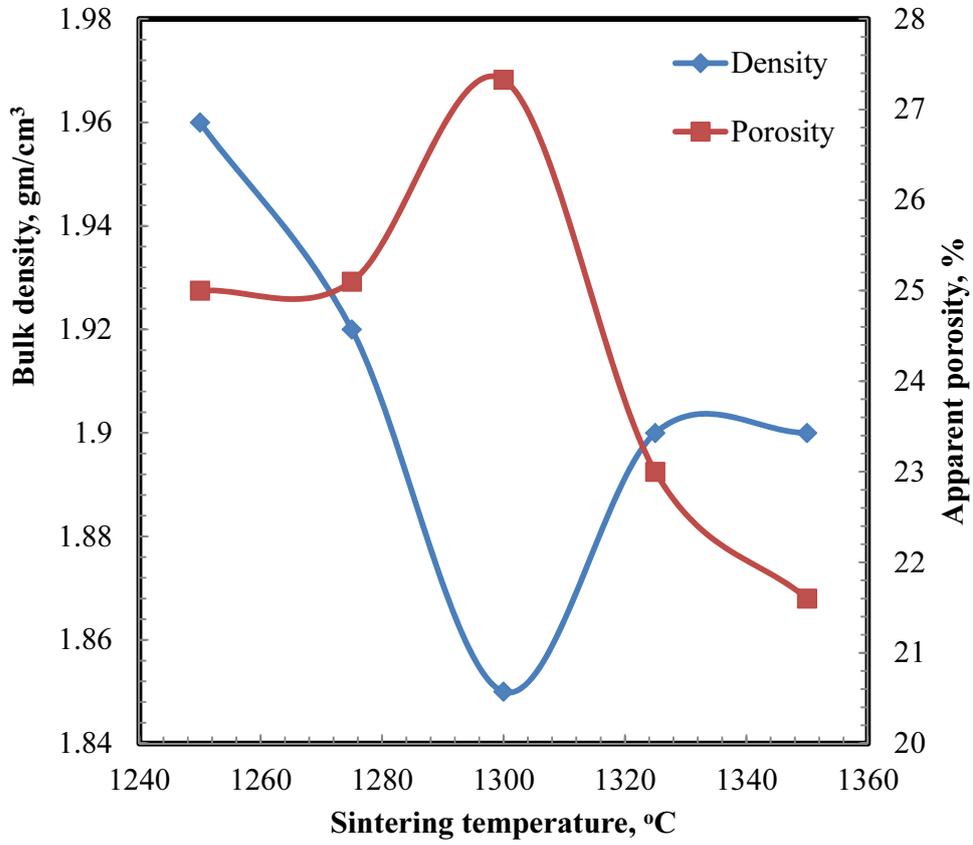


Fig. 2 Effect of different sintering temperatures on the bulk density and apparent porosity of the produced compacts

