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STUDY ON THE BEHAVIOUR OF FLOTATION FAYALITE WASTE FROM COPPER PRODUCTION AT NONISOTHERMAL HEATING

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ABSTRACT

The behaviour of flotation fayalite waste from copper slag at non-isothermal heating has been studied. Only a small part from this product (about 2 %) is used in the cement production. The other part, containing rather high percentage of iron is not used and it is deposited thus polluting the environment.

DTA method was used to study the behavior of this waste at the non-isothermal heating up to 1273K and 1673K. Phase transformations have been defined by chemical and X-ray analyses. The phases of hematite, magnetite and cristalobalite were determined in the end product after flotation. Thermograms, obtained by DTA show that the oxidation processes run into two stages at the non-isothermal heating - the first stage up to T = 894K and the second one up to T= 1155K. Experimentally, the melting temperature of the flotation fayalite waste (T_{melt} ≈ 1457K) was found. The melting of the sample in the crucible was observed at this temperature.

Keywords: fayalite, waste, thermal dissociation, DTA analysis.

INTRODUCTION

Converter and flash furnace slag from copper production contain mainly fayalite because of the character of the technological process. It contains also precious metals which are sometimes in higher concentration than in the real ores. These slags can have different applications even as a waste product. The results of the researches [1, 2] based on the phase composition and structure of converter and flash furnace slag from copper production show that the basic crystal phases are only fayalite and magnetite but fayalite prevails. For systems similar in composition to the waste we have investigated, the thermo-gravimetric study [3] have shown an exothermic peak at 1003K. According to the author this peak is not a result of oxidation process, but a result of recrystallization. Other authors [4] have studied the stability of pure fayalite system thermo-gravimetrically. According to their research the transformations in the investigated samples occur at temperature of 1470K and 1481K in oxidation atmosphere. They are related to the mass increase. Thermal decomposition of pyro -metallurgical copper slag in oxidation atmosphere in synthetic air was investigated at temperature range of 773K - 973K [5].

The aim of present study is to investigate the behavior and the phase transformation as a result of heating of the flotation fayalite waste from copper slag.

EXPERIMENTAL

Output materials

The chemical composition of the output flotation fayalite waste is defined by weight and ICP-OES analysis.

The sulphur of this flotation waste is 1.26 % and the dampness is 1.34 %. X-ray analysis of the flotation waste output is presented in Fig. 1.

Table 1. Chemical composition of output flotation waste.

Element	Cu	Fe	Pb	Zn	Ti	Ni	Si	Ca	Mg	Ba	Al
Mass %	1.20	45.90	0.20	0.69	0.09	< 0.01	10.08	0.72	0.38	0.08	2.5

Table 2. Chemical composition of waste flotation after heating to 1273K.

Element	Cu	Fe	Pb	Zn	Ti	Ni	Si	Ca	Mg	Ba	Al
Mass%	0.72	45.15	0.72	0.69	-	0.07	11.69	0.6	0.51	-	-

The X-ray analysis of the flotation waste output shows: Fayalite - Mg.347Fe1.548Mn.105SiO_4; silica - SiO_2; magnetite-Fe_3O_4; mica-(Na0.148K1.973Ca0.007Ba0.053)(Al0.034Fe1.978Mg3.222Mn0.019Ti0.7).

RESULTS AND DISCUSSION

The flotation fayalite wastes were heated non-isothermally to 1273K (1000°C) and 1673K (1400°C). The thermogram of sample 1 is presented in Fig. 2 while X-ray analysis is shown in Fig. 3.

X - Ray analysis of sample 1 shows that after heating to 1273K the product contains mainly hematite, magnetite and fayalite (Mg0.075 Fe 1.741Mn0.123SiO_4).

The chemical composition of sample 1 after heating is determined by atomic adsorption method and presented in Table 2. The weight analysis shows that the sulphur is 0.272 %.

Sample 2 of the same flotation waste is heated to 1673K. The thermogram obtained by non-isothermal

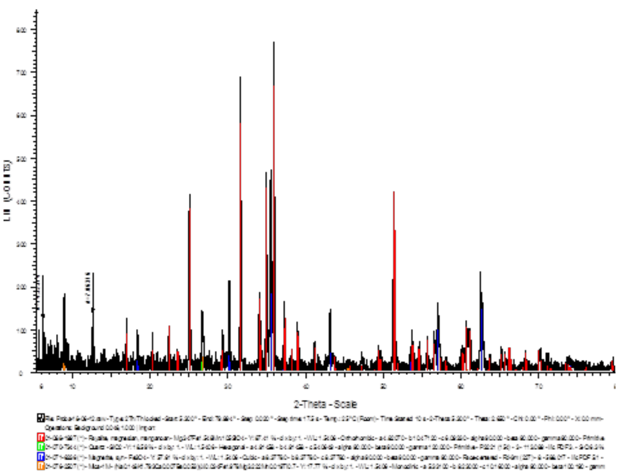

Fig. 1. X-ray analysis of flotation waste output.

