Environmentally Friendly Pyrometallurgical Scheme for Vacuum Separation of Non-Ferrous Metals from Waste Products in Incineration Plants

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Abstract

Residues from the municipal solid waste processed in incineration plants in European countries are an important raw material to obtain valuable components, including non-ferrous metals. State and private companies specializing in the processing of waste incineration slag as products most often receive concentrates of non-ferrous metals, which, on average, contain, in mass. %: 20÷60 Cu; 10÷30 Zn; 5÷15 Pb; ~ 1 Al; ~ 1 Sn; ~ 1 Fe, up to 50 g/t Au and up to 3,000 g/t Ag. Concentrates are sent for processing to smelters without taking the cost of zinc into account. The paper presents the study on the separation of metallic zinc into a separate product (zinc concentrate) from the collective concentrate of non-ferrous metals by a vacuum-thermal method, the safest from the environmental point of view. The study was performed with non-ferrous metal concentrate of +0.3-0.8 mm in size, containing wt. %: 68.07 - Cu; 12.4 - Zn; 14.78 - Pb; 0.99 - Al; 1.2 - Sn; 0.15 - Fe, up to 2.0 kg/t - Ag. The material was heat treated at 800÷900°C with the residual pressure in the system of less than 0.13 kPa. Zinc concentrate was obtained, containing more than 96% of the main component. At the same time, the Cu content increased by 14.09% in the residue from the heat-vacuum treatment. Other metals (Pb, Al, Sn) including noble metals were also concentrated in the residue. The results of the study show that it is possible to separate zinc into a separate product from non-ferrous metal concentrates containing more than 10% Zn in the initial material by the proposed method.

1. Introduction

Every year, the amount of municipal solid waste (MSW) increases, in particular, developed countries and megacities around the world are faced with this problem [1-4]. Disposal of the ever-increasing amount of MSW is not only an environmental problem but also a social and economic one [5-7]. Disposal of MSW practiced over the past decades is widely recognized as a futureless direction, both from the environmental and economic sides [8-10]. One of the solutions for this problem is the construction of waste incineration plants which, along with waste disposal, receive energy (in the form of steam or hot water) [11,12] and materials that serve as a secondary source of valuable components [13,14].

Waste disposal technology at thermal waste treatment plants is accompanied by the production of slags containing up to 10% of metal scrap that is a secondary raw material for the extraction of non-ferrous metals, including ferrous metals - about 7-8%, non-ferrous metals - remained 2-3% [15,16].

The metal content in slag and ash from MSW combustion averages 25 kg per ton of MSW [17]. The resulting slag is sent for mechanical processing (grinding) and subsequent sorting, based on the different physical properties of the slag constituents. The material is washed, the iron is removed by magnetic separation, the "light" (aluminum) and "heavy" components of the slag (copper, brass, lead, zinc, silver, gold, etc.) are separated [18,19]. The obtained concentrate of "heavy" non-ferrous metals contains up to 37% Cu, up to 13% Zn (as brass), up to 4% Pb and Sn (as bronze), meets the requirements for secondary copper raw materials and is sent to copper-smelting plants for copper extraction [20,21], as a rule, by the pyrometallurgical method. Similar technologies [14,22] are used in the largest companies for the disposal and processing of waste incineration plant slags in Europe and Switzerland [23].

Many scientific studies are devoted to the problem of MSW processing, in particular in incineration plants, with subsequent extraction of valuable components from the slag and ash. There are innovative technologies that enable sufficiently complete extraction of valuable components from the slag and ash from MSW combustion into non-ferrous metal concentrate [14,24-27]. The objectives of the study included the development of an environmentally safe, reagent-free, pyrometallurgical scheme for the vacuum separation of non-ferrous metals from waste products of incineration plants. Previously, similar studies were performed at the Institute of Metallurgy and Ore Beneficiation to obtain high-purity selenium from copper-smelting waste [28,29]. The authors studied various two-component systems, including the thermodynamics of formation and evaporation of lead-tin alloys to determine the optimum conditions for vacuum-thermal zinc extraction [30].

2. Experimental Work

2.1. Materials and reagents

The collective concentrate of non-ferrous metals is obtained on an innovative technological line for the processing of slags formed as a result of thermal utilization of residual solid domestic waste at incineration plants. The basis of the process is the dry mechanical process that allows the processing of about 120,000 tons of slag annually. The output products (non-ferrous metal concentrate) consist of a mixture of non-ferrous and noble metals. The concentrate is non-magnetic and has electrical conductivity. Table 1 shows the material composition of the collective concentrate after beneficiation of metals on the technological line for dry mechanical processing of slags.

Table 1 - Material composition (particle size and elemental) of the collective concentrate after beneficiation of metals in the dry processing technological line.

class, mm	Output		Content, g/t		Content, %				
	in parts	%	Au	Ag	Al	alloy steel	Cu	brass	Zn
-8.0+5.0	1.6	24.24	50	2,800	1.0	1.0	20.0	50.0	28.0
-5.0+3.0	2.2	33.33	80	3,500	1.0	0.0	20.0	50.0	29.0
-3.0+0.8	2.4	36.36	100	3,500	1.0	0.0	30.0	45.0	25.0
-0.8+0.3	0.4	6.07	80	2,500	1.0	0.0	40.0	40.0	20.0
Total:	6.6	100.0							

We can see from the data in Table 1 that zinc in sufficient quantities is contained in the collective concentrate of non-ferrous metals, and its cost is not taken into account when the concentrate is supplied to the copper processing plant. The idea of the project was to develop an environmentally safe technological scheme for vacuum distillation separation of non-ferrous metals present in the collective concentrate into separate products, in particular: zinc separation into zinc concentrate.

Non-ferrous metal concentrate with a particle size of +0.3-0.8 mm was used as a test material shown in Fig. 1.

