Fabrication and Characterization of Porous Mullite Ceramics

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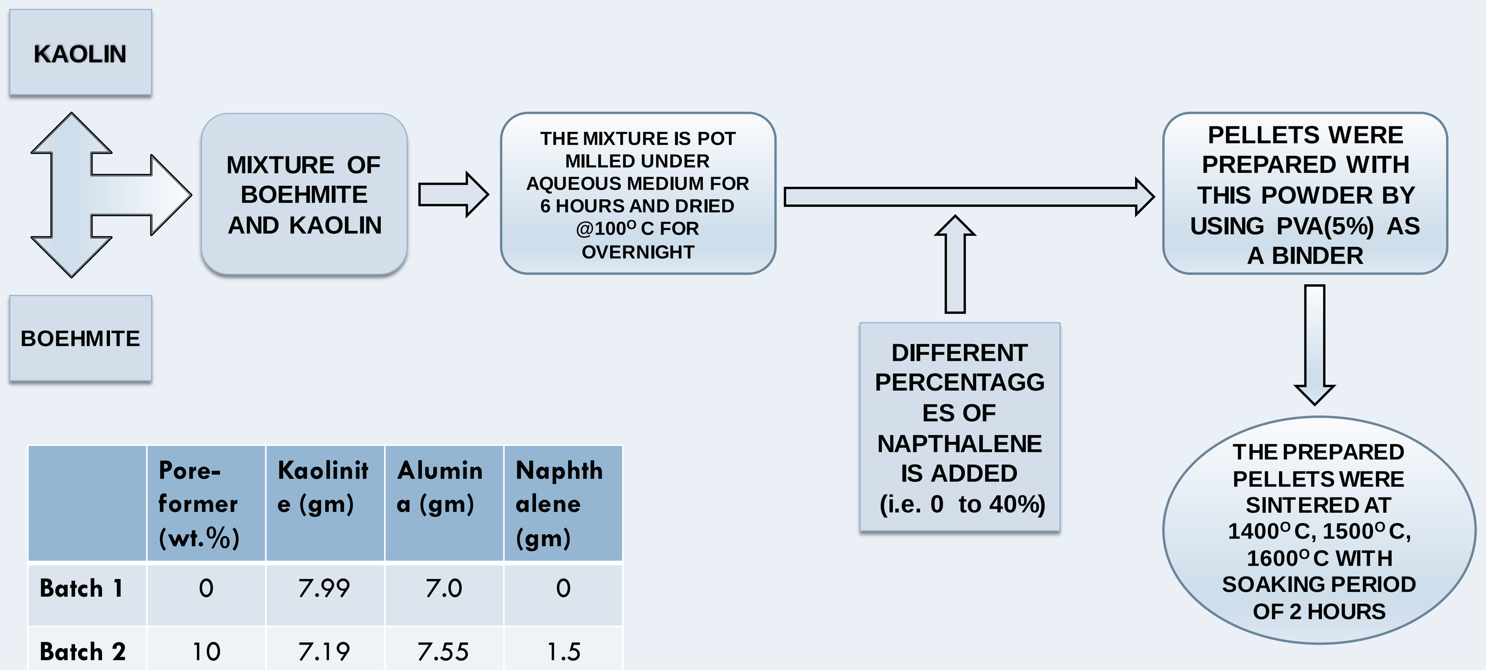
ABSTRACT

Porous mullite was fabricated from kaolin and alumina using naphthalene as a pore-former. For different wt.%of naphthalene addition, the apparent porosity (%) was analysed. The mechanical strength was estimated through flexural strength and diametral tensile strength. Fabricated pellets were sintered at 1400ºC, 1500ºC, 1600ºC to study the effect of pore former on phase formation and porosity distribution through X-Ray diffraction study and FESEM microstructure analysis. The analysis showed 39.79% of porosity in the case of 20wt.% naphthalene addition with flexural strength of 21.93MPa. This pointed towards the application of this product as a membrane support material.

