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## A Comparative Study of Green Alumina Foams Prepared by Microwave and Conventional Drying

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### Abstract

*Drying is one of the important processes in ceramic fabrication. A comparative study on physical and mechanical properties of green alumina foams prepared by conventional and microwave drying (MWD) techniques has been done. The alumina-zeolite foams were fabricated through polymeric sponge method. Linear shrinkage of green alumina foams which gone through MWD was lowered by 50% compared to the conventional drying method. The equivalent value of dry moisture content for sample dried at 80 °C for 6 hrs in an oven could be achieved by MWD for only 7 minutes. Strength of green alumina foams was significantly being improved through MWD technique. The increment in flexural and compressive strength was found to be 14% and 39% respectively. Besides that, the shapes of alumina foams were maintained in most of the structures. In this study, the results revealed that MWD has successfully given an enhancement to the properties of green alumina foams.*

*Key words:* Microwave drying, Green alumina foam, Strength, Linear shrinkage, Moisture content

### 1.0 Introduction

Ceramic foams have been found in many applications including catalyst supports, thermal insulation, fuel cell electrodes, impact absorbing structure and also have become increasingly popular in the application as filters media for diesel emission, molten metals etc <sup>(1-3)</sup>. Such bulk applications due to its inherent properties, however, require a thorough control of the processes involve in the ceramic foams production. Drying is one of the most important processes and much more complicated than drying other objects because green ceramic bodies typically exhibit shrinkage during firing. This shrinkage can lead to cracking and loss of acceptable quality in the ceramic final products <sup>(4)</sup>.

Drying can be defined as a process of removing water from an unfired ceramic object or raw material in the green or as-formed state or in the as-received state. Drying will be accomplished by supplying energy to the ceramic in order to complete evaporation. Drying can be done in air, oven or by microwave heating <sup>(4)</sup>. There have been many attempts throughout history to increase the rate of drying of ceramic articles in order to shorten the cycle which may up to more than 30 hours, as is typically employed in normal convection processes. A significant enhancement of manufacturing speed and improvement of productivity of ceramic fabrication are needed to reduce the drying period <sup>(5)</sup>. The speed up of drying process can lead up to the reduction of energy requirements in processing. However, the characteristics of ceramic body need a close monitoring as it could be affected by the defects such as crack, warpage and density distribution comes from rapid drying <sup>(4)(5)</sup>.

Several methods have been utilized for the purpose of ceramic drying starting with conventional drying method to the advanced or faster drying technologies such as MWD. Conventional air drying typically takes between 8-24 hrs. During this time there is a possibility of deshaping of the green body. Oven drying takes around 15 min to 6 hrs and here also the possibility of deshaping is difficult to avoid. Whereas MWD takes around 5-30 mins depending on the power and the amount of loading, the shapes of the object is maintained in most of the structures. Microwave processing of materials offers the potential for reducing production times for ceramic materials <sup>(4)</sup>. Microwave energy has proven to be an efficient and reliable form of heating for a wide range of industrial processes. Over the past years, the MWD method is becoming more and more important. This phenomenon is proved by several studies deal with the advantages provided by MW especially on drying acceleration <sup>(3)(5-8)</sup>. Meanwhile, other studies deal with the possibility of predicting higher reduction in drying times through the optimization of microwave and convection parameters <sup>(9)(10)</sup>.

In this study, the influence of MWD time onto the physical and mechanical properties of green alumina foams prepared by polymer-sponge method are investigated. Comparison between the properties of green alumina bodies which dried through MW and conventional method is carried out in order to obtain further and explicit understanding of the drying mechanism.

