

Microwave Drying Effects on the Properties of Alumina-Zeolite Foam

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Abstract

The effects of microwave drying on the properties of alumina-zeolite foam were investigated. A polymeric sponge method was implemented for the foams preparation. Alumina powder with the particle size below 50 μm was used in this study. The samples were dried in microwave oven for about 7 minutes before went through sintering process at 1350°C. The results showed a small linear shrinkage of green and sintered alumina foams. The microwave drying has managed to produce porous foam bodies with strength of 2.11 MPa and porosity more than 40%. The obtained properties exhibited higher values compared to the alumina foams that have gone through conventional drying process. The implementation of microwave drying has significantly reduced the drying time of green bodies and together improved the properties of alumina-zeolite foams. This alumina zeolite foam is aimed to be used as a filter in automotive application.

KEYWORDS: microwave drying, strength, porosity, foam

1. Introduction

Ceramic foams are widely used in applications such as 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 [1-3]. 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 [4].

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 [4]. 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 [5]. The speed up of drying process can lead up to the reduction of energy requirements in processing.

However, the characteristics of sintered body need a close monitoring as it could be affected by the defects such as crack, warpage and density distribution comes from rapid drying [4-5].

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 radio frequency (RF) drying and microwave (MW) drying. 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 MW drying 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 [3, 5-8]. Meanwhile, other studies deal with the possibility of predicting higher reduction in drying times through the optimization of microwave and convection parameters [9, 10].

In the present work, the changes in physical and mechanical properties of alumina-zeolite foams prepared by polymer-sponge method due to different approach of drying process are discussed. This study also examines the capability of MW drying technique to provide an enhancement onto the properties of sintered body.

2. Experimental Procedure

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2.1. Slurry Preparation

A commercial alumina powder with particle size below 50 μm and zeolite of clinoptilolite type were used for the preparation of ceramic slurry. Ovalbumin and 10% of water acted as the carrier and binder in the slurry. 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 required 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. A common domestic microwave oven with 800 watts was used for drying process. The drying period about 7 minutes appeared to be sufficient to evaporate all free water and most bound water. The temperature and rate of heating were not controlled as those parameters were fixed for this microwave.

After drying, the green bodies were sintered in a temperature programmable furnace. The sintering involved two stages, the first stage was to burn out the organics from the slurry and followed by densification of ceramic bodies at 1350°C with the heating rate and holding time of 2°C/min and 2 hours respectively. The typical scheme of above procedure is depicted 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.

2.3. Properties Characterization

The green and sintered ceramic foams were characterized by their physical and mechanical properties such as flexural strength and linear shrinkage. 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. The linear shrinkage of foams after drying and sintering 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.

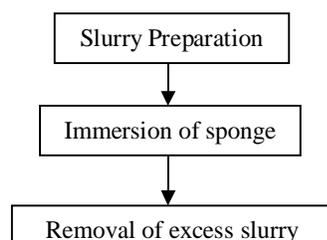


Fig. 1. Polymer sponge fabrication process of ceramic foam

Mercury porosimetry (Model AutoPore IV 9500) was used to measure the porosity and bulk density of the ceramic foams. Meanwhile, microstructure analysis was conducted by using a scanning electron microscopy, LEO model JSM-6380LA operated at 15 kV.

3. Results and Discussion

3.1. Linear Shrinkage

The drying process is the major source of defects in most ceramic products. These defects range from visible defects (cracks) to a reduction in physical properties. The consequence of water removal in drying is shrinkage.