INTRODUCTION

- Porous mullite ceramics are attracting interest due to wide range of application in thermal insulators, catalytic supports, gas/liquid filters, separation membranes, high temperature structural materials, and kiln furniture etc.
- To achieve porous ceramic with a tailorable porosity and tuneable pore size, several methods like ice-template spray drying, emulsification technique, sacrificial template etc. have been developed in the past years.
- Here sacrificial templated method is implemented, due to the easiness of the process. In this method sacrificial agents like graphite, naphthalene etc. are used to tune the pore size and porosity of the structure
- Here, kaolin ($Al_2O_3.2SiO_2.2H_2O$) is used as primary raw material. But despite from the chemical formulae of mullite ($3Al_2O_3.2SiO_2$), kaolin has deficiency of alumina to form stoichiometry mullite structure. Hence required amount of boehmite is added from outside to make it stoichiometric

MATERIALS AND METHODOLOGY



	Pore-former (wt.%)	Kaolinite (gm)	Alumina (gm)	Naphthalene (gm)
Batch 1	0	7.99	7.0	0
Batch 2	10	7.19	7.55	1.5
Batch 3	20	6.39	5.60	3
Batch 4	30	5.59	4.90	4.5
Batch 5	40	4.79	4.20	6

RESULTS AND DISCUSSION

XRD ANALYSIS

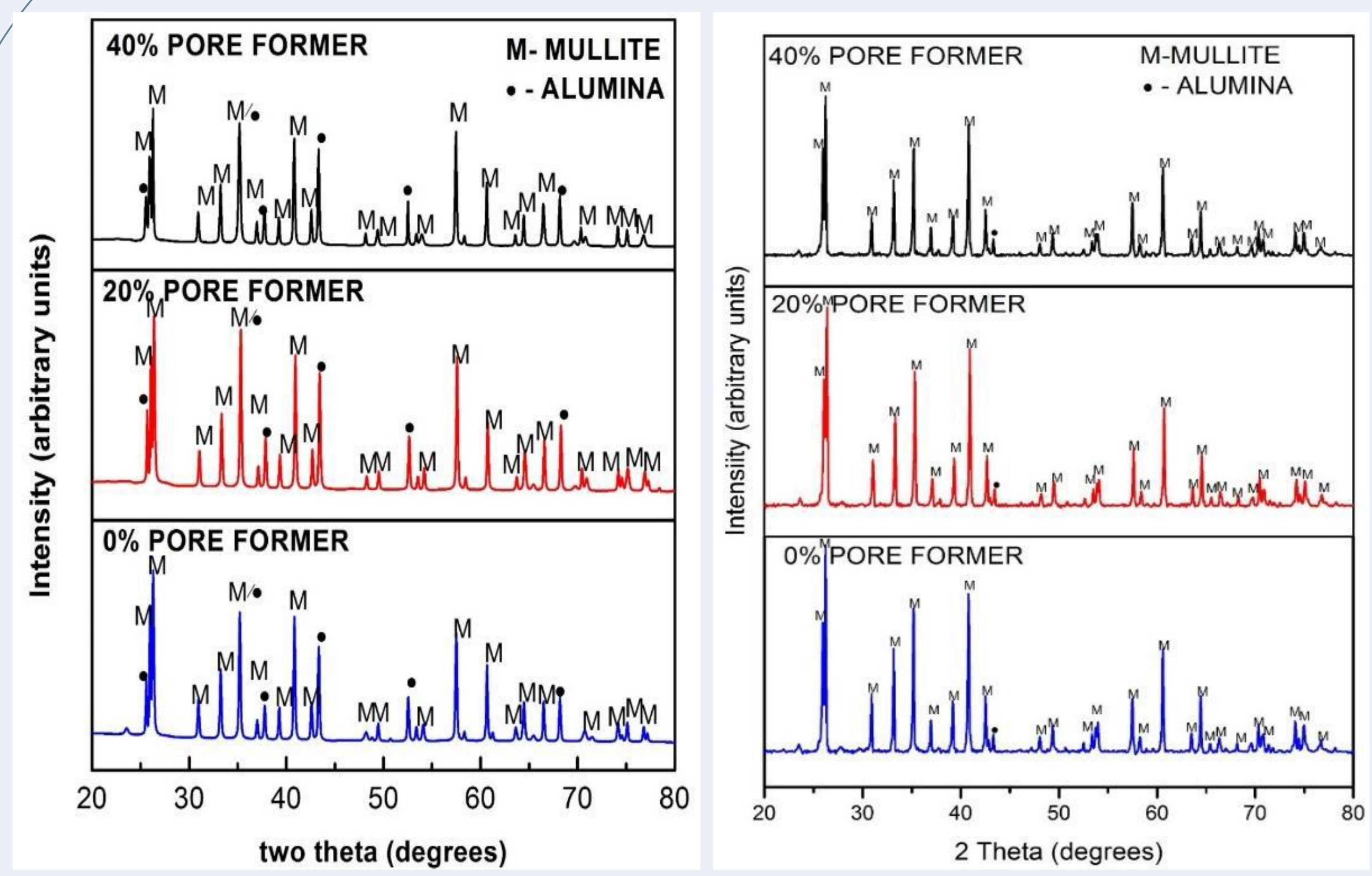


Figure 1. XRD analysis of sample sintered at (a). 1400oC and (b). 1600oC with different weight percentages of pore formers added.

- The XRD analysis of the 1400ºC and 1600ºC sintered samples are shown in Figure 1(a) and (b). As per Figure1(a) most of the peaks are corresponding to mullite phase along with some prominent peaks of α-alumina.
- Here with the addition of pore former, alumina peak intensity increases i.e., mullite phase formation is hindered by the addition of pore former. For 1600ºC sintered sample complete mullite phase formation is observed with negligible existence of α-alumina phase (Figure 1(b)).

APPARENT POROSITY AND MICROSTRUCTURE.