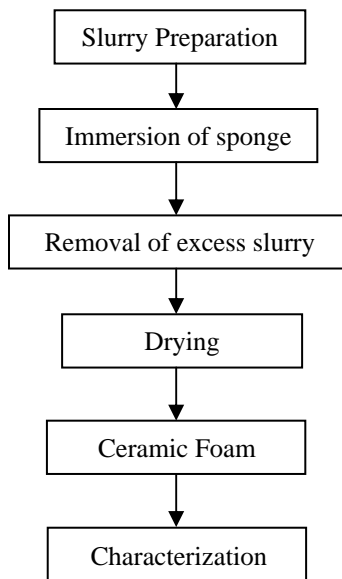
## 2.0 Experimental Procedure

### 2.1 Slurry Preparation

Ceramic slurry was prepared from a mixture of commercial alumina powder with particle size below 50  $\mu\text{m}$  and zeolite of clinoptilolite. Ovalbumin and 10% of water acted as the carrier and binder in the slurry. Other additive such as polyvinyl acetate was also used. The mixture was stirred to get the final slurry. After the slurry was prepared, the next step involves the impregnation of polymeric sponge with the slurry. Polyurethane sponge was compressed to remove air and then immersed into the slurry and allowed to expand. This process was repeated to achieve the desired ceramic loading. Excess slurry needs to be removed from the infiltrated sponge.

### 2.2 Drying Conditions

After the desired loading, the infiltrated sponge was dried. The drying was carried out in MW at 3, 5, 7, 9 and 11 minutes to evaporate all free water and most bound water. A common domestic microwave oven with 800 watts was used for drying process. The temperature and rate of heating were not controlled as those parameters were fixed for this microwave. The standard procedure for green foams preparation is shown in Fig. 1. Ceramic foams which gone through conventional oven drying for 6 hours at 80°C were prepared as control sample for comparison purpose.



**Fig. 1. Polymer sponge fabrication process of ceramic foam**

### 2.3 Properties Characterization

The green alumina foams were characterized by their physical and mechanical properties such as moisture content, linear shrinkage, flexural strength and compressive strength. In this study, percentage of moisture removal after drying was measured. The calculated moisture removal (% dry) referred to the dryness of dried sample. The linear shrinkage of foams after drying was determined using the following equation:

$$\text{Linear shrinkage} = \frac{l_o - l_f}{l_o} \times 100\% \quad (1)$$

where  $l_o$  is the length of foam before drying and  $l_f$  is the length of green or sintered foam. The length was measured by vernier caliper.

Flexural strength was measured through three-point bend test by using Autograph AG-I Universal Testing Machine with applied load at a cross head speed of 1.0 mm/min. Universal Testing Machine was also utilized to investigate the foams compressive strength. Microstructure analysis was conducted by using a scanning electron microscopy, LEO model JSM-6380LA operated at 15 kV.

### 3.0 Results and Discussion

#### 3.1 Moisture Removal

Drying under MW technique has managed to improve the dryness of the samples about 2% to 5% in 7 to 11 minutes of drying compared to the conventional oven drying. Results in Table 1 shows that 7 minutes of drying in MW is capable to produce slightly higher dried moisture of green foams compared to the foams which has been dried for 6 hours in conventional oven.

**Table 1. Moisture removal (% dry) of green alumina foams**

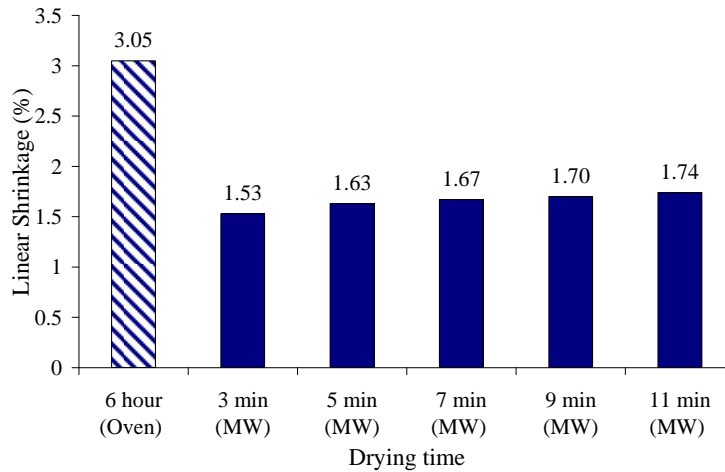
Drying time	6 hrs	3 mins	5 mins	7 mins	9 mins	11 mins
Moisture removal (% dry)	35.55	34.54	34.89	36.30	36.34	37.30

