Preparation and Characterization of Bioceramic Cordierite for Medical Applications

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Research Article

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Abstract

The study concentration on the fabricate of bioactive behavior of pure cordierite system and investigate the structure of phase transformation and physical characteristics. cordierite powders were synthesized by chemical coprecipitation, combined Cordierite gel with water-based sol–gel which begin fundamental from magnesium oxide MgO, Alumina Al_2O_3 and silicon oxide SiO_2 . Five specimens were prepared with different weight percentage of Mgo, Al_2O_3 and SiO_2 powders. cylindrical shape samples were press by hydraulic press at 4 bar for 30 minutes. All specimens were sintered at 1250°C .

The microstructure examination of specimens was carried out using Field Emission Scanning Electron Microscope (FESEM), x-ray diffraction (XRD) and particle size analysis The analysis revealed cordierite phase formation at 1250°C , and the intense peaks were identified for composites S4 and S5. The results also indicated the formation of spinel, cristobalite, corundum, protoestatite and cordierite for all the specimens.

1. Introduction

Cordierite is one type of a ceramic material which include aluminum oxide, magnesium oxide and silicate having formula $(\text{Mg}\frac{1}{2}\text{Al}\frac{1}{2}\text{Si}\frac{1}{2}\text{O})_2$. It resembles to hexagonal form of structure (cyclosilicate sheet)[1]. There are Several methods for cordierite preparation [2], including crystallizing the glasses , solid state reactions, mechanical milling, , sol–gel technique, co-precipitation and combustion synthesis [3]. Among these method, the simplest one is co-precipitation, it considered to be low cost and suitable for large production compared to other methods[4][5][6], the solid-state sintering is the most common method for synthesis and preparation of cordierite system at high temperatures. The benefit of sol–gel method is higher purity and homogeneity, final structure had finer grain size, heat treatment at low temperatures, and low cost can be considered in comparison with the solid state method for preparation of such structures[7]. The advantage of Cordierite ceramics is very low coefficient of thermal expansion and high thermal shock resistance[8], high chemical stability and high mechanical strength[9]. which are used in metallurgy, chemical industry , environmental protection and electronics, automobile[10], hot gas filters, membrane supports and membranes, catalysts and catalyst carriers.[11] recent research focusing on medical application of cordierite system ,especially in field of 3D scaffold for bone replacement and cell growth on bioceramic[12][13][14][15]. **Stoyanova** studied the crystallization temperatures of spinel-like cordierite, corundum, mullite, and MgAl_2O_4 . The naturally occurring clay materials were used for the preparing of kieselguhr, and supports: bentonite,. The resulting of the composition samples varied over the following ranges: clay material, from 45 to 60%; MgO, from 10 to 20%, and Al_2O_3 , from 30 to 42%;. The found that the conditions responsible for spinel formation in mullite phases and cordierite over temperature range $1050\text{--}1150^\circ\text{C}$. they discovered that the mixture was prepared by coprecipitation is more thermal stable than composites prepared by mechanical mixing [16]. **Maris Rundans** studied the improvement of Cordierite phase from natural raw materials, and they observed that Cordierite with ceramic material contain can be successfully obtained when substituting around 20 wt.% of required

material with dolomite and/ or 10 wt.% of illite clay. Temperature of 1200°C is enough to obtain these ceramics. As improve by XRD analysis, the main crystalline of all specimen is that of a cordierite, with anorthite as secondary phase [17]. **QingdongHou** discussed the cordierite powder preparation of by solid reaction sintering, rotation evaporation , and chemical coprecipitation, the grain size and morphology of samples densify at different temperatures were conceder. Secanning electron microscope shown that the cordierite crystal structural was divided into three processes; in the range of temperatures around 1000 °C (the hexagonal flake–like grain formation) and when the temperature rise to 1200 °C (the flaky texture grain formation), so that these two sintering transition points is important. The partical size was well developed in the range of 196.5–222.7 nm. The main crystal phase was cordierite phase for the samples sintered at 1200 °C which confirm by Phase analysis and mullite phase, a little spinel phase and at 1400 °C the peak intensity of the characteristic peaks was more intense. [18]

In this paper, the cordierite was synthesized by precipitation method, using raw materials of high purity powder of Al_2O_3 , MgO , and SiO_2 according to cordierite stoichiometric ratio. The impact factors such as, sintering temperature, cordierite particles size and granulation process for cordierite powders synthesis were investcated by Field Emission Scanning Electron Microscope (FESEM) and X-ray diffraction.

