International Journal of Current Science, Engineering & Technology
ISSN : 2581-4311
Original Research Article | Open Access | AMCT 2017 Malaysia | *Special Issue*

# Influence of Slurry Rheology Behaviour for Fabricating Reticulated Macroporous Cordierite

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**ABSTRACT.** The reticulated macroporous cordierite body had been fabricated through polymeric replica template method by using different water content slurry. The rheology behavior of slurry study was performing by adjusting the degree of water content of slurry (45%, 50%, 55% and 60%). The viscosity and rheology behavior of different degree of water content's slurry was determined by using Viscometer at different shear rate. The viscosity profile indicated that, all the slurries was presented in pseudoplastic flow behaviour respectively. The SEM micrograph revealed that the macroporous cordierite prepared by using 50% water content slurry showed the bulky and mitigate flaws appearance with high porosity level (90.95± 0.81%). However, the low solid content slurry resulted deficient in compressive strength (0.0306± 0.012 MPa).

***Keywords:*** *Macroporous cordierite, Rheology behaviour of slurry, Water content;*

*Received: 15.10.2017, Revised: 15.12.2017, Accepted: 30.02.2018, and Online: 20.03.2018;*

**DOI: *10.30967/ijcrset.1.S1.2018.373-377***

*Selection and/or Peer-review under responsibility of Advanced Materials Characterization Techniques (AMCT 2017), Malaysia.*

## 1. INTRODUCTION

Recently, porous ceramic was attracting the attention of industries such as automotive technologies, membrane application, medical (bone implantation) and catalyst supports application due to high melting point, good chemical, low density, high porosity, low thermal conductivity, high permeability and high specific surface area [1-5] . With the excellent properties, porous ceramic habitually applied as application where involved high temperatures and corrosion media atmosphere such as diesel particular filters (DPFs), water purification filters, metal molten filter and catalyst support [1-5].

Cordierite ($2MgO.2Al_2O_3.5SiO_2$) has been found as alternative materials to fabricated as porous body instead of alumina ($Al_2O_3$) due to excellent properties, such as low thermal expansion coefficient ($3.0 \times 10^{-6}/^oC$), excellent thermal shock resistance, good chemical durability, and mechanical properties [6–8]. Presently, the non-stoichiometric cordierite composition of $2.5MgO.1.8Al_2O_3.5SiO_2$ with 5 wt.% $TiO_2$ has been discovered to give high purity (96.4 wt.%) of α-cordierite phase at sintering temperature of 1375 °C for 2 hours through solid state reaction method [9].

The final properties of porous ceramic were highly impacted by fabrication process route. The polymeric foam replica method is the most popular method to fabricate macroporous ceramic due to reproducibility and suitability of the process that allow the templates to hold its original shape [5,10]. The process involved impregnation the polymeric foam into a ceramic slurry and a layer of ceramic slurry coating over the strut of template. Followed, the dried green foam was undergoing the sintering process to an appropriate temperature to pyrolysis the template and crystallization the ceramic. During the sintering process, the pyrolysis of polymeric foam and organic content such as binder (PVA) contributed the formation of pore and longitudinal crack at edge of the struts. Thus, in this work, the porous cordierite prepared by using the slurry with different water content without any additives of deflocculants had been studies in order to mitigate the formation of flaws on the sintered porous cordierite.

## 2. MATERIALS AND METHOD

Cordierite with non-stoichiometric formulation of $2.5MgO.1.8Al_2O_3.5SiO_2$ with 5 wt% of $TiO_2$ as nucleating agent was used in this study for slurry preparation. The water content of the slurries was varied at 45%, 50%, 55% and 60% with 3 wt.% PVA binder (M.W 50000-85000) based on the total solid loading. The deionized water was heated to 60 °C to 70 °C to liquefy the binder. Subsequently, the cordierite powder was added based on the required amount of solid loading to the diluted solution. The replication route begins by fully immersed the PU foam (pore size range; 300-600 µm) into slurry to ensure the slurry get into and filled the cell of the template. The excess slurry was removed by squeezed with two parallel rollers with 5 mm gap. The immersion and squeezed procedure was repeated for four times for each sample. The green body coated PU templates was dried at room atmosphere for 48 hours. Followed, the dried green porous body were slowly sintered in a conventional high temperature furnace (MHI M18-40) in normal atmosphere. The sintering process was programmed as follows; the samples was sintered from room temperature to 575 °C at 2 °C/min for 1 hour and further sintered to 1375 °C for 2 hours at heating rate of 5 °C and furnace cooled to room temperature.

