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TECHNICAL NOTE

Preparation of Porous and Dense Bulk Samples in Calcium Magnesium Silicate Systems using Steel Slag and Different Additive by Conventional Sintering Method

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ABSTRACT

In order to obtain the bulk brick-like samples, the mixture of steel slag, and sintering aid additive (like dolomite, glass, perlite borax and phosphate sodium) were sintered at 1100°C for 2 minutes. The flat and bloated surface appearances were obtained. The bulk densities of final sintered composites ranged from 1.3 to 2.41 g/cm^3 and total porosities were from 15 to 40%. The bending strengths varied from 5.5 to 15.9 MPa. The final phase as detected by XRD according was calcium magnesium silicate

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1. INTRODUCTION

Recycling of industrial waste is one of the tasks in the field of protection of the environment that should be solved in the near future [1]. One kind of industrial waste is metallurgical slag from the metallurgical plants in Iran which as the main constituents contains Fe_2O_3, Al_2O_3, SiO_2, CaO, MgO and a small amount of ecologically risky oxides such as V_2O_5.

Up to know several attempts have been made on porous and dense glass-ceramic [2-6], made from steel slag, the preparation process were based on melting, quenching and heat treatment of the obtained frites, with the addition of foaming agent, (like for SiC) but this method is very expensive. In this work, the aim was obtaining a dense and porous compound by the very cheap and available method, corresponding to the mixing of the slag with the foaming additives, pressing and sintering. Up to now the foam structural materials have been produced by using perlite and pumice [3].Also, polyurethane compounds [4] were known as a good foaming agent. However, in this work the attempt is based on the waste management. It is thought that structure of composite built in presented way may prevent the migration of hazardous waste materials into the environment.

2. MATERIALS AND METHODES

The metallurgical slag was supplied by the factory for production of Steel slag and the waste glass was obtained from window glass. Other foaming agent compounds like sodium phosphate, boric acid, and borax were obtained from Merck company, Dolomite and perlite minerals were prepared from Pars mining company. No processing was carried out on as received slag. Figure 1 shows the morphology of grinded and sieved slag powder.

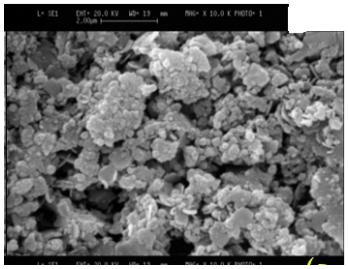

Figure 1. the morphology of sieved slag

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TABLE 1. The chemical composition of as received slag

Oxides	L.O.I.	P_2O_5	MnO	V_2O_5	TiO_2	SO_3	FeO	Fe_2O_3	MgO	SiO_2	Al_2O_3	CaO
Weight%	<6	0.805	0.45	1.115	1.345	0.16	18.44	33.396	6.89	20.85	5.60	29.30

TABLE 3. The Wt% of prepared mixtures

Sample	slag	Dol.	glass	Sic	Sodium silicate	Sodium phos.	borax	Boric acid	Silica	Per
A1	75	10	15	-	-	-	-	-	-	-
A2	60	10	30	-	-	-	-	-	-	-
A3	50	10	40	-	-	-	-	-	-	-
A4	45	10	45	-	-	-	-	-	-	-
B1	52.3	-	42.7	4	-	-	-	-	-	-
B2	48	-	48	4	-	-	-	-	-	-
C2	80	2.5	-	2.5	10	5	-	-	-	-
D1	80	2.5	-	2.5	-	5	10	-	-	-
D2	80	2.5	-	2.5	-	5	-	10	-	-
D3	75	2.5	-	2.5	-	5	15	-	-	-
E3	80	-	-	5	-	5	5	-	5	-
E4	80	-	-	5	-	5	-	5	5	-
F1	80	-	-	5	-	5	5	-	-	5
F2	80	-	-	5	-	5	-	5	-	5

