FABRICATION OF POROUS CORDIERITE BY GELCASTING METHOD

by

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LIST OF ABBREVIATIONS

CTE Coefficient of thermal expansion

DPF Diesel Particulate Filter

DSC Differential Scanning Calorimetric

GRF Glutinous rice flour

DEW Dried egg white

FESEM Field emission scanning electron microscopic

rpm Revolution per minutes

XRF X-ray florescence

XRD X-ray diffraction

TEOS Tetraethylorthosilicate

TTT Time temperature transformation

LIST OF SYMBOLS

Tm Melting temperature

Tg Glass transition temperature

Tp Crystallization temperature

To Initial temperature

T Temperature

ρ Density

αl Coefficient of thermal expansion

 θ Plane angle

Ø Diameter

°C Degree Celsius

Md Weight of dry pellet

Ms Weight of suspended pellet

Mw Weight of saturated pellet

wt % Weight percent

Equivalency or similarity between two value

 λ CuK α radiation

FABRIKASI KORDERIT BERLIANG MENGGUNAKAN KAEDAH PENUANGAN GEL

ABSTRAK

Di pelbagai bahan seramik untuk tujuan penapisan, (2MgO.2Al₂O₃.5SiO₂) berliang menawarkan ciri pengembangan pekali rendah haba yang rendah, rintangan yang tinggi kepada kejutan haba dan kekuatan yang tinggi. Dalam kajian ini, korderit telah disediakan dengan kaedah penghasilan kaca menggunakan SiO₂, Al₂O₃ dan MgO sebagai bahan mentah. Serbuk korderit yang dihasilkan kemudiannya digunakan untuk fabrikasi korderit berliang dengan menggunakan kaedah penuangan gel. Kaca yang telah dikisar dicampur dengan air suling dan agen penyerak selama 2 jam pada 600 rpm. Ejen pembuih telah ditambah ke dalam buburan diikuti dengan ejen gel kemudian dituang ke dalam acuan dan dikeringkan. Akhir sekali, sampel telah disinter pada 1350 °C. Kesan saiz partikel korderit (masa kisaran yang berbeza), jumlah pepejal, dan jumlah bahan penyebaran, telah dikaji untuk mendapatkan ciri-ciri optimum korderit berliang. Setelah korderit berliang yang optimum diperolehi, jumlah tepung beras (GRF) dan putih telur kering (DEW) yang berbeza telah ditambah. Pencirian telah dilakukan melalui analisis saiz partikel, pembelauan sinar-X (XRD), kalorimetri pengimbasan pembezaan (DSC), ketumpatan, keliangan, kekuatan mampatan, modulus pecah (MOR), mikroskopi imbasan elektron (SEM) dan ujian kelepasan habuk. Keputusan XRD menunjukkan jumlah korderit sebagai fasa utama meningkat dengan peningkatan suhu lebur. Analisis saiz partikel menunjukkan pengurangan saiz partikel telah meningkatkan ketumpatan, mengurangkan keliangan dan meningkatkan kekuatan. Tambahan pula, pengurangan jumlah pepejal menunjukan pengurangan ketumpatan, peningkatan keliangan dan pengurangan kekuatan. Manakala, pengurangan jumlah agen penyerak menunjukkan pengurangan ketumpatan, peningkatan keliangan dan pengurangan kekuatan. Sifat-sifat optimum korderit berliang telah diperolehi dengan penambahan 4.0 g kanji (GRF), mempunyai 0.5 g/cm³ ketumpatan, 74% keliangan, 9.13 MPa kekuatan mampatan dan 4.07 MPa MOR. Ujian kelepasan habuk menunjukkan ketebalan optimum korderit berliang adalah 10.0 mm yang memberikan kecekapan 100% dan kebolehtelapan yang tinggi dengan pengurangan tekanan yang rendah pada 200 Pa. Secara umumnya, keputusan yang diperolehi menunjukkan korderit berliang yang dihasilkan adalah berkemungkinan menjadi bahan alternatif bagi penapis udara dandang kilang minyak kelapa sawit.

FABRICATION OF POROUS CORDIERITE BY GELCASTING METHOD

ABSTRACT

Among various ceramics materials for filtration purpose, porous cordierite (2MgO.2Al₂O₃.5SiO₂) offers promising properties due to low coefficient thermal expansion, high resistance to thermal shock and high strength. In this study, cordierite was prepared by glass melting method using SiO₂, Al₂O₃ and MgO as raw materials. Cordierite powder produced was then used to fabricate porous cordierite using gel casting method. The glass was milled, mixed with distilled water and dispersant for 2 hours at 600 rpm. The foaming agent was added into slurry followed by gelling agent then cast into a mould and dried. Finally, the sample was sintered at 1350 °C. The effect of cordierite particle size (different milling time), solid loading and amount of dispersant were studied to obtain optimum properties of porous cordierite. After the optimum porous cordierite obtained, the different amount of glutinous rice flour (GRF) and dried egg white (DEW) was added. Characterizations were done through particle size analysis, X-ray diffraction (XRD), Differential Scanning Calorimetric (DSC), density, porosity, compressive strength, modulus of rupture (MOR), Scanning Electron Microscopy (SEM) and Dust Passage Test. XRD results shows the amount of cordierite as the main phase has increased with the increasing of melting temperature. Particle size analysis shows that decreasing of particle size increased the density with decreasing of porosity and increasing of strength. Furthermore, the decreasing of solid loading shows decreasing of density, increasing of porosity and decreasing of strength of porous cordierite. While, decreasing of dispersant amount shows decreasing of density, increasing of porosity and decreasing of strength. The optimum properties of porous cordierite obtained with the addition of 4.0 g GRF , having $0.5~\rm g/cm^3$ density, $74~\rm \%$ porosity, $9.13~\rm MPa$ compressive strength and $4.07~\rm MPa$ MOR. Dust Passage Test shows that the optimum thickness of porous cordierite was $10.0~\rm mm$ which demonstrate $100~\rm \%$ efficiency and high permeability with low pressure drop at $200~\rm Pa$. Generally, results obtained shows that porous cordierite produced from this study was possible to become alternative materials for palm oil mill boiler filter.