2. Materials And Methods

In order to synthesis ceramics cordierite samples, three white powders include magnesium oxide from (Parchem Fine & Specialty Chemicals, USA) with purity 99%, aluminum oxide from (Riedel-de Haën, Germany) and silicon dioxide from (Daejung Chemicals) were used, five different groups were prepared according to calculated chemical composition and molecular wieght fraction for each group of ($x\text{Mgo}$, $x\text{Al}_2\text{O}_3$, $x\text{SiO}_2$) respectively as shown in table 1. Firstly ,100 g of raw material for each system was utilized to synthesis cordierite, dissolved with 100ml of distilled water and mixed for 3 hours with 120 RPM, secondly, the mixture was stirred at 80 r/m for 2h to obtain a well-mixed sol. Thirdly, the filter papers utilized to dry mixture then finally incubated with 100C° for 1hr. the dried samples exposed to hydraulic press with 4 bar for 30 minute , after that it was calcined and sintering temperature was 1250C° for 2h and heating rate was 42 C° / min.

Table 1.1: chemical composition of prepared powder samples

sample	mole fraction	system	Mgo wt%	Al2O3 wt%	Sio2 wt%	molar mass(g/mol)	sintering temperature(C°)
S1	(1:1:1)	$\text{MgAl}_2\text{SiO}_6$	20%	50%	30%	202.344	1590.577808
S2	(1:2:1)	$\text{MgAl}_4\text{SiO}_9$	13%	67%	20%	304.304	1578.32206
S3	(1:1:2)	$\text{MgAl}_2\text{Si}_2\text{O}_8$	15%	39%	46%	262.424	1520.560833
S4	(2:1:2)	$\text{Mg}_2\text{Al}_2\text{Si}_2\text{O}_9$	27%	34%	40%	302.728	1602.89736
S5	(2:2:5)	$\text{Mg}_2\text{Al}_4\text{Si}_5\text{O}_{18}$	14%	35%	51%	584.928	1496.339878

Flow diagram for cordierite based on coprecipitation method as shown in figure 1

3. Results And Discussions

ternary equilibrium diagram (Figure 2) Represent the three compositions in the $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$ system, the concentration of oxides was determining without loss on content and ignition of the MgO. The compositions of S1, S2 and S3, were found in the region sapphirine which is small area in vicinity of 6 invariant points of cordierite [19]. The compositions S4 & S5 were located in cordierite region. The differences observation was related to raw materials which starting with and S4& S5 formulation with talc which gives the nearest composition of cordierite stoichiometric point, mainly to MgO and SiO_2 concentration. One of the major problems of e cordierite is its small area of existence due to the vicinity of 6 invariant points

XRD measurements are performed at an angle of $10\text{-}80^\circ$ with scanning speed of $5^\circ/\text{min}$ using an LabX XRD-6000 X-ray Diffractometer from Shimadzu with $\text{CuK}\alpha$ radiation ($1.5406\text{\AA}$) operated at 30 mA and 40 keV. The X- Ray data was plotted using the origin Pro software and the peaks were matched with JCPDS software to get the phases present. The corrected Debye-Scherrer formula was used calculated grain size (Dhkl) of the identified crystalline phase:

$$G \cdot S = \frac{k\lambda}{\beta \cos \theta} \quad (3 \cdot 1)$$

Where

β = FWHM (full width at half maximum),

K = is the shape factor (= 0.9),

θ = Bragg angle

λ = wavelength of $\text{CuK}\alpha 1$ ($1.54056\text{\AA}$). However, the cordierite nanocrystalline size was determined as shown in Table 1.2 and it was in the range of 30–80 nm.