Fig. 2 shows the different in linear shrinkage of green and sintered foams between drying in conventional and microwave oven. Ceramic foams which gone through drying process in conventional oven present higher linear shrinkage compared to foams from microwave drying. Typical linear

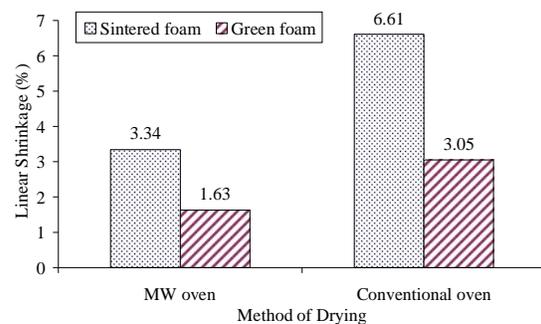


Fig. 2. Linear shrinkage of green and sintered foams from different method of drying. shrinkage is about 2-4% for most plastic formed ceramic product [4]. Comparable to the theoretical

statement, microwave drying gave a lower shrinkage of the green and sintered foams which are 1.63% and 3.34% respectively. Therefore, the microwave drying has managed to reduce the linear shrinkage almost 50% compared to the shrinkage caused by conventional oven.

In microwave, the drying process is more uniform 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. 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 [4, 11].

3.2. Flexural Strength

Fig. 3 presents the effect of different drying methods onto the flexural strength of ceramic foams. Microwave drying has significantly improved the flexural strength three times higher than the strength of the sintered foams dried by conventional oven. The flexural strength of the sintered foams from microwave drying is 2.11 MPa. On the other hand, drying process seems not to give major impact on the strength of green foams as both drying process exhibits nearly similar results.

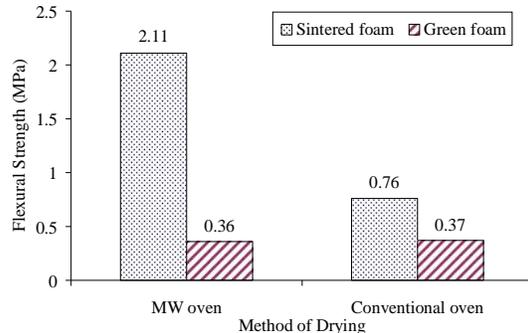


Fig. 3. Flexural strength of green and sintered foams from different method of drying

Through the ability of microwave to heat uniformly, the foams can be dried evenly and give a compromise to the structural properties. The microwave drying able to exhibit higher flexural strength sintered foams in spite of its high porosity.

3.3. Porosity and Density

Microwave drying has produced higher porosity sintered foams compare to conventional drying. Table 1 displays the results of porosity and density test for sintered foams. The shrinkage of ceramic bodies during sintering will give an influence to the porosity. An increment in shrinkage tends to cause a

reduction of porosity in the sintered bodies [12]. This trend explains the reason behinds the lower percentage of porosity obtained from the sintered foams dried in conventional oven.

Table 1. Porosity and density of sintered foams from different drying method.

Property \ Drying	Porosity (%)	Density (g/cm ³)
Microwave oven	42.25	1.78
Conventional oven	37.66	2.05

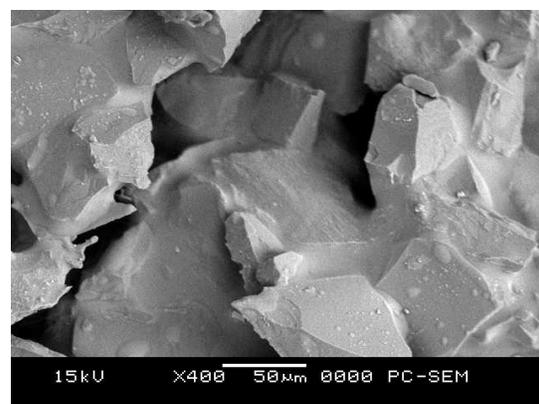
Density and porosity are interrelated properties. The finding reveals a slight declined in density of sintered foams dried by microwave oven. Sintered foams from conventional drying give a density of 2.05 g/cm³, whereas, microwave drying produces foams with 1.78 g/cm³. Theoretically, ceramic bodies with higher porosity normally will give a low density. The declination of bulk density is due to the increment of porosity in ceramic foams.

