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# Synthesis of cordierite powder from talc, diatomite and alumina

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**Abstract**

A cordierite powder was synthesized from a batch composition of talc, diatomite and alumina. Sintered samples of the synthesized cordierite were characterized by X-ray diffraction (XRD), differential thermal analysis (DTA), and Rietveld analysis. The XRD analysis showed hexagonal or α-cordierite (indialite) as a major phase along with $MgAl_2O_4$ spinel and cristobalite as secondary phases in the samples sintered at 1300 °C for 1 h, whereas indialite phase only was observed at 1350 °C for 3 and 5 h as well as at 1400 °C for 1 h. Density and linear thermal expansion coefficient of the sample synthesized at 1400 °C for 1 h were 2.47 g $cm^{-3}$ and $1.8 \times 10^{-6}$ $°C^{-1}$, respectively.


*Keywords:* D. Cordierite; Diatomite; Talc; Rietveld

## 1. Introduction

Cordierite ($2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$) is one of the important phases of the $MgO–SiO_2–Al_2O_3$ system. Cordierite ceramics have low thermal expansion coefficient, excellent thermal shock resistance, low dielectric constant, high volume resistivity, high chemical durability, high refractoriness and high mechanical strength. Therefore, they are widely used as honeycomb-shaped catalyst carriers in automobile exhaust systems, as substrate material for integrated circuit boards and as refractory materials [1–6].

There are several methods such as solid-state reaction, sol–gel and crystallization from glass to synthesize cordierite. Among these methods, sintering of oxide powders through solid-state reactions or crystallization of glass powders are the most conventional. Chemical routes such as the sol–gel technique generally yield high purity cordierite. In industrial production of cordierite ceramics such as for refractory application, natural raw materials are often used. Some of the starting raw materials reported in literature include (i) a mixture of magnesium compounds and kaolinite [2], (ii) alkaline-earth-aluminosilicate glass, kaolin, alumina and magnesite [3], (iii) talc, calcined alumina and fly ash [4], (iv) kaolin, talc, silica and alumina [5], (v) talc, kaolinitic clay and gibbsite [6], (vi) kaolin, talc and magnesium oxide [7], (vii) talc, kaolin, silica, sepiolite and feldspar [8], and (viii) kaolin and talc [9]. A few fundamental studies have dealt with pure cordierite synthesis, but commonly other minor and/or secondary phases such as mullite, corundum, spinel, forsterite, clinoenstatite, phosterite, and cristobalite were observed together with the cordierite phase.

The focus of this study was to produce pure cordierite, i.e. with the absence of secondary crystalline or amorphous phases. Moreover, the influence of the sintering parameters (e.g. time and temperature) on the formation of cordierite from a raw material mixture containing talc, diatomite and alumina was investigated.

## 2. Experimental procedure

The starting raw materials used were talc (Egypt), diatomite (Kutahya, Turkey) and alumina obtained from Seydisehir Aluminum Plant (Turkey). The chemical compositions of the talc and diatomite are given in Table 1. The

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Table 1
The chemical compositions of talc and diatomite (wt.%), determined by X-ray fluorescence

| Materials | $SiO_2$ | $Al_2O_3$ | MgO | $Fe_2O_3$ | CaO | $(Na,K)_2O$ | LOI |
|---|---|---|---|---|---|---|---|
| Talc | 62.70 | 0.67 | 30.30 | 0.27 | 0.69 | 0.01 | 5.25 |
| Diatomite | 83.91 | 5.21 | 0.75 | 1.42 | 0.95 | 1.21 | 6.41 |

composition of prepared batch, determined by X-ray fluorescence, is 12.32 MgO, 35.18 $Al_2O_3$ and 51.36 $SiO_2$ with some other minor constituents such as 0.53 CaO, 0.18 $K_2O$, 0.16 $Na_2O$ and 0.24 $Fe_2O_3$ in wt.%, formulated according to the general structural formula of cordierite [10].

$$[^{Ch}(K_{0.02}Na_{0.03}Ca_{0.05})_{\Sigma 0.1}\,{}^{VI}(Mg_{1.81})_{\Sigma 1.81}\,{}^{IV}(Al_{4.03}Fe^{3+}{}_{0.02}Si_{5.02})_{\Sigma 9.07}\,xO_{18}]$$

