



The effects of mechanical activation on the sintering and microstructural properties of cordierite produced from natural zeolite



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ABSTRACT

In this study, natural zeolite was used as a precursor for cordierite manufacturing. MgO and Al₂O₃ were added to zeolite to obtain the cordierite stoichiometry. A mixture of MgO/Al₂O₃/SiO₂ in the ratio 2:2:5, which was adjusted according to the cordierite stoichiometry for these raw materials, was mechanically activated using a planetary high-energy ball mill for different mechanical activation durations (0.5, 1 and 2 h). The activated powder mixtures were sintered at 1250 °C for 1 h. Both the activated powders and the sintered samples were examined by various analysis techniques. The globular morphology, agglomeration, amorphisation and particle size reduction were observed for powders characterised using scanning electron microscopy (SEM), X-ray diffractometry (XRD) and particle size analysis. The densities of the powders processed for 0.5, 1 and 2 h were measured by the pycnometer method to be 2.655 g/cm³, 2.647 g/cm³ and 2.637 g/cm³, respectively. The sintering behaviour of the samples was determined by measuring the linear shrinkage, water absorption, density and apparent porosity. The sintering and microstructural properties of the samples changed with an increase in the mechanical activation time. The densities of the sintered samples were measured using the Archimedes principle. The densities of the samples processed for 0.5, 1 and 2 h were 2.583 g/cm³, 2.554 g/cm³ and 2.525 g/cm³, respectively. The XRD analyses of the sintered samples revealed that spinel and a glassy phase accompanied the cordierite main phase. The average grain sizes of the samples produced from the powders processed for 0.5, 1 and 2 h were 2.29 µm, 1.91 µm and 1.02 µm, respectively. The hardness values of the samples processed for 0.5, 1 and 2 h were 8.26 GPa, 8.63 GPa and 9.03 GPa, respectively.

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1. Introduction

Cordierite (2MgO · 2Al₂O₃ · 5SiO₂) is one of the phases of the ternary MgO–SiO₂–Al₂O₃ system, along with mullite, cristobalite, tridymite, enstatite, forsterite and sapphire [1,2]. Cordierite is a technically important ceramic that has well-known properties, such as a low thermal expansion coefficient ($\alpha = (1-3) \times 10^{-6} \text{ }^\circ\text{C}^{-1}$), resistance to thermal shock, a low dielectric constant at high frequencies, high resistivity ($\rho > 10^{12} \text{ } \Omega \text{ cm}$), high thermal and chemical stability, high refractoriness and high mechanical strength [3–7]. Therefore, cordierite is widely used as a manufacturing multilayer in circuit boards, catalytic converters, filters, kiln furnaces, thermal insulation materials, membranes, refractories, electrical porcelains, heat exchanger for gas turbines etc. [6–9]. The production of cordierite can be performed by the direct mixing of

- simple compounds, such as oxides, hydroxides and carbonates;
- double compounds, such as clays, talc, steatite, sepiolite, spinel, mullite and forsterite; and
- triple compounds, such as chlorite.

The most commonly used mixtures are as follows:

- clay + talc + (alumina + silica)
- clay + Mg(OH)₂ + minor additives

Minor adjustments to achieve a target stoichiometry are implemented via the addition of different sources of silica, alumina or magnesia [10].

Cordierite ceramics have a very narrow sintering temperature range, and they are not easily sintered without any sintering aids used to operate a liquid-phase process close to their melting point (1460 °C). Some attempts have been made to improve the ability to sinter cordierite. These studies mainly point to both the use of sintering aids and the development of new methods of synthesis [11]. Synthesis methods proposed to reduce the synthesis temperature and improve the properties of cordierite are co-precipitation, solid-state reaction, sol–gel processing and crystallisation from glass [1,10,12].

Currently, different processes affecting the reactivity of the solids are being emphasised. One of these processes is mechanical activation, which is promising for precursor preparation. In this process, as a result of milling, the particle size decreases and the contact surface area between the particles increases. In addition, the energy of the system

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Table 1
Chemical composition of zeolite used as the raw material.

Components (wt.%)										
SiO ₂	Al ₂ O ₃	Na ₂ O	K ₂ O	Fe ₂ O ₃	CaO	MgO	TiO ₂	SrO	Rb ₂ O	ZnO
79.28	11.22	0.15	4.22	1.20	2.52	1.22	0.08	0.06	0.03	0.02

that causes the decrease in the reaction temperature increases. Changes in the texture and structure of the solids can occur [2,13].

Until now, many raw materials, such as clay, kaolinite, talc, andalusite, stevensite, diatomite, steatite, gibbsite, sepiolite and forsterite, were used as raw materials in the production of cordierite. However, to date, there has been no study on the production of cordierite ceramic from zeolite. Zeolites are hydrate aluminosilicate minerals that have the capacity for cation exchange, adsorption, catalysis, molecular sieving and effectively absorb toxic elements [14–17]. Therefore, zeolites have found increasing application in various respects, such as use in the deodorisation of ammonia-polluted environments, in the treatment of municipal and industrial wastewaters, in the preparation of lightweight aggregates, in the recovery of degraded or polluted soils, in the removal of lead from water, in water purification, in radioactive waste treatment, in the removal of toxic heavy metal cations from groundwater, as additives for cement and as inorganic stabilisers of white wines. However, very little research concerns use of zeolite as a raw material in the ceramic industry [15–22].