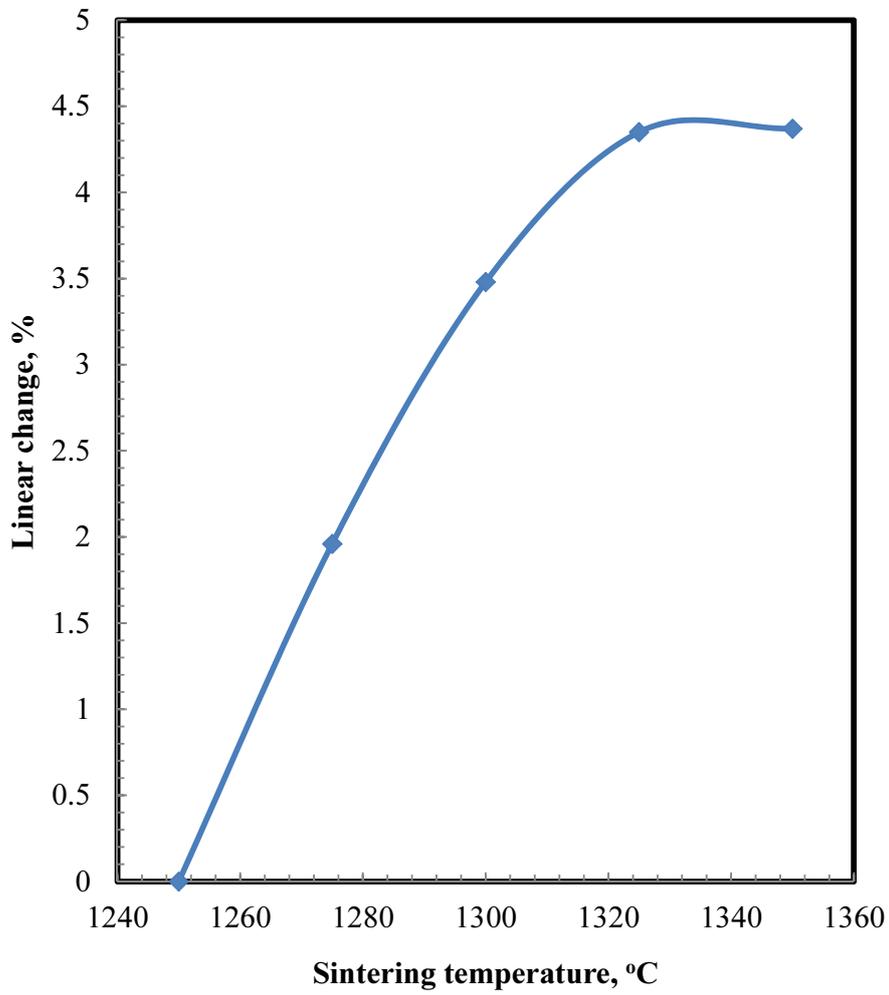


Fig. 3 Effect of different sintering temperatures on the linear change of the produced compacts

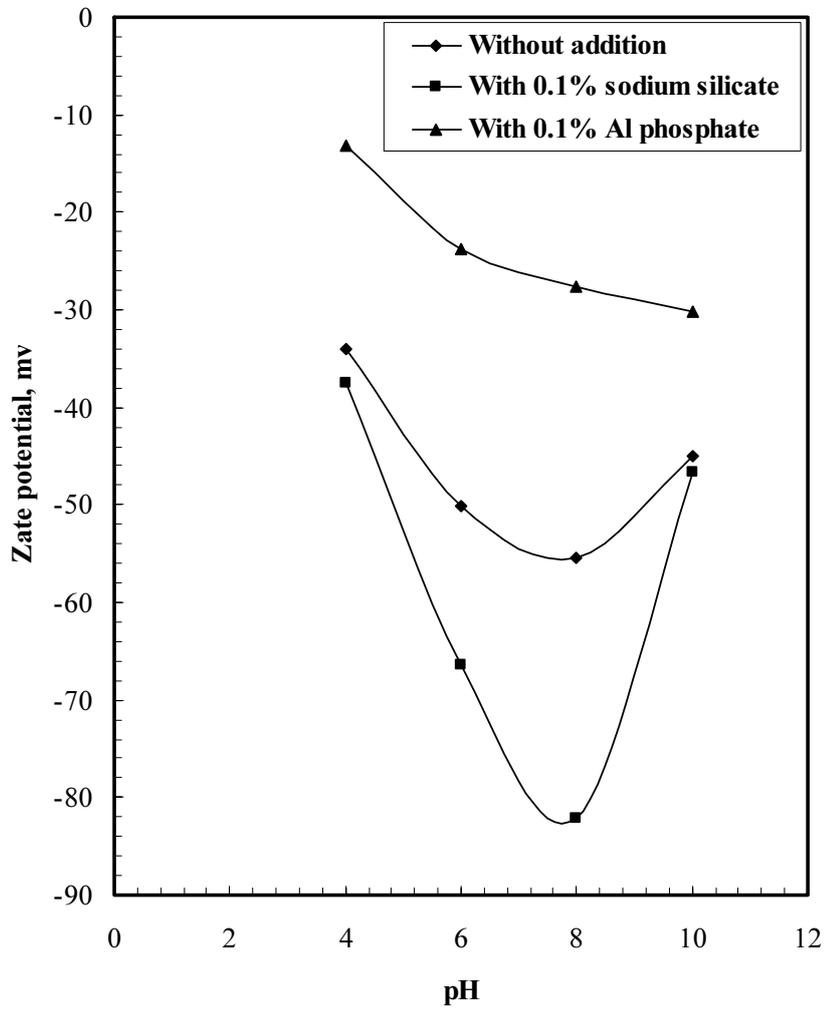


Fig. 4 Effect of pH change on the zeta potential of mixture containing both of silica fumes and aluminum with and without dispersion agent