The driving force for MWD is similar to conventional air drying and oven, but the notable physical difference is that there is internal heat generation with MW. Water in the green bodies absorbs the MW radiation through its cross section and this absorption generates heat through the bulk of the part that can be used for evaporation. In contrast to conventional drying, the heat for evaporation is transferred to the surface of the part by convection and then through the bulk of the part by conduction which needs longer time to complete the drying process<sup>(4)</sup>.

#### 3.2 Linear Shrinkage

Shrinkage in ceramic bodies is due to the removal of water during drying process which brought to the decrement in interparticle separation<sup>(11)</sup>. Shrinkage is one of the physical defects in ceramic products caused by drying process. Fig. 2 exhibits the green foams dried in MW gave linear shrinkage in the range of 1.53% to 1.74%. Whereas, the dried foams from conventional drying presented higher linear shrinkage which is 3.05%. Theoretically, for most plastic formed ceramic product, the linear shrinkage is about 2-4%<sup>(4)</sup>. Therefore, the MW drying has been able to reduce the drying linear shrinkage nearly 50% compared to the shrinkage caused by conventional oven.

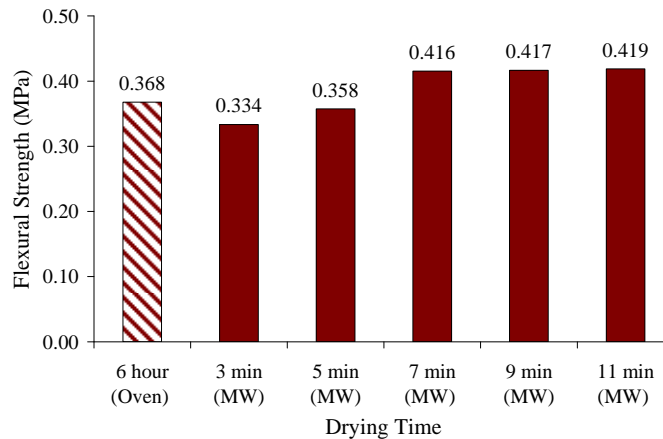
The drying process in MW is more uniform. It occurred over the part's cross section due to the penetration depth of the microwave radiation and allows the shrinkage to take place from the centre of the foam and progress to the surface. As a result, rapid drying favoring minimized shrinkage in many ceramics. Unlike conventional drying, which acts only on the surface of the foam, it generates shrinkage from surface inward which cause the pore structure shrinks and this bring to higher shrinkage when the remaining water vapour need to be removed from more restricted path<sup>(4)(12)</sup>.



**Fig. 2 Linear shrinkage of green foams dried in conventional and MW oven with different drying time**

### 3.3 Flexural Strength

The effect of different drying time in MW oven on the flexural strength of green foams is depicted in Fig. 3. The increment of drying time in MV oven from 3 minutes to 11 minutes has raised the flexural strength between 0.334 MPa to 0.419 MPa. 7 minutes of drying in MW has significantly improved the strength of green foams about 13% compared to the 6 hours of conventional drying. Through the ability of microwave to heat uniformly, the foams can be dried evenly and give a compromise to the structural properties.