Low-cost natural zeolites are abundantly found as raw materials in many countries; they are very easily crushed and milled; and they facilitate sintering in the production of ceramic bodies [23,24].

The aim of this study was to investigate the feasibility of using zeolite for cordierite manufacturing and to consider the effect of mechanic activation on sintering and microstructure.

2. Experimental procedure

In this study, natural zeolite (clinoptilolite), MgO (99%, Alfa Aesar) and Al₂O₃ (99.9%, Alfa Aesar) were used as raw materials. The zeolite used in the present study (90–95% purity – SiO₂) was supplied from the ETI Holding Company located in Turkey. First, zeolite was ground using dry ring milling and sieved to pass through a 75- μ m filter. The chemical composition of the zeolite used as a raw material is presented in Table 1. A mixture of MgO/Al₂O₃/SiO₂ in the ratio of 2:2:5, adjusted according to the cordierite stoichiometry, was prepared and mechanically activated for 0.5, 1 and 2 h using a planetary high-energy ball mill (Fritsch Mono Mill Pulverisette 6) with tungsten carbide (WC) balls measuring 10 mm in diameter. The milling was performed under dry conditions at 600 rpm, and the ball-to-powder mass ratio was 20:1.

The morphology of the mechanically activated powders was examined by scanning electron microscopy (SEM) (JEOL JSM 6060 LV). The average particle size, particle size distribution and the nature of the agglomerates were determined using a particle size analyser (Microtrac S3500). The pycnometer method was used to measure the densities of the powders. Phase analyses of the powders were realised by X-ray diffraction analysis (XRD) using a Rigaku D/MAX 220 PC diffractometer operating with Cu-K α ($\lambda = 1.54056 \text{ \AA}$) radiation.

After being characterised, the powders were shaped by uniaxial dry pressing under a pressure of 170 MPa. Then, the samples were dried at 110 °C for 24 h in an oven and sintered in an electrical furnace at a heating rate of 10 °C/min at 1250 °C for 1 h in air. The samples were cooled to room temperature in the furnace.

The sintering behaviour of the samples was evaluated by measuring the linear shrinkage, water absorption, density and apparent porosity of the samples. The density of the sintered specimens was determined by the Archimedes principle. The phases of the sintered samples were identified by X-ray diffraction analysis with a Rigaku D/MAX 220 PC diffractometer operating with Cu-K α ($\lambda = 1.54056 \text{ \AA}$) radiation.

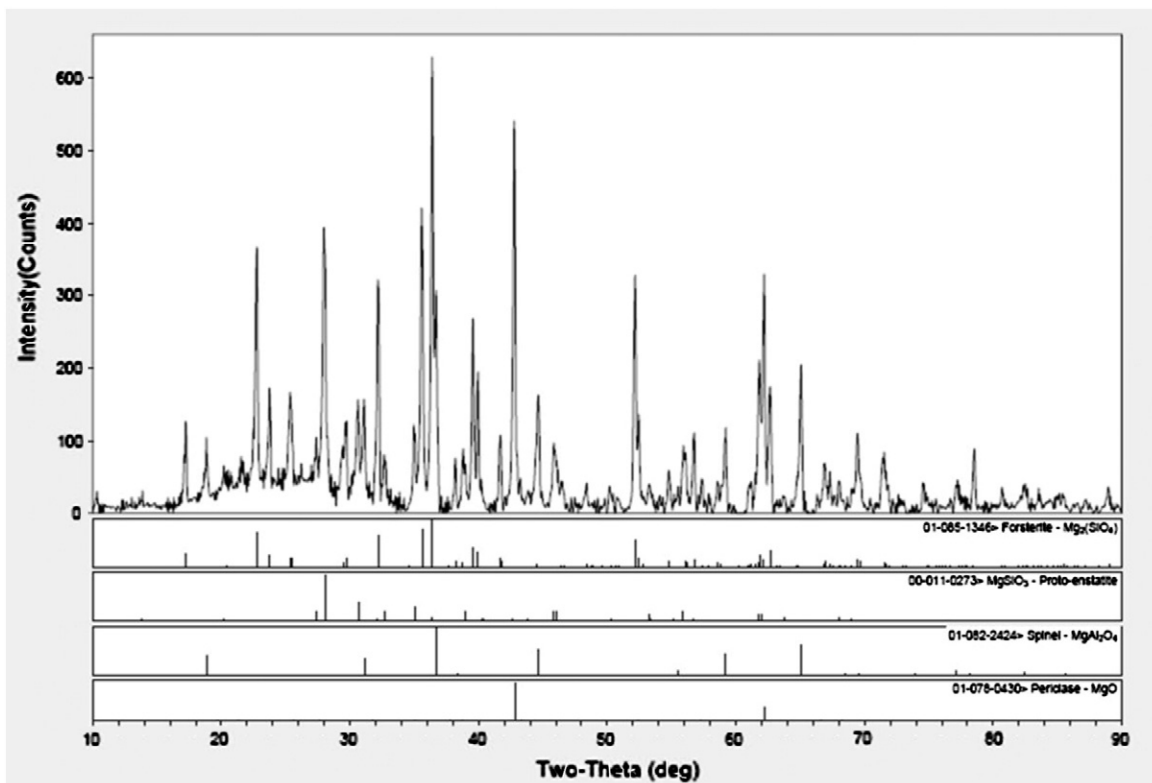


Fig. 1. XRD pattern of the sample sintered at 1400 °C for 1 h produced with non-activated powder mixture.

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