The rheology behavior of different water content slurry was determined by using viscometer (Brookfield DV-II+ Pro) at different shear rate respectively. The microstructure analysis of sintered porous cordierite was established using Hitachi TM3000 Tabletop Microscope. The bulk density and total porosity of sintered porous samples were determined by electronic densimeter (Hildebrand H-300S). The compressive strength test was carried out by using universal testing machine (Instron) at ambient temperature. The crosshead speed and load cell was 0.5 mm/min and 100 kN, respectively.

## 3. RESULTS AND DISCUSSION

***3.1 Rheology behavior of slurries.*** The rheological behaviour of the slurry plays a key role in impregnation process. Fig. 1 shown the rheology profile of the slurries with different water content at various shear rate respectively. The slurries showed high viscosity at low shear rate and gradually decreased as increased in the shear rate, this rheology behaviour refer to as shear thinning or pseudoplastic flow and indicates a flocculated state of slurry. All slurries had shown the pseudoplastic flow behaviour as evidenced by the viscosity profile. The viscosity of slurry decreased as the solid content decreases and this result is similar to the work done by Jamaludin et al. [5] and Zhu et al. [11]. The flocculated ceramic slurry was greatly dependent on the solid loading as well as concentration of ions in the system. In water-solid system, the concentration of ions was dependent on the water and solid loading ratio. Hence, the association equilibrium in this work was expressed as.

$$2H_2O \leftrightarrow H_3O^+ + OH^- \rightarrow pH = -\log[H_3O^+] \quad (1)$$

All the flocculated system required time to break and restructure and shear thinning behaviour was greatly related with flocculated suspension [11]. When force (shear) applied to the flocs system, the particles network in the suspension becomes unstable and lead the particles rebuild and form a new network structure in static equilibrium by Brownian motion driving force [12, 13]. The high solid loading slurry provided high concentration of bivalent ion in the association equilibrium system which reduce the repulsive inter particles force and the motion of particles was restricted by the consolidated layer and new incoming particles [13]. Therefore, the time allowed for particle rearrangement in freedom particle network in static condition is insufficient and resulted the slurry become viscous [11, 13]. Furthermore, $H_2O$ molecule in the flocculated system act as dipole and re-compensation the unsaturation bonding site of $O^{2-}$ at the outermost layer of pure oxide particles by increasing the repulsive force between the particles in the aqueous system. Hence, the slurry with high water content (60%) obtained lower viscosity at low shear rate compared with high solid loading slurries.