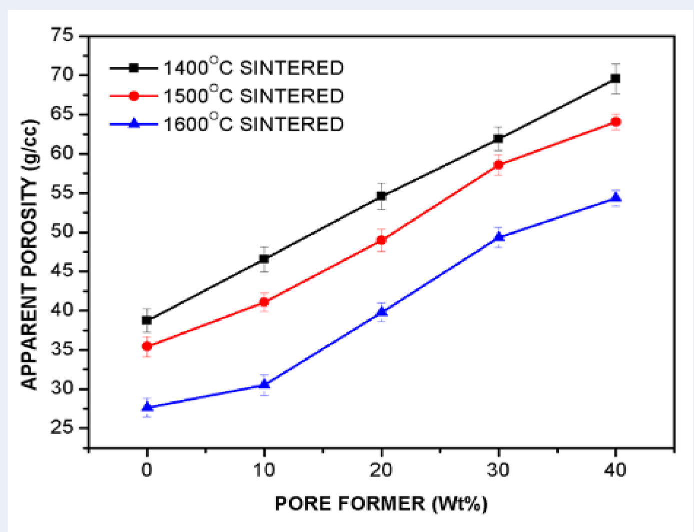


Figure2. Apparent porosity as a function of temperature and pore-former

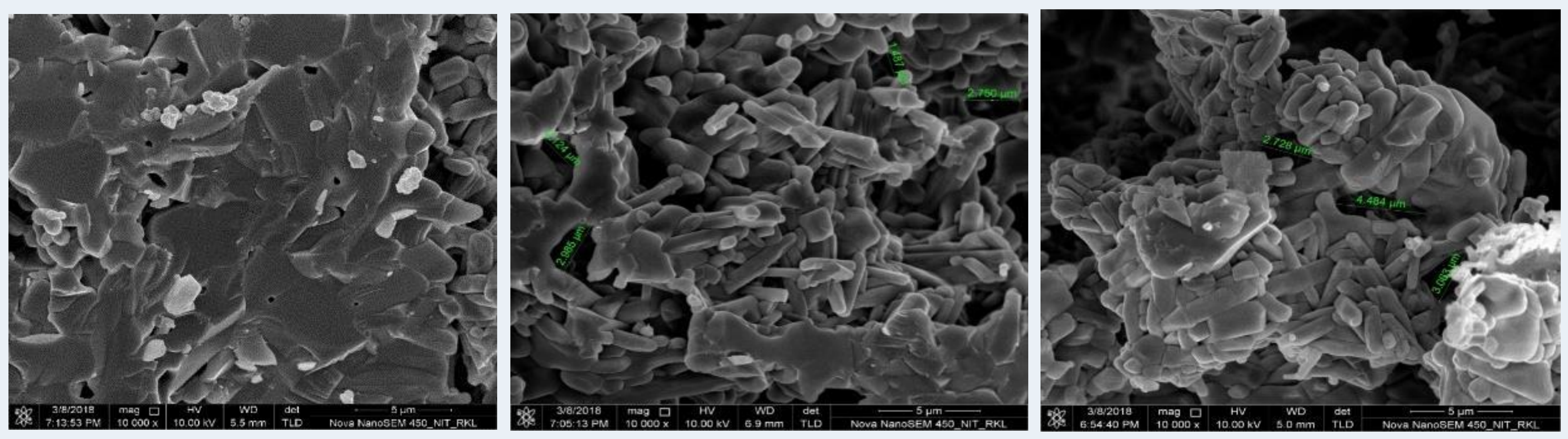


Figure 3. FESEM micrograph of sample surface sintered at 1600oC (a) 0 wt.% naphthalene (b) 20 wt.% naphthalene (c) 40 wt.% naphthalene

- From the Figure 2 it is evident that as the temperature is increasing the apparent porosity is decreasing due to sintering effect and as the naphthalene (wt.%) increases the apparent porosity increases.
- Figure 3(a, b, c) shows the microstructure of the samples sintered at 1600ºC containing different weight percentages of pore former. Here, better densification is observed in batch 1 compared to batch 3 and 5. The higher amount of pores present in batch 3 and batch 5 effect mullite phase formation because in presence of pores contact area between silica and alumina decreases which causes less diffusion and effect mullite phase formation.