Table2: XRD spectral data for different samples

samples	2-Theta	d(nm)	I%	FWHM	G.S(nm)
S1	17.902	0.4957	6.3	0.159	65
	52.319	0.1742	50.5	0.162	69
	57.332	0.1607	98.5	0.195	54
S2	25.827	0.3448	85.6	0.218	42
	36.922	0.2435	89.3	0.326	27
	44.906	0.2018	62.9	0.299	30
S3	28.04	0.3175	86.6	0.17	59
	32.74	0.2731	18.4	0.195	49
	66.471	0.1404	86.4	0.153	81
S4	43.275	0.2089	17.4	0.177	58
	47.633	0.1905	5.9	0.191	53
	66.461	0.1406	67.6	0.161	75
S5	19.13	0.4635	5.5	0.167	60
	35.272	0.2545	19.2	0.153	72
	57.643	0.1598	100	0.163	70

The table 2 shows a summary of the results of the process for the X-ray diffraction examination, which include all parameters values (were obtained for all the systems prepared by the chemical precipitation method that were clarified in the paragraph of the practical part.

The results in the table showed values at three peaks in the first system (S1)(1,1,1), which are at with grain size (G.S) 65nm,69nm and 54 nm respectively . This is shown in Figure 3, where this figure appeared at a temperature of 1250°C several systems added to the cordierite system, namely spinel, corundum and cristobalite , these systems result from an internal interaction of the three reactants (Mgo, Al_2O_3 , SiO_2) at temperatures below 1250°C, with regarding second system S2 (1,2,1) , it can be noted that system include cordierite with appearing sub system (spinel, cristobalite, corundum) .

As the ratio of silica to alumina change because of the reaction of alumina and silica present in the chemical composition. The cordierite peaks cordierite began to show only at temperature of 1200 °C, and then intensified when the temperature reach 1250 °C. The cordierite phase was observed in the five compositions, at 1250 °C. Askin et al. [20] reported similar results.

The changes that have occurred to the system S3 (1,1,2) can be observed where S3 include high ratio in comparable with Al_2O_3 and MgO , from figure 3, we notice the decline of the cordierite curve and the emergence of other systems (S3, S5) clearly as a result of side reactions between the materials.

The system S4 (2,1,2), the result show that spinel curves was dominant in addition to cordierite system and this is attributed to the reaction of Al_2O_3 and MgO at temperature under 1250°C and formation of spinel. finally, the system S5 is a fixed system in which the cordierite system is achieved in a stable and clear way, in addition to other systems, and this means that at a temperature of 1250°C , cordierite can form at a ratio (2,2,5) and this is agreement with other author's results [17,18]

The densification temperature plays important role in the cristobalite and spinel phase transformation into cordierite phase. As the temperatures of sintering range $1200\text{--}1300^\circ\text{C}$, cordierite phase will be dominant phase, but cristobalite and spinel are undetectable.[21]

The peaks Mullite phase will start to disappear at 1200°C , according to [22] When the temperature reach 1250°C the mullite phase may be react with (MgO) magnesium oxide in the talc to create cordierite phase. The average diameter of the particles less 100 nm, which is similar to previous studied reported that it is possible to obtained cordierite phase from talc and pure kaolin, [23]. For the system S5 which will the particles have diameter smaller than 100nm this may contributed to form cordierite phase, the kinetics reaction of solid state is effected by surface area. It was also found different sizes of the particle in the mixture of different components, it will be favor of wider distribution of the particles, lead to high area of the contact which is significant parameter in formation and diffusion new phase.

The diameter of the about all particles of composite S5 are a smaller than $2\text{ }\mu\text{m}$, which may related to the cordierite formation, since kinetics of the solid state reaction is infected by surface area. The waste mixture also was found that containing particle with different sizes favored distribution of a wider particle size, will increasing the contact area, which is a important factor in the formation and diffusion of cordierite phase. The largest mean particle size of Composition S5 70 nm.

Figure 3 X-ray diffraction shows, the XRD patterns of the compositions after sintering at 1250°C . The most detected phases were spinel, magnesia, cristobalite and corundum peaks as shown in Figure 3. The result of reaction between silica, alumina and magnesia at 1250°C , in the system ($\text{MgO}\cdot\text{Al}_2\text{O}_3\cdot\text{SiO}_2$) resulted in dominant phase emergence of cordierite, which is the aim of this study.