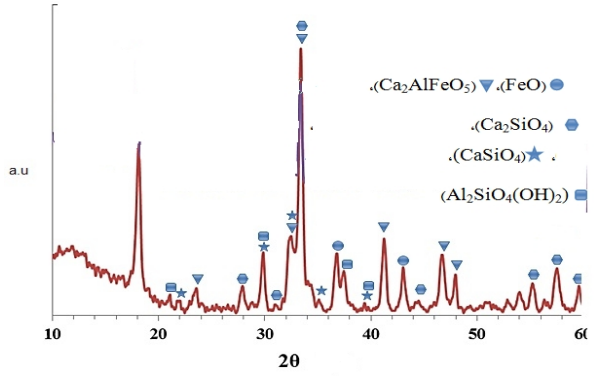

Figure 2. XRD results of as received obtained slag

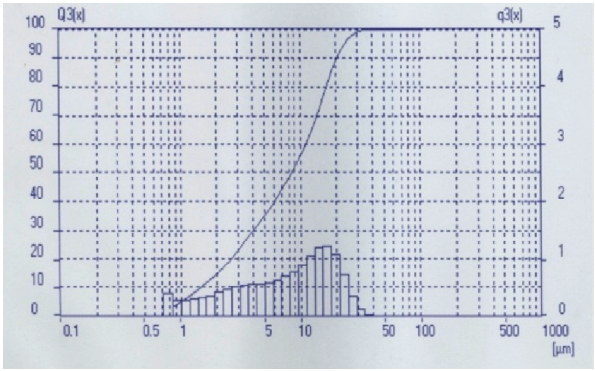

Figure 3. The particle size analysis of slag

TABLE 2. The chemical composition of perlite

Oxides	MgO	NaO	SiO_2
Weight%	0.7	2.7	96.6

Chemical analysis of the materials was carried out using atomic absorption spectrophotometer and wet chemical methods. Table 1 shows the results for perlite and slag. The crystalline phases were identified by powder X-ray diffraction (XRD) patterns using Philips PW 1130. Identification of the crystalline phases was made using Xperts analyser. XRD of the slag showed the presence of $CaSiO_4$, Ca_2AlFeO_5, FeO, Ca_2SiO_4, $Al_2SiO_4(OH)_2$.

The thermal changes of compositions were determined by DTA. The material was ball milled (Fritsch pulverisette 5) for 180 min. Particle size analysis of the milled iron slag and glass are shown in Figure 3

Iron slag and different additives were made by ball milling and the chemical compositions are presented in Table 3. The samples were prepared by a powder technology, uniaxially pressed at 50MPa using 5%PVA as binder, and sintered at 1000-1100°C for 2 minutes using a heating rate of 20°C /min in oxygen atmosphere. The samples were 70×10×10mm which was used for strength measurement. The flexural strength of specimens were determined by three point bending test

on specimens of 70mm×10mm×100mm in size, at a span of 50mm and cross head speed of 0.6mm.min^{-1}. The values for bending strength, was calculated according to the following Equation (1):

$$\delta_f = 3P_F L/2bh^2 \quad (1)$$

where P is the load at fracture (N), L the sample length (mm), b the sample breadth (mm), and h the sample height. The annealing process was performed at 800°C for all samples for 2 hrs, in order to minimize the thermal stresses. Bulk densities of the sintered samples were determined by the Archimedes method ASTM C373 as below:

$$V=W-S,\ P=W-D/V,\ B=D/V \quad (2)$$

W: wet weight of sample, S: immerse weight of sample, P: apparent porosity, D: dry weight of sample
V: volume of sample

The behavior of the samples were compared by their vol% of open porosity with conventional foamed bricks and also in point view of strength..

3. RESULTS AND DISCUSSION

Sintering was carried out at the temperature ranges of 1000-1100°C. Sintering slags with other additives at temperatures above 1100°C exhibits melting. Table 4 summarizes the results for sintered samples. According to Table 4, by using Dolomite as a foaming agent [7], in A1 to A4 systems, no evidence of bloating was found on the surface of the samples (Figure 4), Probably, the glass existening in these samples (as B1 to B2), mixed up with Fe_2O_3, caused an abrupt decrease in viscosity at sintering temperature, which allowed all the carbonates to exit. (Figure 5). Perhaps A1 to A4 samples can be identified as paving stuff. The substitution of SiC for dolomite, in C2 to D3 systems was performed, also sodium (phosphate, silicate), boric acid and borax instead of glass, was added to the system. These additives were added to create the vitreous feature, but apparently very low viscosity of the systems beside the incomplete oxidation of SiC led to limited gas removal, causing the samples to have open pores (almost 0.5mm in size) on the surface (Figure 6).

a **b**

Figure 4. The pressed and sintered (at 1100°C) samples of (a) A1 (b)A2, no evidence of foaming was observed on the surface.

Figure 5. The pressed and sintered (at 1100°C) samples of B1 and B2, the melted appearance was observed

Figure 6. The porous sample with D4 composition

TABLE 4. The appearance of surface, green density, sintered density and porosities of sintered samples

Samples	Sintered temperature	Application	ρ_g (g/cm^3)	ρ_s (g/cm^3)	Por%
A1	1100	Flat surface	1.56	2.01	22
A2	1100	Flat surface	1.87	2.11	28
A3	1000	melting	2.01	2.25	0
A4	1000	melting	2.15	2.42	0
B1	1100	Flat surface and fracture	1.58	1.64	20
B2	1100	Flat surface and fracture	1.60	1.69	22
C2	1100	spalling	1.29	1.9	25
D1	1050	Porous surface	1.60	1.51	23
D2	1050	Porous surface	1.1	1.27	15
D3	1050	Porous surface	1.36	1.46	24
E3	1100	Porous surface	1.2	1.3	25
E4	1100	Porous	1.68	1.9	24
F1	1100	Porous	1.57	1.7	36
F2	1100	Porous	1.53	1.7	40