APPENDICES

Appendix A [Calculation of cordierite fabrication based on non-stoichiometry formulation]

xCaO. (21-xMgO) .26Al2O3. 53SiO2

For CaO = 0

(0)CaO. (21-(0)MgO).26Al₂O₃. 53SiO₂

 $= 21 MgO.26 Al_2O_3.53 SiO_2$

Actual weight percent of individual materials to be mixed were calculated use in nonstoichiometry for 100 wt.%.

For MgO:

 $21/100 \times 100 \text{ wt.}\% = 21 \text{ wt.}\%$

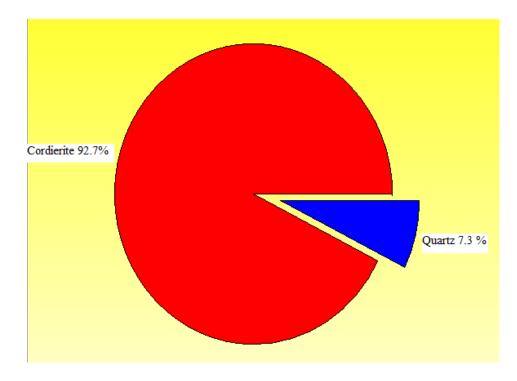
For Al₂O₃:

 $26/100 \times 100 \text{ wt.}\% = 26 \text{ wt.}\%$

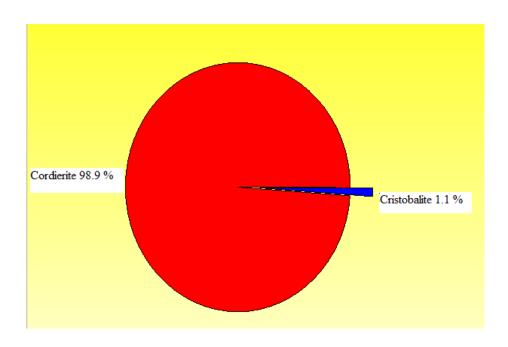
For MgO:

 $53/100 \times 100 \text{ wt.}\% = 53 \text{ wt.}\%$

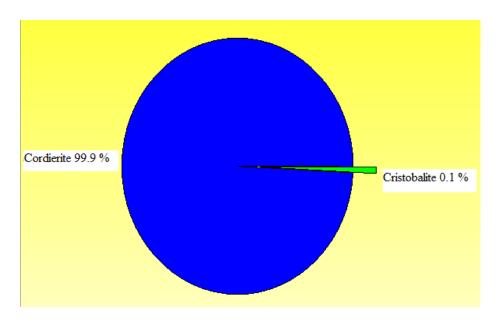
Appendix B [XRD phase analysis]



1540 °C



1550 °C



1580 °C

CHAPTER ONE

INTRODUCTION

1.1 Cordierite

Cordierite is a useful crystalline phases in MgO-Al₂O₃-SiO₂ ternary system (Guo et al., 2014) with interesting properties such as low dielectric constant, low coefficient of thermal expansion, high resistance to thermal shock, and good mechanical properties (~ 243 MPa) (Sanad et al., 2014), high chemical durability and a high electrical resistivity (Demirci and Günay, 2011). Due to the properties offered by cordierite, it was successfully applied for diesel particulate filters, steam engine heat exchanger and substrate material for integrated circuit boards (Guo et al., 2014). Besides that, cordierite based materials are extensively used in a wide range of applications such as automotive, electronic, filters and refractories (Bejjaoui et al., 2010). It is considered as potential candidate for advanced applications in various field (Benhammou et al., 2014).

Synthesis of cordierite ceramic materials were greatly explored and suggested. Among various synthesis methods, solid-state sintering, sol-gel routes and glass melting routes MgO, Al₂O₃ and SiO₂ in ratios corresponding as raw materials to the chemical composition of cordierite (2MgO.2Al₂O₃.5SiO₂) were widely used. Natural mineral raw materials such as talc, kaolin, feldspar and dolomite were raw materials that commonly used to synthesis cordierite ceramics in industry production (Bejjaoui et al., 2010).

1.11 Porous Cordierite

Ceramics structure with more than 30 % porosity could be considered as porous ceramics. The porous ceramics system consist of solid substance that form walls between the hole part that called pore and the pore fill with air (Ewais, 2009). Porous ceramics materials have a very wide application due to the unique structure that provide several properties such as low density, high porosity, low thermal conductivity, high temperature resistance, high specific surface area and high permeability (Colombo and Hellman, 2002).

Porous cordierite is among the special porous ceramic materials due to its extensive properties which can withstand high temperature with very low thermal expansion ($a = 1-3 \times 10^{-6} \,^{\circ}\text{C}^{-1}$) over a wide range of temperature and thus, offers outstanding resistance to the thermal shock in an abrupt temperature change (Albhilil et al., 2013). According to Sandoval et al. (2012), the porous cordierite are used as thermal insulators because of its low thermal expansion coefficient and low thermal conductivity. Besides that, the porous cordierite is a very good option due to its high resistance to thermal shock (Benhammou et al., 2014). There is no doubt porous cordierite offers as one of the high temperature applications.

Furthermore, the porous cordierite is also well known as high mechanical properties (up to 10 MPa). There are several porous ceramics which high mechanical strength properties, however, porous cordierite has advantage with high mechanical strength at elevated temperature (Sandoval et al., 2012).