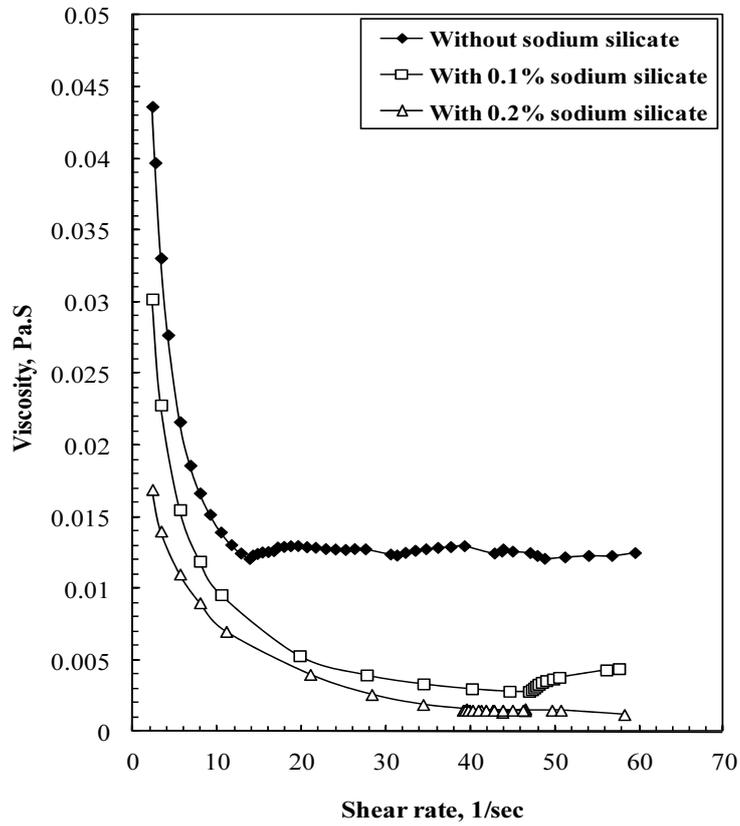


Fig. 5 Shear rate/viscosity relationship of mixture containing silica fumes, aluminum slag and talc at different dispersant concentration

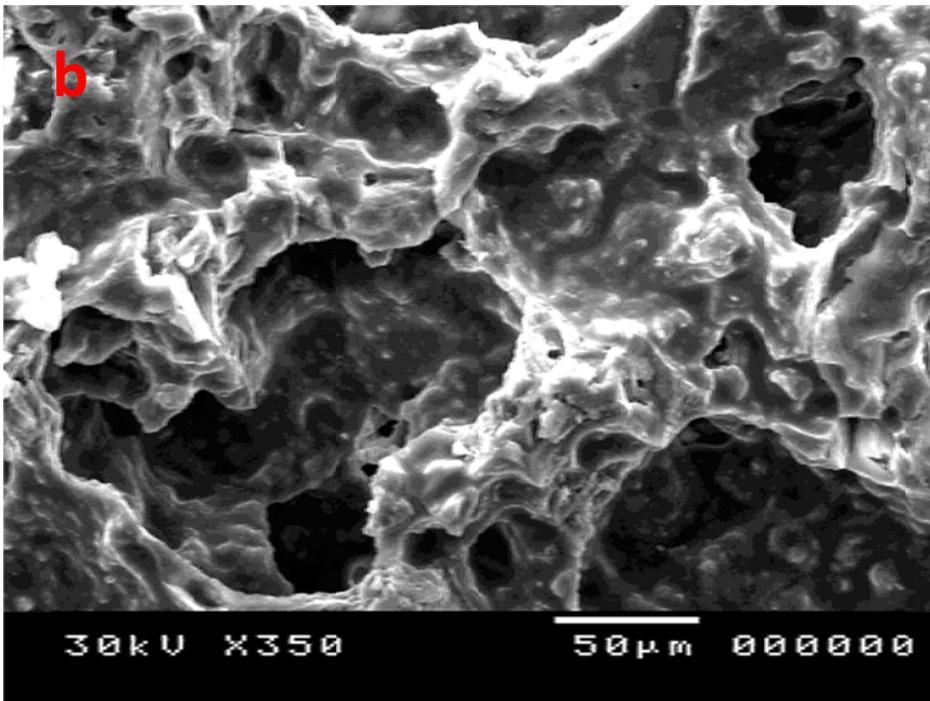
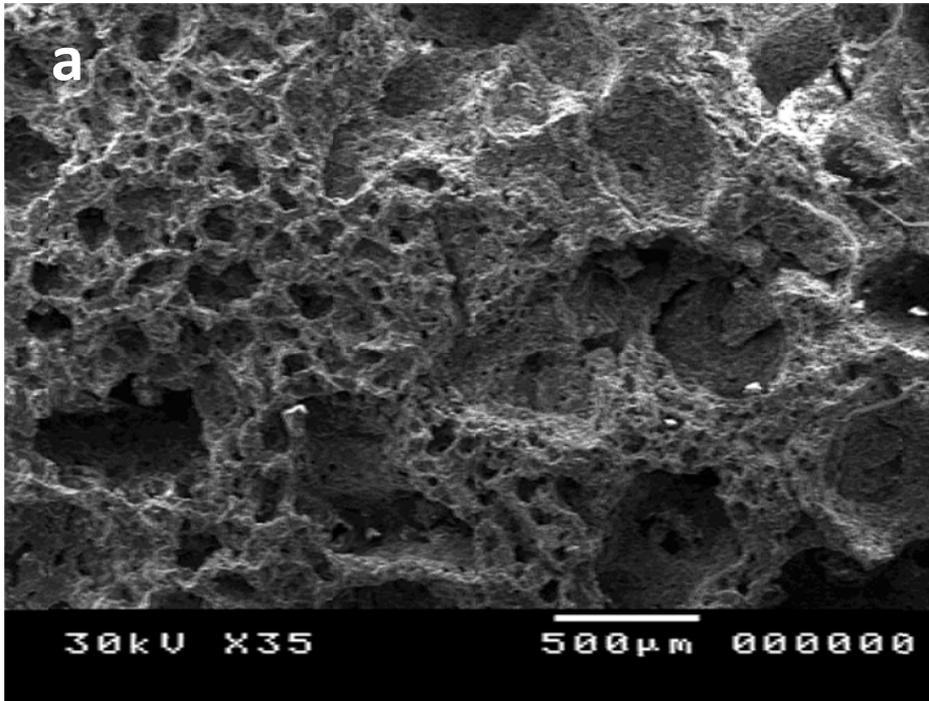