Figure 7. F2 sample sintered at 1100°C with non-homogeneous porosities

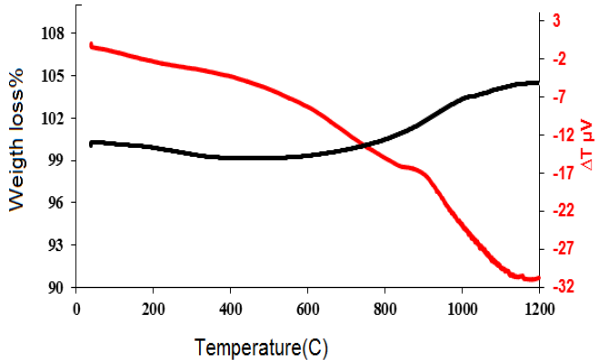

Figure 8. DTG results of obtained F2 sample with 10°C/min heating rate

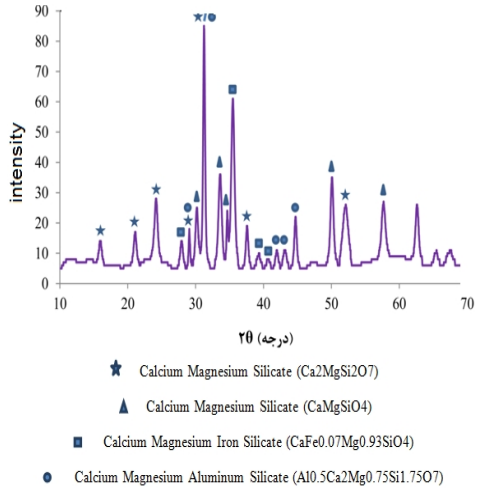

Figure 9. The XRD result of F2 sintered at 1100°C, the detected phases were calcium magnesium silicate

TABLE 5. Bending strength and density of composites.

Samples	F2	F1	E4	E3	A1
Bending Strength (MPa)	6.7	5.5	7.9	8.2	15.9
Weight loss	-0.36	-0.74	-0.47	-0.46	+1.1
density	1.73	1.74	1.94	1.35	2.01

In order to mitigate the fluxing effect of sodium phosphate, boric acid and borax on the system's viscous flow, the silica and perlite were added to the E3 – F2 samples [8]. In this system, large porosities which has been detected on surface of C2 to D3.samples were not observed, but the porosities in E3 to F2 were in the bulk of the samples and non-homogeneous in point view of size. The results are shown in Figure 7.

In order to see the effect of SiC oxidation temperature and weight loss of samples, the DTG was performed and the results are shown in Figure 8.

As can be seen in Figure 8, the weight of F2 sample increased, which can be related to the oxidation of SiC [9]; also a very weak exothermal peak was observed at 900 °C which could be related to the SiC oxidation according to the following equations.

$$SiC+2O_2 \rightarrow SiO_2+CO_2 \quad (3)$$

$$SiC+O_2 \rightarrow SiO+CO \quad (4)$$

here, the enthalpy of the reaction is: ΔH_f =-73.22 kj/mol. Accordingly, the XRD results of F2 sample are shown in Figure 9.

It can be seen in Figure 9 that after sintering the existening free MgO in slag was dissolved in calcium silicate and the calcium magnesium silicate solid solutions were obtained. It should be mentioned that in the case of F2 sample, because of Fe_2O_3, borax and sodium phosphate the melting temperature of the slag decreased and the MgO could be dissolved in calcium silicate structure. The bending strength of porous and dense composites were measured and are reported in Table 5.

In literature [10], it was pointed out that the insulating products which are used as construction stuff have usually compressive strength of at least 0.8-1.0 MPa; however, it is thought that compared to the other foam products, these samples have reasonable strength.

4. CONCLUSION

The aim of the present work was obtaining the dense and porous samples using steel slag containing calcium-alumina-silicate phases. It was shown that existence of slag, Fe_2O_3, and glass in the studied compound, lead to the narrow sintering temperature range, which caused complex gas removal. However, in the case of F2 sample even by non-homogenous porosity the strength of samples was reasonable. In this work the viscosity and abrupt melting was controlled by addition of perlite and silica. The sintering temperature was found at 1100°C for duration of 2 minutes. Also, in this work the dense and porous calcium magnesium silicate samples were prepared which can be used in construction industry.

5. REFERENCES

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چکیده

به منظور دست‌یابی به نمونه‌های آجری‌شکل، مخلوط سرباره فولاد و افزودنی‌های کمک تف‌جوشی (مثل دولومیت، شیشه، پرلیت، بوراکس و فسفات سدیم) تهیه و در دمای ۱۱۰۰ درجه سانتی‌گراد به مدت ۲ دقیقه تف‌جوشی شدند. ظاهر سطح نمونه‌ها صاف و متخلخل به دست آمد. محدوده چگالی توده‌ای کامپوزیت‌های تف‌جوشی شده نهایی در حدود ۱/۳تا ۲/٤۱ گرم بر سانتی‌متر مکعب و تخلخل نهایی در محدوده ۱۵ تا ٤۰٪ بود. استحکام خمشی از ۵/۵ تا ۱۵/۹ مگاپاسکال تغییر کرد. فاز نهایی شناسایی شده کلسیم –منیزیم –سیلیکات بود.

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