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PHASE CHANGES DURING MICROWAVE SINTERING OF FLYASH

B.B. Kar[1], T.Panda[2],S.Dash[1] and S.M.Mustakim[3]

[1]Professor, School of Applied Science, KIIT University, Bhubaneswar, Odisha, India

[2]Professor, GIFT, Bhubaneswar, Odisha, India

[3]Technical Officer, IMMT, Bhubaneswar, Odisha, India

Abstract : Class F flyash sample is subjected to roasting both by conventional process as well as microwave technique. The phase change during roasting with microwave heating has been investigated by XRD method and the chemical composition has been monitored continually by quantitative as well as AAS method. The results show that the flyash samples couple well with microwave and the temperature can reach approximately 1000^0 C in 7 minutes. The phase change in iron has been observed along with SiO_2 and Al_2O_3. By proper control of the roasting temperature flyash can be sintered into a reasonably strong and relatively porous glassy ceramic material in a short period of time using microwave technique.

Keywords : Flyash, microwave roasting, phase changes, porous material.

I. INTRODUCTION

Flyash, generated power plants has become a great challenge for researchers and scientists for its disposal utilization and treatment. Fly ash is a fire, light, glassy substance, consisting mostly of silica, alumina, iron oxide, calcium oxide etc. Flyash consists of tiny spherical particles. During conventional heating, as the heating rate increases, the outer surface get heated immediately and the inner material get heated after long time. Due to this reason, non uniform heat distribution takes place due to which the aggregations of flyash particles don't take place properly(1-8). In composition, microwave roasting has been studied for flyash sample. As microwave generates volumetric heating thus, has better aggregation capacity.

II.EXPERIMENTAL

Microwave roasting was carried out using WD 800B Philips microwave over at a frequency of 2.5 GHz at 750 W power level .The experiments were carried out with x-ray diffraction to analyses the material composition of flyash samples before and after microwave roasting. The fly ash from power plant used in the study is dried at 9^0 C FOR 6 hrs to microwave the absorbed moisture. The chemical composition of fly ash is represented in Table 1.

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Table-1 : Chemical composition of flyash (Class F –flyash)

Constituents	SiO_2	H_2O_3	Fc_2O_3	CaO	MgO	Na_2o	K_2O	SO_3
WT%	51.5	19.3	16.1	6.7	1.6	1.0	2.1	1.98

III. MICROWAVE ROASTING OF FLY ASH

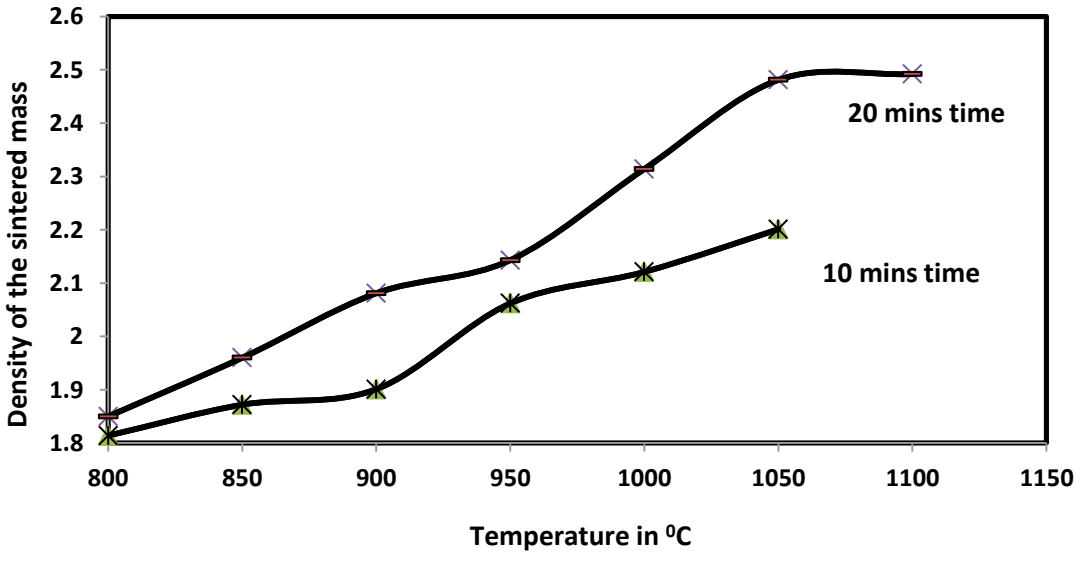
Microwave roasting was carried out using class F flyash. There is various phase changes in iron oxide which can be represented in the following sequence as Fe_2O_3 , Fe_3O_4-FeO . SiO_2 during microwave roasting leads to the formation of $FeSiO_3$ and CaSiO3 partially. In order to study the role of particle size during microwave roasting and phase changes in fly ash , experiments were carried out using different size of flyash i.e. 250mm , 500 mm and fine and pellets with different diameters .