This molecular formula was calculated on the basis of 18 oxygen atoms per anhydrous formula unit. The symbols in the formula were Ch, IV and VI for cavity, tetrahedral and octahedral region cations, respectively. $Ca^{2+}$ ion like $Na^+$ and $K^+$ enter the structural channels of cordierite, altering the molar ratios in ideal cordierite [11]. The stoichiometric cordierite ($2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$) was dispersed in de-ionized water and wet milled with alumina balls (Ø 10 mm) for 8 h. The average particle size of the ground powder, determined by particle size analyzer (Malvern Inst., Mastersizer 2000), was around 9.2 μm. The green compacts were prepared, and then sintered in air at 1300, 1350 and 1400 °C with a rate of 5 °C $min^{-1}$ for 1 h. Some of the samples were also sintered at 1350 °C for 3 and 5 h, respectively. Densities of the sintered samples were measured by the water displacement method. The linear thermal expansion coefficient ($\alpha$) was measured by dilatometry (Linseis Dilatometer) in the temperature range 50–650 °C. The formed phases were analyzed by X-ray powder diffractometry (Rigaku, miniflex) using Cu Kα radiation with Ni filter. The Rietveld analysis for cordierite sintered at 1400 °C with soaking time of 1 h was performed by the Rietveld (DBWS) module of Cerius$^2$ (Accelrys Inc., USA) software at Highly Filled Materials Institute of SIT (USA).

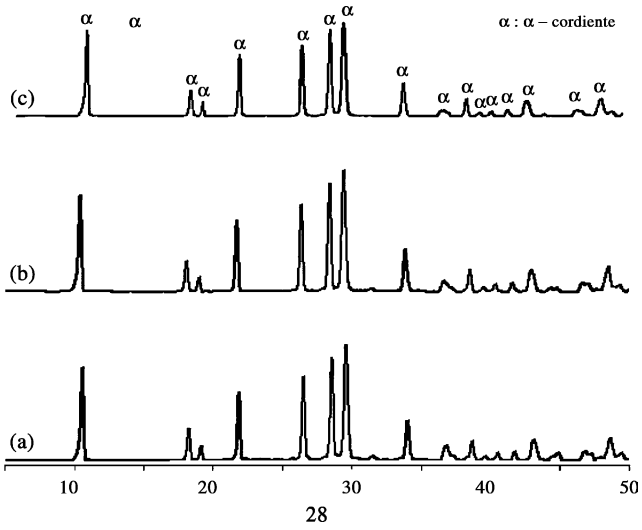


Fig. 2. XRD patterns of the cordierite sintered at (a) 1350 °C for 3 h, (b) 1350 °C for 5 h and (c) 1400 °C for 1 h.

## 3. Results and discussion

The thermal behavior of the cordierite mixture characterized by differential thermal analysis (DTA; Linseis, Thermowaage L81) in air up to 1300 °C at a heating rate of 10 °C $min^{-1}$ is shown in Fig. 1.

The first major endothermic peak represents the dehydration of diatomite. The peak at 955 °C was attributed to the dehydroxylation (or decomposition) of talc to amorphous magnesium metasilicate (enstatite), amorphous silica and water vapor. The decomposition of talc took place in a wide range of temperatures from 650 to 960 °C where μ-cordierite was revealed. The minor exothermic peak around 980 °C corresponds to the transformation of μ- to α-cordierite. The XRD pattern at 1200 °C confirmed that sintered sample contains protoenstatite, cristobalite, and spinel ($MgAl_2O_4$). Indeed, protoenstatite could transform from enstatite and cristobalite could crystallize from amorphous silica. The endothermic peak at 1277 °C shows protoenstatite to decompose and dissolve in the liquid

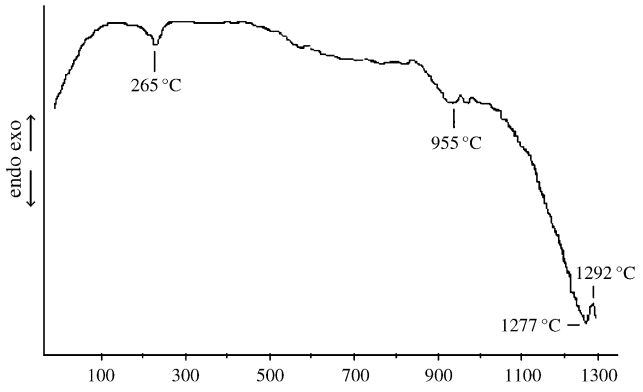


Fig. 1. DTA diagram of the raw materials mixture.