FLEXURAL STRENGTH AND DIAMETRICAL TENSILE STRENGTH

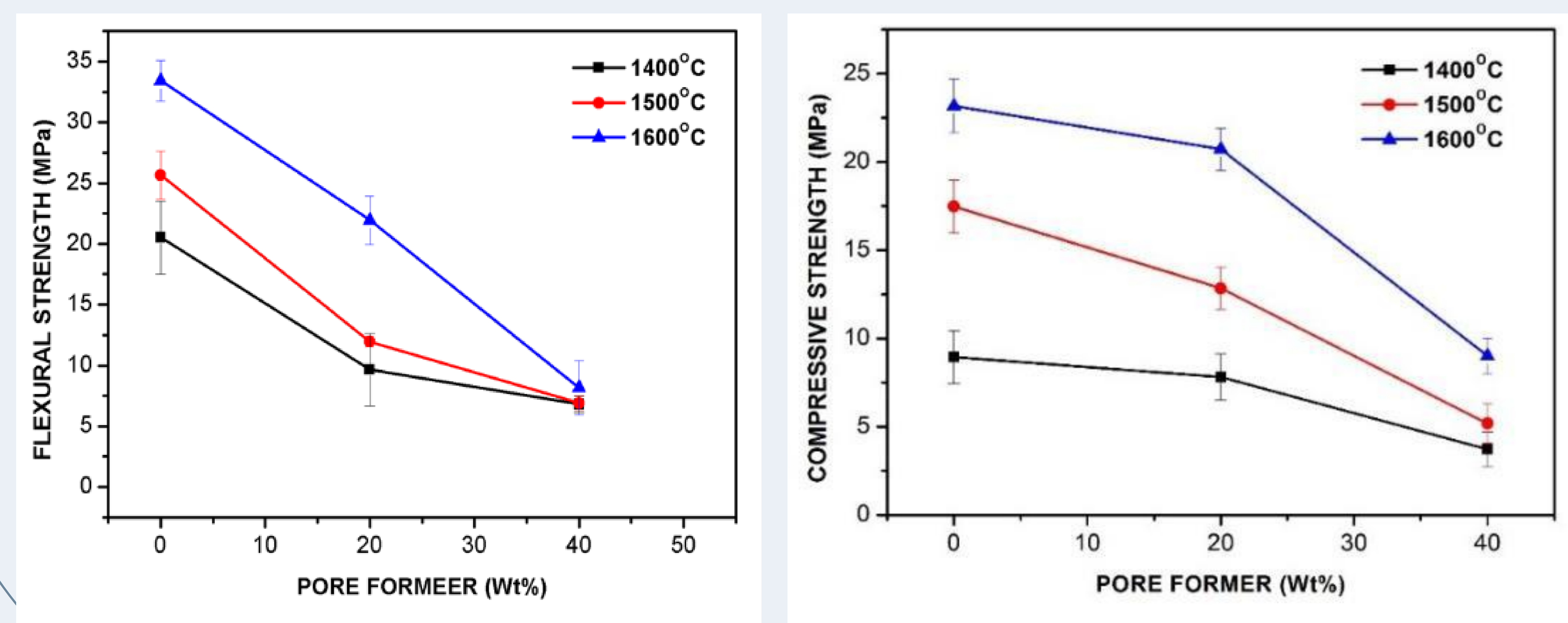


Figure 4. (a)Flexural strength (b) Diametrical tensile strength

- The mechanical properties were analysed by flexural strength (i.e. three-point bending test) and diametrical tensile strength analysis. Three-point bending strength shows that increase in sintering temperature increases the flexural strength but this enhancement of strength is negligible for 40 wt.% naphthalene containing pellets indicating highly porous nature of the samples.
- From Figure 4(a) for 20 wt.% naphthalene at 1600ºC strength was quiet good with a porosity of 39.79%.
- Similarly, in Figure 4(b) Diametrical tensile strength shows that the much decrease in strength in the 20wt.% to 40wt.% indicates weakening of sample due to the presence of higher amount of porosity in the range of 54 to 70%.

CONCLUSION

Porous mullite from clay and boehmite is formed using naphthalene as a pore former. The sample sintered at 1600ºCwith 20wt.% naphthalene exhibited apparent porosity of 39.79% with flexural strength of 21.93MPa and diametral tensile strength of 20.729MPa. From XRD and microstructure analysis it was evident that increase in sintering temperature favours the formation of mullite structure whereas addition of pore former delayed the mullitization process. microstructural analysis also indicated the presence of distributed pores with increasing naphthalene content.

REFERENCES

1. F. Yang, C. Li, Y. Lin and C. A. Wang, Mater. Lett. 73, pp.36-39 (2012).
2. M. Rashad and M. Balasubramanian, J. Eur. Ceram. Soc. 38(10), pp.3673-3680 (2018).
3. J. Ma, F. Ye, B. Zhang, Y. Jin, C. Yang, J. Ding, H. Zhang and Q. Liu, 2018. Ceram. Int. 44(11), pp.13320-13327 (2018).
4. Z. Hou, B. Zhang, R. Zhang and L. Liu, Ceram. Int. 43(12), pp.8809-8812 (2017).
5. C. Y. Chen, G. S. Lan, and W. H. Tuan, J. Eur. Ceram. Soc. 20(14-15), pp.2519-2525 (2000).
6. S. Sutardi, R. Septawendar and A. Rachman, J. Ceram. Process. Res. 14, 400-404(2013).
7. G. Ruan, Z. Zhang and M. Yin, Rare Metals 30, 506-510(2011).
8. S. Bhattacharyya and P.S. Behera, Appl. Clay Sci. 146, 286-290 (2017)
9. P. S. Behera and S. Bhattacharyya, Effect of processing route for preparation of mullite from kaolinite and alumina. AIP Conference Proceedings 1953, 090047 (2018