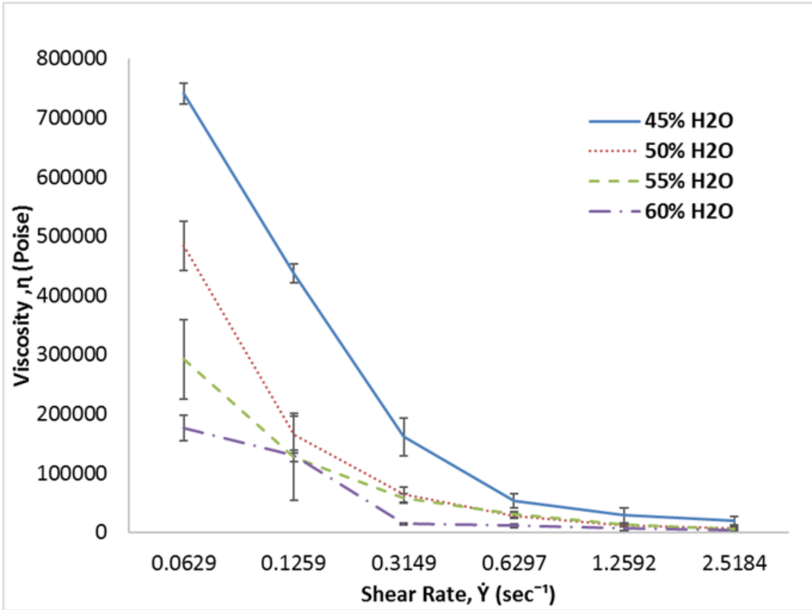


**Fig. 1** Viscosity profile of MAS slurries with different water content respectively

***3.2 Microstructure Analysis.*** Fig. 2 shown the SEM of sintered macroporous cordierite prepared from various water content slurries. All the samples were presented in three dimension interconnected open cell versatile pore structure with pore size range of 300 - 600 µm. As can been see in Fig. 2(a), the sintered cordierite foam prepared via 45% water content's slurry shows a clear longitudinal crack along the edge of strut. Furthermore, the pore size in range of 3.53 - 8.44 µm was observed on the wall of the strut. This is due to the high flocculated slurry system leaded an uneven coating layer especially at the shape edge of template. As a result, formation of longitudinal crack along the edge of strut due to thermal stress and solid diffusion occur during sintering process. Besides, an inadequate thickness slurry coating layer also promoted the formation of flaws after sintering as evidenced in sintered the samples fabricated via 55% water content slurry (Fig. 2(c)) and 60% water content's slurry (Fig. 2(d)) respectively. On the other hand, the sintered samples prepared from 50% water content' s slurry (Fig. 2(b)) obtained bulky and smooth appearance with minimized flaws compared with high solid loading slurry (60%). Due to the slurry with appropriate fluidity at high shear rate and sufficient viscosity recovering during impregnation process facilitated an uniform slurry layer coating over the template [11]. This mitigated the formation of flaws after sintering.

***3.3 Physical properties.*** Fig. 3 illustrated the compressive strength of the samples fabricated from different water content slurries with corresponding total porosity and bulk density, respectively. Obviously, the compressive strength of sintered porous cordierite was directly proportional to bulk density and disproportional to total porosity. The high solid loading slurry (45% water content) attained higher compressive strength (0.0709 ± 0.0066 MPa). The high solid loading's slurry promoted thicker coating layer during impregnation process resulted the higher bulk density (0.29 ± 0.017 g/cm$^3$) after sintering. Conversely, the sintered samples fabricated from 50% water content's slurry, the compressive strength (0.0306 ± 0.012 MPa) was dramatically decrease as well as bulk density (0.2157 ± 0.015 g/cm$^3$), although the morphology analysis (Fig. 2(b)) revealed that the sintered body showed mitigated flaws structure.