Greater intensity peaks of composition S4 was observed from the Analysis of the peaks of cordierite for the system S3 and S4, this due to the smaller grain size, which making them more reactive. furthermore, at this temperature there was a small increase in cristobalite peaks

at 1200°C , Cordierite started to form the intense peaks showed increasingly at 1250°C . for Composition S5 figure 3 which was similar to composition S4,

the mechanism can be explaining this, always more probable of solid state has been considered reaction between binary compounds, and then among three compounds. For this reason, the formation of the cordierite ($2\text{MgO} \cdot 5\text{SiO}_2 \cdot 2\text{Al}_2\text{O}_3$) will start from the two compounds reaction, or a binary compound and an oxide reaction.

The presence of impurities in the raw materials which been used, can cause the formation of glassy phase, but considering the effect is not significant so that it will be not considered.

Scanning electron microscope examination

The structure was examined with Field-Emission scanning electron microscopy of MIRA3 TESCAN, the figure 4 represent the morphology of system S1 where the particles agglomerate slightly and most of them are spherical with a small number of reunion at lower sintering

The shapes and magnifications show the coherence and complete interaction between the materials and that the value of the diameters $D1 = 76.27 \text{ nm}$, $D2 = 42.63 \text{ nm}$, $D3 = 73.51 \text{ nm}$, and the figure 5 represent the percentages of the substances involved in the reaction, and it can be observed that there is a consistency and accuracy in the preparation process that was interpreted by EDX test.

The figure 6 shows an electron microscope image of the S2 system. Through the image, it is shown that the average particle size is between 44-59 nanometers, and the shape of the precipitated particles is close to the cuboid. It is noted in the image that there is great homogeneity in the reactants and high fusion at degrees of 1250°C . The results of EDX in figure 7 show that there is a great convergence of the stoichiometric ratio of practical work with the results of EDX according to the system (1,2,1) ($\text{MgAl}_4\text{SiO}_9$) and this explains the accuracy of the results of using the preparation method. For system S1 and S2 the high content of alumina has not high impact on cordierite formation because the reaction with other constituent need slightly higher than this temperature.

regarding system S3 ($\text{MgAl}_2\text{Si}_2\text{O}_8$), the figures 8 was clarified that the average particle size is between 31 to 123 nanometers. It is also shown that the particle size is semi-cubic and semi-rectangular. As for the EDX test, the results in figure 9 showed a degree of accuracy between the theoretical side and the results of the EDX test, and this proves the success of the appropriate preparation process.

Figure 10 shows the FESEM images of sample S4 ($\text{Mg}_2\text{Al}_2\text{Si}_2\text{O}_9$), the average grain size range is (34-58) nm with homogeneity of the structure, Clearly, it is not possible to distinguish between the materials, as we can observe the uniform distribution of the particles and figure 11 show EDAX result

The figure 12 shows the system S5 ($\text{Mg}_2\text{Al}_4\text{Si}_5\text{O}_{18}$), and we note the formation of cordierite in a homogeneous and clear manner, and the grain size ranges between 33 and 49 nanometers, as can be explained by the EDX test in figure 13, the highest peak intensity for oxygen element followed by silica, alumina and magnesia respectively and this is explaining the accuracy of chemical precipitation method for preparing cordierite system.

Microstructure analysis for the specimens systems S3, S4 and S5 as shown in Figures (8,10,12), after heat treatment with applied 42°C/min heating rate and 120 minutes holding time at 1250 °C as sintering temperature.

The microscopic structure of specimen S3, which was prepared by sintering at 1250°C, shows that the crystal structure was circular in shape similar to round petals, a shape which is almost similar to that observed by the source [24].

The researchers investigate the growth and nucleation of cordierite crystals processes from liquid phase and indicated there are three different shapes, presented the structure such as first solid nucleate crystals inclusions in liquid phase and radially growth as rose petals, second The crystals isolated growth from both compact crystal layers and outer surfaces third growth from the center of specimen. The configuration of the crystals of the S4 system which is sintered at 1250 °C hexagons and elongated petals presented the morphology, the appearance is similar to finding which is reported by [25].

Crystals structure of component S4 is showglassy phase with heterogeneous crystals and an elongated tube-like morphology, which is due to the composition hashigh amount of flux. Also needle structure are visible may be related to mullite phase as shown in figure 10. Hexagonal crystals structure showed in composition S5 which is similar to morphology observed by [26,27]. This component was formulated with precursor of MgO and talc, which is may be responsible of different morphology, like the nucleation and parallel surfaces growth of the hexagons structure.