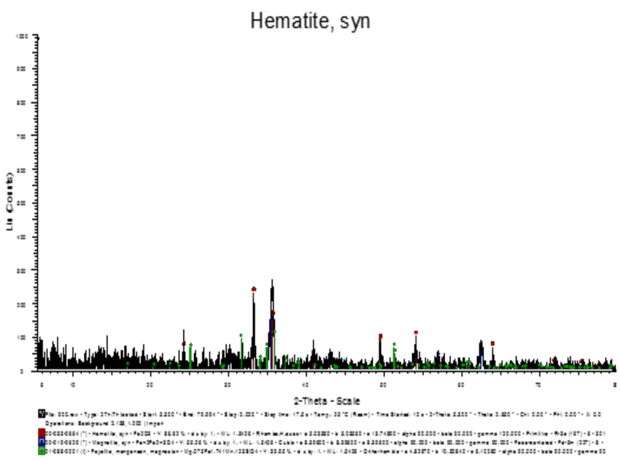

Fig. 3. X-ray analysis for sample 1 heated non-isothermally to 1273K.

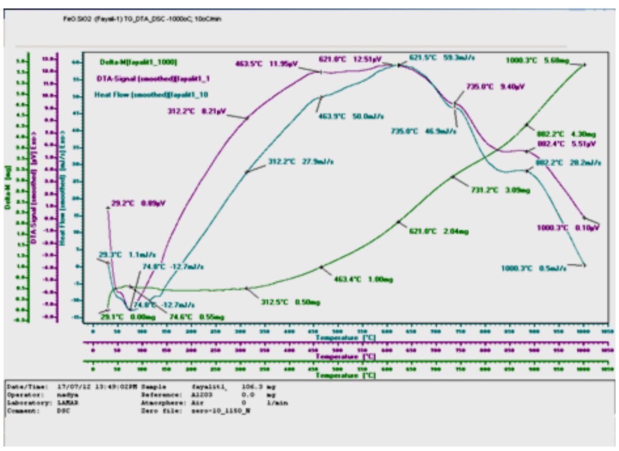

Fig. 2. Thermogram of sample 1 heated non-isothermally to 1273K.

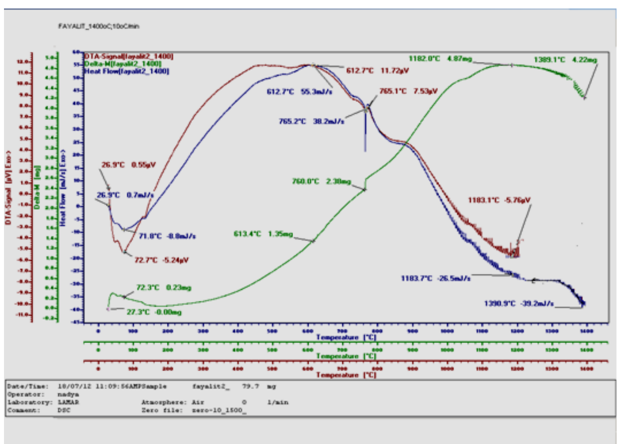

Fig. 4. Thermogram of sample 2 heated non-isothermally to 1673K.

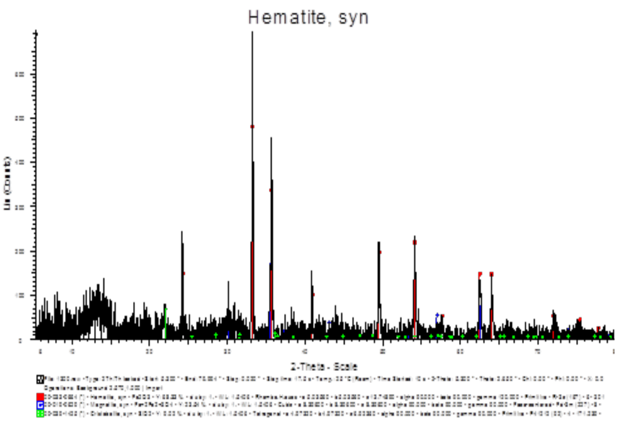

Fig. 5. X-rays analysis for sample 2 heated non-isothermally to 1673K.

heating is presented in Figure 4 and the respective X – ray analysis – in Fig. 5.