IV. RESULTS & DISCUSSIONS

Effect of Temperature during microwave roasting

The results of microwave roasting of fly ash has along with increase in roasting temperature has been represented in Figure-1 . The first heating behavior of the sample may be attributing to the presence of silica, which absorbs microwave adequately and mixed in the other dielectricmaterial, which facilitate the coupling withmicrowave. In addition to it , presence of iron as Fe_2O_3 changes its phase to Magnetite (Fe_3O_4) which further act as an hyperactive material in microwave . The presence of magnetite phases enhances the heating behavior of the fly ash sample.

Figure-1 Effect of Temperature on microwave roasting of fly ash



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Effect of Particle Size On Microwave Roasting

The effect of particle size an microwave roasting has been studied during the investigation fire particle along with pellet of various diameter in are taken into consideration .It has been observed that phase changes are maximum upto the pellets having 8mm diameter after which the uniformity in temperature and phase transformation. During the preparation of pellets, 10wt% water is added to the fly ash fire and pellets have been prepared with diameter. These pellets are subjected to compressor for studying their green strength. The average density of different pellets is being represented in Table-2.

Table-2 Average green strength of the pellets

Pellet size in mm	Average Green Strength in gm/cm^2
2mm	1.42
4mm	1.48
6mm	1.56
8mm	1.65
10mm	1.74
12mm	1.79
14mm	1.84
16mm	1.88

The addition of water favors during the absorptum of microwaves of the fly ash sample at low temperature.

Roasting of Fly Ash

Microwave roasting was carried out in a microwave at 245 GHz, which has been converted from a commercial microwave with a turn table of 1200 W. In order to carry out roasting, a zirconium cylinder is placed at the centre of the Table. The details are represented in figure-1. During each experiment 10 pellets were placed inside the cylinder (of each diameter). Zirconium Cylinder in used for such studies as, Zirconium is a good absorber of microwave radiations which accelerates the preheating process of the pellets. Secondly as a thermal insulator it will not radiate the heat outside the cylinder and will maintain a good roasting environment. During the experiment, sufficient care was taken to assure that temperature is well controlled, radiation is continuous and uniformity is maintained to provide heat to each pellet under consideration. Temperature was measured using a (Pt-Pt-Rh) Thermocouple, inserted from the top of the microwave and touches exactly the heating surface of the cylinder. In order to study the comparative phase changes between microwave roasting and conventional sintering, the pellets were sintered in a carbolated furnace and the

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heating rate was maintained similar to that at the microwave roasting. In this study, the samples were kept in graphite credible. The assembly is shown in figure-2 .The sintered samples densities were measured by weighting methods. The Tensile Strength [R] were tested using an universal testing machine with a loading rate of 0.4 mm /min and calculated using the mathematical relation .

$$\delta = 2P/T D\Pi,$$

δ= diametric tensile strength, P = load applied at failure, T = Thickness of the sample, D=diameter of the sample.

The structural changes and phase transformation has better studied using SEM and X-Ray diffractometry respectively.

The densities of the sintered samples are listed in Table-3 (A&B) both for conventional as well as microwave sintering. It has been observed that the density of microwave processed sample when heated from 700-1000^{0}C the density changes ranges from 1.72 to 2.38 within 2 minutes where as in case of conventional sintering the sample density was obtained when pellets were heated up to 90 minutes, within the same temperature range.

Table-3A:- Calculation of densities of the microwave sintered samples

Temperature	5 minutes	10 minutes	15 minutes	20 minutes	25 minutes
700	1.48	1.53	1.57	1.63	1.68
750	1.58	1.62	1.67	1.71	1.74
800	1.62	1.70	1.75	1.79	1.81
850	1.70	1.78	1.82	1.86	1.89
900	1.82	1.85	1.88	1.91	1.98
950	1.89	1.92	1.97	2.01	2.07
1000	1.98	2.08	2.13	2.18	2.21

Table-3B:- Calculation of densities of the conventionally heated sintered samples

Temperature	10 minutes	30 minutes	60 minutes	75 minutes	90 minutes	105 minutes
700	1.33	1.36	1.38	1.46	1.50	1.51
750	1.42	1.45	1.50	1.58	1.62	1.63
800	1.53	1.58	1.62	1.65	1.69	1.69
850	1.61	1.64	1.68	1.70	1.73	1.74
900	1.68	1.70	1.74	1.78	1.82	1.83
950	1.74	1.77	1.83	1.89	1.95	1.95
1000	1.81	1.89	1.94	2.01	2.15	2.16

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The density increases with the increase in time and temperature. It has been observed the density which is obtained within 25mins in case of microwave sintering, in conventional sintering it is obtained at around 150 mins time. The result revealed that, higher density is resulted due to microwave sintering which increases the phase changes in fly ash.