3.4. Microstructure Analysis

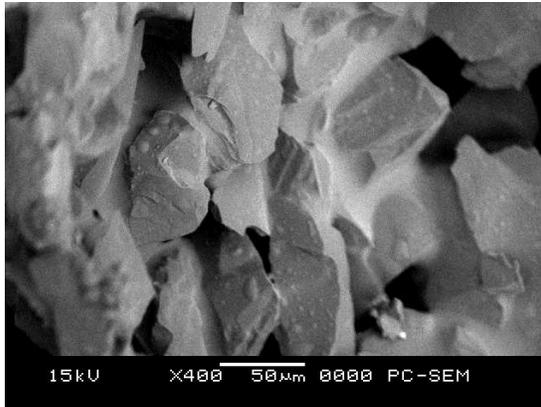
The SEM micrographs in Fig. 4 present the microstructure of sintered foams. Larger grains are obtained in the sintered foams from conventional drying. The development of grains size in the ceramic structure leads to more effective pores shrinkage [13-14]. On top of it, this is one of the reasons for the reduction of porosity and flexural strength of sintered foams dried in conventional oven.

Opposite to the microstructure developed from the conventional drying, the application of microwave has successfully produced smaller grains in the sintered foam structure. Generally, this contributes to the growth of porosity and enhancement of the foams mechanical properties such as flexural strength.

Meanwhile, Fig. 5 (b) shows a uniform microwave drying of green foams is achieved in the pores structure of the sponge.

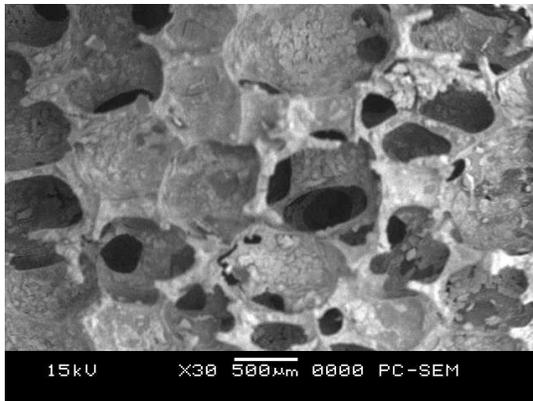


(a)

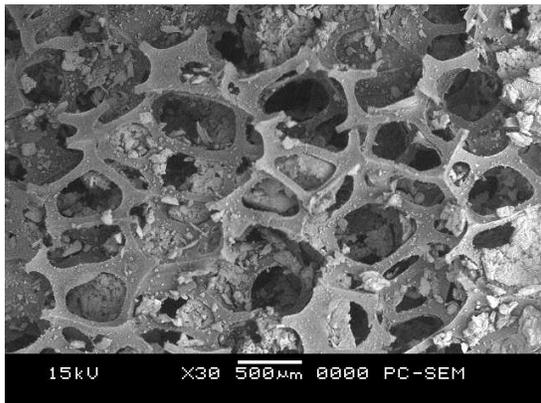


(b)

Fig.4. SEM micrographs of sintered foam from (a) conventional drying and (b) microwave drying



(a)



(b)

Fig. 5. SEM micrographs of green foam from (a) conventional drying and (b) microwave drying

4. Conclusion

The application of microwave heating in drying process of alumina-zeolite foams has high potential to improve the foams characteristics. Due to the high frequency and short wavelength of microwave radiation, there are several advantages to microwave

drying such as more uniform drying and greater energy efficiency. From this study, 7 minutes of microwave drying is sufficient to reduce the foams linear shrinkage about 50% and improve the flexural strength up to three times higher than foams which gone through conventional drying process. At the same time, the higher porosity of the final sintered bodies could be maintained. The outstanding properties of the sintered foams from microwave drying allow it to be used in a wide range of application including as a filter media in automotive area.

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