X – ray analyses of sample 2 shows that the product contains mainly hematite, magnetite and cristobalite.

Two effects are observed on DTA and DSC curves for sample 1– one endothermic effect (Q= -12700 J/s) at T ~ 348K and one exothermal effect at T ~ 894K (Q =59300 J/s). The endothermic effect at that low temperature is associated not only with the release of moisture but also with a process associated with absorption of heat and increase in the mass of the sample (0.55 mg or 0.52%).

The exothermal effect at temperature of 894K is a result of partial oxidation of magnetite to hematite by the reaction:

$$4Fe_3O_4 + O_2 \Leftrightarrow 6Fe_2O_3 + Q\ (464800 J/mol) \qquad (1)$$

On DTA and DSC curves another two exothermal effects at T~1008K (Q= 46900J/s) and T~1155K (28200J/s) are observed. X – ray analysis of the final product of non-isothermal heating shows mainly magnetite, hematite and cristalobalite.

Studying the thermodynamics of the two reactions, it has been concluded [3], that reaction (2) does not proceed satisfactorily in the temperature range of 298K to 1500K in contrast to reaction (3).

$$2Fe_3O_4 + 3SiO_2 = 3Fe_2SiO_4 + O_2 \qquad (2)$$

$$2FeO + SiO_2 = Fe_2SiO_4 \qquad (3)$$

Two effects are observed on DTA and DSC curves for sample 2 - one endothermic effect (Q = -8800 J/s) at T ~345K and one exothermal effect at T ~ 886K (Q = 55300 J/s).

The endothermic effect at the lower temperature is associated not only with the release of moisture but also with a process, associated with absorption of heat and increase in the mass of the sample (0.23 mg or 0.29 %).

The maximum exothermal peak is a result of oxidation process and the mass of the sample increases with about 1.35 mg or 1.69 %. This corresponds to the first stage of the oxidation process. On the DTA and DSC curves another two exothermal effects have been observed at T~1038K (Q = 38200 J/s) with increase in the mass by 2.38 mg or 2.99 % and at up to T~1155K (20200 J/s) with increase in the mass 3.20 mg or 4.015 %. The oxidation process is observed at 1455K in accordance with maximum mass increase of 4.87 mg or 6.11 %. This corresponds to the second stage of the oxidation process and it is due to the partial oxidation of magnetite to hematite (reaction 1). At 1457K (Q = - 26500 J/s) and 1663K (Q = - 39200 J/s) two endothermic effects are observed which are related to the melting of fayalite and FeO ($T_{melting}$ = 1650K) [4]. The mass of the sample starts decreasing. X-ray analysis shows magnetite, hematite and cristalobalite. Hematite and magnetite can be reduced by carbothermy method. A definite amount of CaO must be added which is necessary for the connection of the cristalobalite phase or to use as reductive agent CaC_2.

According to authors [4] the oxidation processes occur in two stages - the first occurs with 0.3 % increase of the mass and the highest rate is at T = 602K, and the second with 3.6 % increase of the mass at temperature T = 877K. At these temperatures exothermic effects have also been observed. The temperatures of exothermic effects, obtained in the present work are closed to these one obtained by authors [4]. A third stage has been marked, associated with mass reduction. According to them, this is due to the reverse process.

At 1422K an endothermic peak is observed, which is associated with the melting of the fayalite. The slight discrepancy in the results is due to the different raw materials used in conducting the experiments. The authors found that $2FeO.SiO_2$ melts congruently at 1478K.

CONCLUSIONS

As a result is study the following conclusions can be drawn:

The behaviour and the phase transformations of flotation fayalite waste at non-isothermic heating up to 1273K and 1673K, using DTA analysis have been investigated.

The X-ray analysis of the heated fayalite waste from the flotation shows three phases – hematite, magnetite and cristalobalite. The carbothermal reduction is the most suitable for hematite and magnetite phases but a definite amount of CaO must be added which is necessary for the connection of the cristalobalite phase.

It is found out that the oxidation processes in flotation fayalite waste samples run into two stages at the non-isothermal heating - the first stage up to T = 894K and the second one up to T= 1155K.

Experimentally, the melting temperature of flotation fayalte waste T= 1457K was found. It is similar to the temperature found out by other authors (T=1422K). Solid-phase reduction of flotation fayalite waste pellets must be carried out close to this temperature.

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