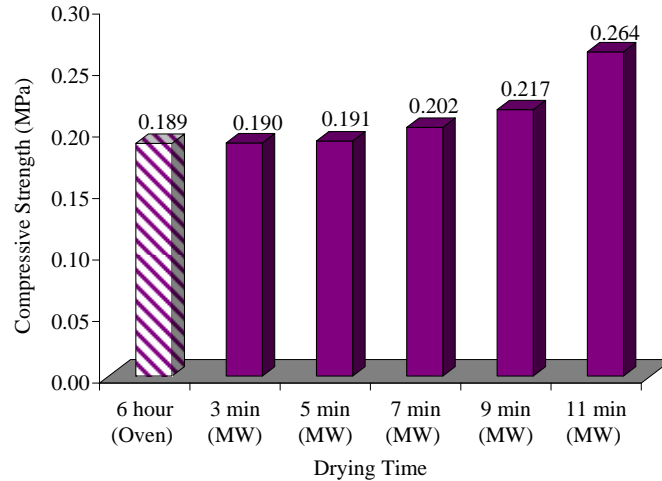
**Fig. 3 Flexural strength of green foams dried in conventional and MW oven with different drying time**

### 3.4 Compressive Strength

The results of compressive strength in Fig. 4 displays almost similar trend as the findings in flexural strength. 3 to 5 minutes of MW drying seems not to give considerable effect on the foam's compressive strength. However, for drying time at 7 minutes and above in MW, the flexural strength of the green foams can be enhanced about 7% to 39% compared to the conventional drying.

Compressive strength of green foams is influenced by its moisture content. The higher removal of moisture in ceramic bodies during drying process can help to improve its green strength. Additionally, the ability to

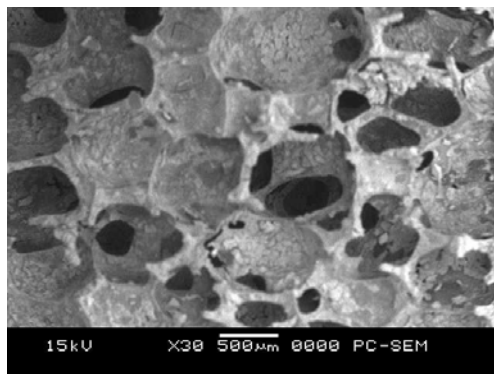
maintain lower shrinkage of green foams during MW drying may be one of the reasons that contribute to the increment of the compressive strength of the green foams. Other than that, the bonding of particles, particles alignment and pores arrangement or distribution in the green bodies also has an effect on the compressive strength.



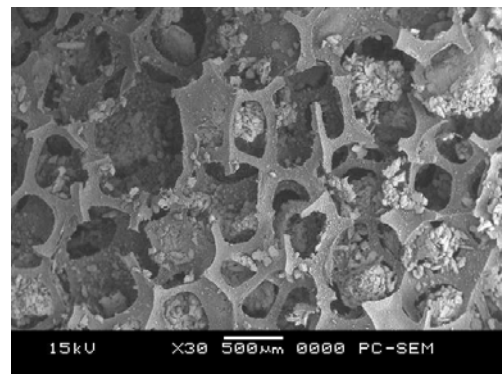
**Fig. 4 Compressive strength of green foams dried in conventional and MW oven with different drying time**