The samples processed at 800^0C after microwave sintering was found to be Greyish Yellow and easily stretchable indicating that the phase transformed is in process. The sample treated at 1000^0C changed to brownish grey, which are very hard and acute green strength, which may be attributed to phase transformation that leads to the formation of a hard pellet.

The X-ray diffractometric study of the sample revealed that formation of a glass-ceramic material at higher temperature of 1000^0C sintered up to 25mins in microwave. The data obtained at various temperatures is reported in Fig. (2). It has been observed that the Crystalline phase observed in the starting phase has varied significantly with rise in temperature.

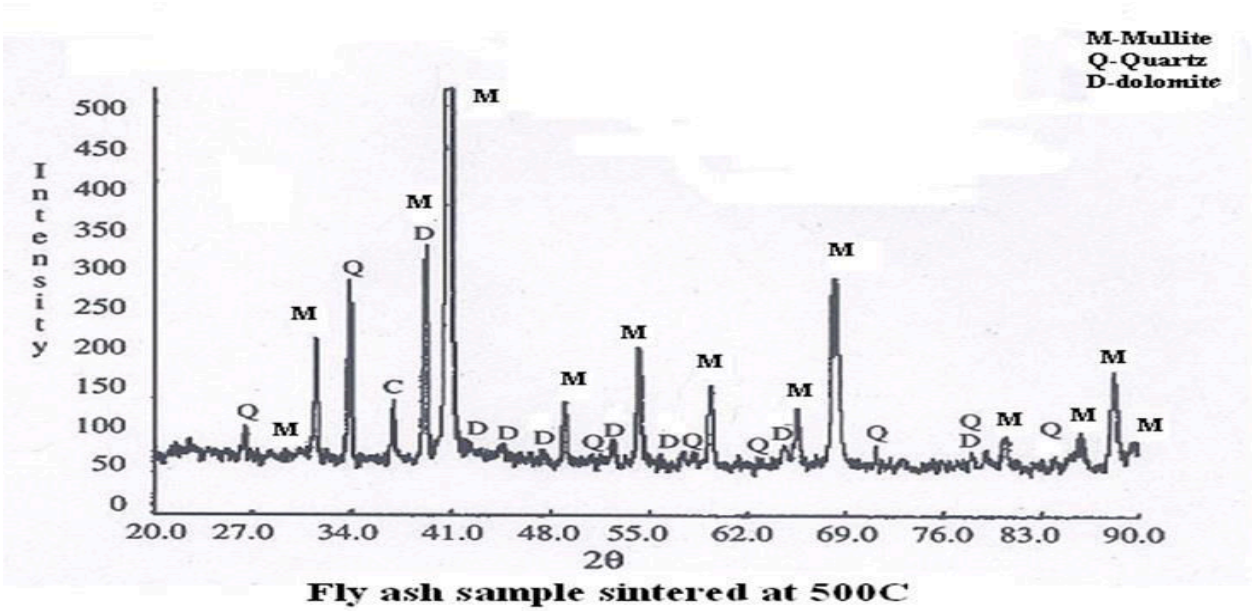

Figure-2 Represents the data obtained at various temperatures

Formation of new peaks at 1000^0C indicates the function of silicate phases in the fly ash pellets. It has been observed, both in case of microwave as well as conventional sintering that the raw fly ash sample provides prominent crystalline phases. When there is increasein temperature gradually the peak height gradually decreases and around 950^0C-1000^0C a new peak appeared prominently. There is formation of mullite phase at lower temperature and Anorthite appeared at high temperature. Thus, we conclude that microwave sintering leads to the formation of glass-ceramic mass.

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V. CONCLUSION

The microwave sintering of fly ash revealed the following conclusions i.e, Microwave sintering of fly ash leads to the function of some more phases at higher temperature, it is quite efficient enough to produce strong material which is highly porous with glass-ceramic structures. The fly ash samples obtained after microwave sintering have high growth, strength and density. Finally it revealed that microwave sintering is efficient over conventional processes.

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