Fig. 6 SEM micrograph of porous cordierite body: (a) lower magnification; (b) higher magnification

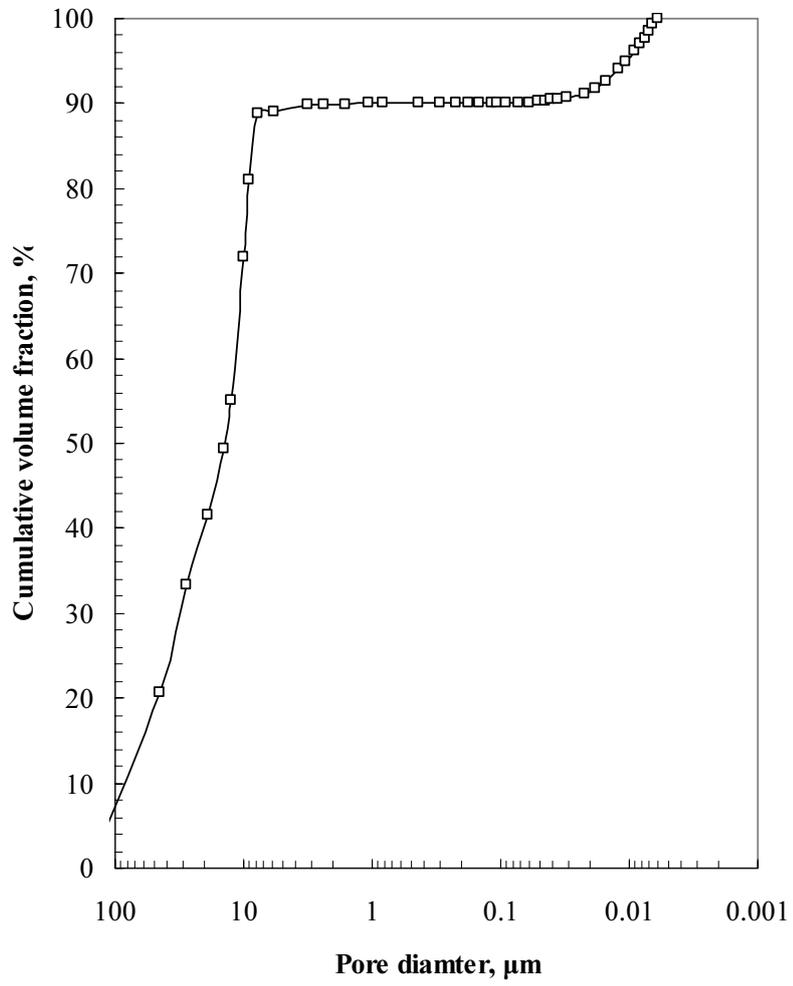


Fig. 7 Cumulative pore diameter distribution by volume