### 3.5 Microstructure Analysis

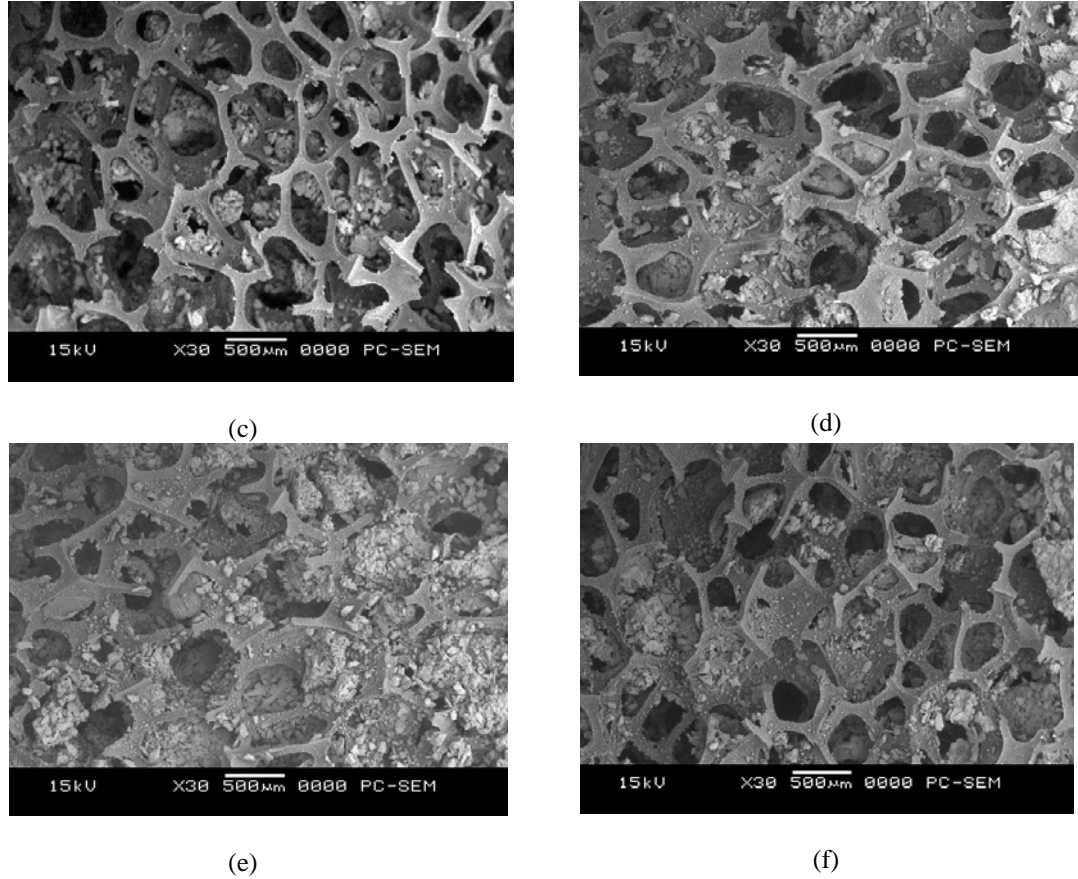
The SEM micrographs in Fig. 5 present the microstructure of green foams drying in conventional and MW oven. In conventional oven drying, the particles are less agglomerated to each other and covered the sponge's struts. Meanwhile, as can be seen from the micrographs, MW offered more uniform drying in the pore structure of the sponge. The particles appeared to be more agglomerated and elimination of sponge's struts before it is replaced with the ceramic structure during sintering has started to occur uniformly in the green foams. The structure development in drying process is known to affect the growth of grains size as well as the final properties of the sintered ceramic structure<sup>(13)(14)</sup>. This explains the importance in monitoring and controlling the drying process for green bodies.



(a)



(b)



**Fig. 5 SEM micrographs of green foams drying in conventional oven (a) 6 hrs and MW (b) 3 mins, (c) 5 mins, (d) 7 mins, (e) 9 mins, (f) 11 mins.**

#### **4.0 Conclusion**

Due to the high frequency and short wavelength of MW radiation, there are several advantages to MWD such as shorter drying time, more uniform drying and greater energy efficiency. The application of MW heating in drying process of alumina foams has high potential to improve the green foams characteristics. For only 7 minutes of MWD, it is sufficient to reduce the linear shrinkage about 50% and produce green alumina foams with flexural and compressive strength of 0.416 MPa and 0.202 MPa respectively. At the same time, the shapes of alumina foams are maintained in most of the structures. The findings of this study revealed that MWD has successfully given an enhancement to the properties of green alumina foams.

#### **5.0 Acknowledgement**

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