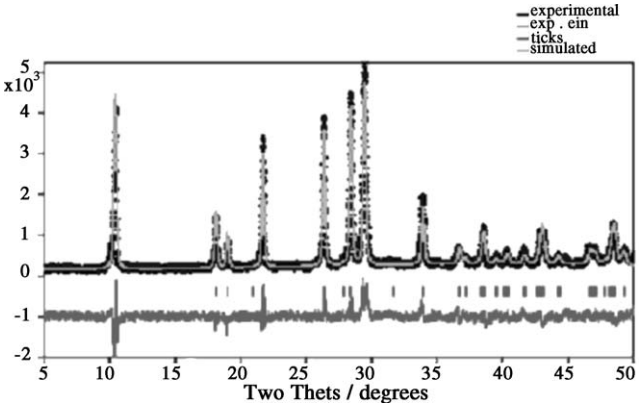


Fig. 3. Observed, calculated and differential pattern of the cordierite.

Table 2
Crystallographic and Rietveld refinement data

| Ceramics | Cell parameters (Å) | | |
|---|---|---|---|
| | *a* | *c* | *c*/*a* |
| α-Cordierite (indialite) PDF # 48-1600 | 9.764 | 9.360 | 0.959 |
| Synthesized cordierite | 9.778 | 9.333 | 0.954 |

Table 3
Densities and coefficient of linear thermal expansion sintered several temperatures

| Temperature (°C) (soaking time, 1 h) | Density (g/cm$^3$) | $\alpha$ (°C$^{-1}$) |
|---|---|---|
| 1300 | 2.24 | $3.1 \times 10^{-6}$ |
| 1350 | 2.32 | $2.2 \times 10^{-6}$ |
| 1400 | 2.47 | $1.8 \times 10^{-6}$ |

silicate phase to allow cordierite crystallization. The last peak at 1292 °C corresponds to the exothermic solid-state reaction to form cordierite as a result of spinel and cristobalite interaction [2–4,12–14].

The X-ray diffraction patterns of samples fired at 1300 and 1350 °C for 1 h indicated the presence of α-cordierite or indialite (PDF # 48-1600) as the prevailing phases along with a significant amount of cristobalite (PDF # 27-0605), $MgAl_2O_4$ spinel (PDF # 21-1152) and trace amounts of corundum (PDF # 46-1212). The cristobalite, corundum and spinel phases disappeared completely in samples fired at 1400 °C for 1 h. The thermal treatment positively affects the interactions to the formation of cordierite upon increasing of soaking time. The samples sintered at 1350 °C for 3 and 5 h exhibit analogous XRD pattern compared with the sample sintered at 1400 °C for 1 h (Fig. 2).

The Rietveld analysis for cordierite sintered at 1400 °C was performed by the Rietveld (DBWS) module of Cerius$^2$ software. Rietveld refinement occurs by improving the parameters of the trial structure so that the simulated diffraction pattern can match an experimental one. In Rietveld (DBWS) analysis and refinement, a simulated powder diffraction pattern is calculated from a trial structure and this is compared with X-ray powder diffraction data obtained experimentally. Fig. 3 shows the simulated refinement pattern with experimental pattern. The value of the agreement factor was $R_P = 12.46\%$ (P is for pattern), which shows good fit. According to the Rietveld analysis, the synthesized cordierite has smaller *c* value than ideal α-cordierite (indialite), and relatively higher *a* value than the ideal one (Table 2).

Density and linear thermal expansion coefficient of samples at different temperature are shown in Table 3. The density of cordierite sintered at 1400 °C and soaking time of 1 h is very close to the theoretical value (2.51 g cm$^{-3}$) for indialite, whereas its linear thermal expansion coefficient of $1.8 \times 10^{-6}$ °C$^{-1}$ is relatively low, as a result of the lower *c* constant compared to the ideal α-cordierite (indialite).

## 4. Conclusions

Cordierite powder was successfully synthesized by using raw materials including talc, diatomite and alumina. The XRD results showed that the synthesized material sintered at 1350 °C for 5 h is formed of indialite phase only. The comparison of samples sintered at 1350 °C for 5 h with 1400 °C for 1 h indicates that sintering time is as important as sintering temperature to improve cordierite recrystallization. It was also found that density and thermal expansion coefficient of synthesized cordierite are very close to the theoretical one.

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