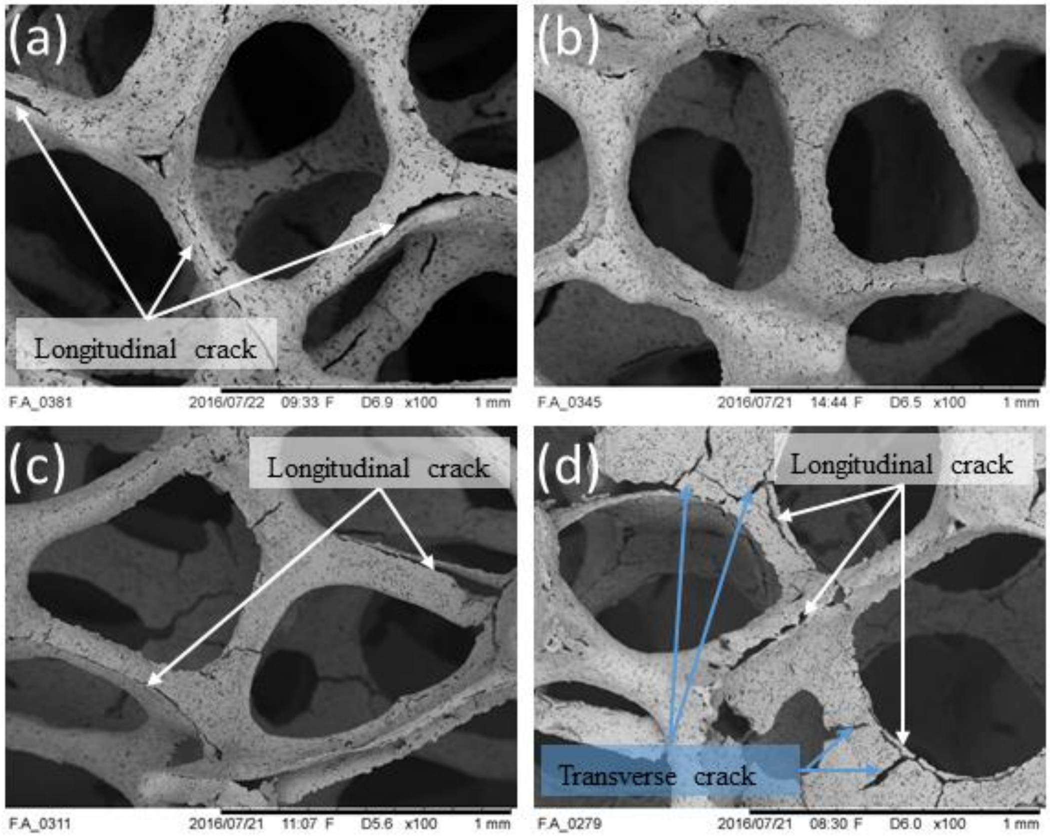


**Fig. 2** The SEM micrographic of sintered porous cordierite prepared by (a) 45, (b) 50%, (c) 55% and (d) 60 % water content slurry

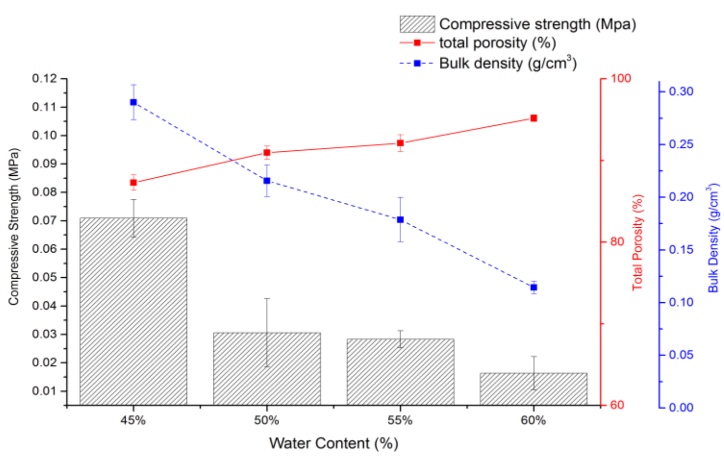


**Fig. 3** The compressive strength of the samples with the bulk density accordingly

## 4. SUMMARY

The macroporous cordierite body with pore size range 300 - 600 μm was successful fabricated from different water content slurries. The slurry with 50% water content was found as appropriate rheology behaviour to reduce the defect of the strut of the porous body due to the adequate shear thinning effect at high shear rate resulted a uniform slurry coating on the sharp edge of template during impregnation route and regained the sufficient viscosity at static condition. Hence, the formation longitudinal cracks along the strut were reduced after crystallization. However, the compressive strength of the sintered porous ceramic was highly depended to the bulk density of samples.

## ACKNOWLEDGEMENT

The authors gratefully acknowledge the financial support from the fundamental Research Grant Scheme (FRGS 900300495) Universiti Malaysia Perlis and equipment support from ADTEC Taiping, Malaysia.

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