4. Conclusion

pure cordierite powders effectively prepared by water-based sol–gel method and density at 1250 C °. According to the chemical co-precipitation method cordierite ($2\text{Mg} \cdot 5\text{SiO}_2 \cdot 2\text{Al}_2\text{O}_3$) was synthesized, it can be concluded that the raw materials chemical compositions, average particle size and sintering temperature influence the cordierite formation. The nucleation temperature of cordierite started at 1250 C °. The present work presents the results of synthesis with different percentages of MgO, Al_2O_3 and SiO_2 , which give different peaks intensity of the cordierite phases the heights intensity achieved with composite S4 and S5. Several reactions involves during the entire process of cordierite synthesis, to obtained cordierite phase is always a secondary phase accompanied, which is the spinel.

Scanning electron microscope technique were used to determine the microstructure. The morphological analysis revealed the formation of cordierite in a homogeneous and clear manner, and the grain size ranges between 33 and 49 nanometers hexagonal tube-like crystals and rose-like structure has been identify.

compositions S3 and S4 were located in the region of sapphirine which is small field in the vicinity of 6 invariant points of the cordierite

The S5 composition was located in the region cordierite. The differences was observed due to the raw materials which is starting and the S5 formulation with cordierite stoichiometric point talc, mainly due to the MgO percentages and SiO₂.

Declarations

Ethics approval and consent to participate

This project involved a number of techniques and data analysis evaluate the Influence of “Preparation and Characterization of Bioceramic Cordierite for Medical Applications”

And I agree to consideration by Journal of Silicon

Consent for publication

All the authors hope this work to be consider by Silicon Journal

Availability of data and materials

The data that support the findings of this study are available on request from the corresponding author, [initials]. The data are not publicly available due to containing information that could compromise the privacy of research participants].

Competing interests

I had full access to all study data, take fully responsibility for the accuracy of the data analysis, and have authority over manuscript preparation and decisions to submit the manuscript for publication

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Please include the sub-sections below of Compliance with Ethical Standards section.

Author A hasn't **received** **research** grants from any Companies or organizations for this research.

* Research involving Human Participants and/or Animals

the studies have been performed in accordance with the ethical standards as laid down in the 1964 Declaration of Helsinki and its later amendments or comparable ethical standard

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Figures

Figure 1

cordierite preparation based on coprecipitation method

Figure 2

ternary equilibrium diagram for SiO₂-Al₂O₃-MgO system.

Figure 3

XRD patterns of cordierite ceramics sintered for different molecular weight fraction samples

Figure 4

FESEM for system (S1) ($\text{MgAl}_2\text{SiO}_6$) with magnification a)200kx with dimensions, b)200kx , c)100kx

Figure 5

EDX for system (S1) ($\text{MgAl}_2\text{SiO}_6$)

Figure 6

FESEM for sample 2 (S2) ($\text{MgAl}_4\text{SiO}_9$) with magnification a) 200kx with dimensions, b) 200kx , c) 100kx

Figure 7

FESEM for sample 2 (S2) ($\text{MgAl}_4\text{SiO}_9$)

Figure 8

FESEM sample 3 (S3)(MgAl₂Si₂O₈) with magnification a)200kx with dimensions, b)200kx , c)100kx

Figure 9

EDX for sample 3 (S3)(MgAl₂Si₂O₈)

Figure 10

FESEM sample 4 (S4) ($\text{Mg}_2\text{Al}_2\text{Si}_2\text{O}_9$) with magnification a)200kx, b)200kx , c)10kx

Figure 11

EDX of sample 4 (S4) ($\text{Mg}_2\text{Al}_2\text{Si}_2\text{O}_9$)

Figure 12

FESEM of sample 5 S5 ($\text{Mg}_2\text{Al}_4\text{Si}_5\text{O}_{18}$) with magnification a) 200kx with dimensions, b) 200kx , c) 10k

Figure 13

EDX for sample 5 S5 ($\text{Mg}_2\text{Al}_4\text{Si}_5\text{O}_{18}$)