2.2. Experimental methods

The content of major elements in the concentrate and the products obtained after heat treatment of the material was determined by chemical method with the help of an atomic emission spectrometer Optima 8300 DV “Perkin Elmer” (made in the USA) and by X-ray fluorescent method with a wave-dispersive combined spectrometer Axios “PANalytical” (made in the Netherlands). The phase composition of the initial materials was determined in a D8 Advance “BRUKER” X-ray diffractometer (made in Germany) in Cu-K α radiation and a JEOL JXA-8230 scanning electron microscope (made in Japan).

Separation of zinc into a separate product was performed with the help of a laboratory vacuum apparatus with a vertical arrangement of a quartz reactor. The scheme of the laboratory setup is shown in Figure 2. The unit consists of an electric tube furnace Nabertherm RT 50-250/11 (1) with a quartz reactor installed inside (2). A set of five graphite crucibles (4) was installed inside the quartz reactor simultaneously serving as condensers of the formed vapor phase in the colder zone. The graphite crucibles were made of a graphite electrode, the height of the crucibles was 600.0 mm, the outer diameter was 42.0 mm, and the wall thickness was 3.0 mm. The lower (loading) crucible had a solid bottom, the subsequent crucibles had a through-hole $\varnothing$ 18.0 mm at the bottom to vent the vapor phase. A special selection on the bottom and top of the crucibles enabled us to assemble a vertical structure from a set of crucibles. The holes at the bottom of the crucible are provided with nipples that protrude 20 mm above the crucible bottom to prevent the resulting condensate from flowing from the upper crucible to the lower crucible to the hotter zone in the case of vapor-liquid condensation. The temperature in the system was additionally controlled using a chromel-alumel thermocouple installed above the sample of the test material. The vacuum in the system was created using a Pfeiffer Vacuum HiPace® 10 series turbomolecular vacuum pump (made in Germany) with a pumping rate of 10 l / s. The residual pressure in the system was monitored using a Pirani VSM77D vacuum gauge (made in Germany). The degree of distillation of the volatile components of the concentrate was determined by the weight loss of the sample using an analytical balance Shimadzu AUW-220 (made in Japan).

3. Results And Discussion

3.1. Characterization of collective non-ferrous metal concentrates

The content of the main components in the concentrate determined by chemical analysis with the atomic emission spectrometer Optima 8300 DV “Perkin Elmer”, is shown in Table 2.

Table 2 Results of chemical analysis of non-ferrous metal concentrate							
Material size	Content, wt.%						
	Zn	Pb	Cu	Fe	Al	Sn	other
+0.3–0.8 mm	12.97	8.592	66.21	0.302	0.648	0.688	10.59

The source material was fused in a graphite crucible in an induction vacuum electric furnace to determine the elemental composition by X-ray fluorescence analysis to average the composition, and a tablet was formed from the material using an industrial press. The results obtained on a wave-dispersive combined spectrometer Axios “PANalytical” are shown in Table 3.

Table 3 - Results of X-ray fluorescence analysis of non-ferrous metal concentrate, particle size + 0.3-0.8 mm

Element	Content, wt. %		
	Concentrate ingot	Compressed Tablet	Average value
Cu	75.269	62.131	68.700
Zn	10,513	14,287	12,400
Pb	12,423	17,137	14,780
Fe	0,148	0,15	0,149
Al	0,055	1,919	0,987
Sn	0,842	1,565	1,204
Ag	0,169	0,175	0,172
Si	0,051	0,274	0,163
Ni	0,286	0,428	0,357
P	0,012	0,045	0,029
S	0,051	0,131	0,091
Cl	0,032	0,276	0,154
Sb	0,147	0,178	0,163
Mg	0	0,116	0,058
K	0	0,03	0,015
Ca	0	0,649	0,325
Ti	0	0,305	0,153
Mn	0	0,036	0,018
Y	0	0,069	0,035
Zr	0	0,033	0,017
Mo	0	0,066	0,033
Co	0,002	0	0,001

The alkali, alkaline-earth, and rare-earth metals (Mg, Ca, K, Y) found in trace amounts by the X-ray fluorescence method in the concentrate sample in the pressed tablet (in the original material without heat treatment, not present in the remelted ingot) were most likely slagged as a result of melting the material in the induction furnace at high temperatures.

The quantitative content of elements in the studied material by X-ray fluorescent method was determined under standard procedures using the obtained spectra without the use of standards for calibration. Obtained overestimated values of the sum of certain elements "before normalization" (above 100%) indicate the high intensity of the spectra, which indirectly indicates the reliability of the results obtained.

The initial material of the sample was sent for mineralogical analysis and scanning electron microscopy to determine the phase composition both inside the metal grains and at their boundaries. Figure 3 shows the images of the thin sections of the sample concentrate, obtained on a scanning electron microscope JEOL JXA-8230.

3.2. Separation of zinc into a separate product

The separation of zinc into a separate product from a concentrate of non-ferrous metals with a particle size of + 0.3–0.8 mm was performed in the temperature range: 800 °C and 900 °C with residual pressure in the system <0.13 kPa (<1 mm Hg). The experimental results are shown in Table 4.

Table 4

Results of experiments on the distillation of volatile components (Zn) from a sample of a collective concentrate of non-ferrous metals with a particle size of + 0.3–0.8 mm

τ , min	t , °C	m of the weighted sample, g	Δm_1 , g	m of condensate, g					Σm of sublimation, g	m of nonviscous, g	Degree of volatilization, %
				Δm_2	Δm_3	Δm_4	Δm_5	Δm of the retorts			
90	800	135.5	-14.2	0	0.1	0.2	11.2	2.5	14	-0.2	10.48
90	900	138.2	-16.8	0.1	0.5	0.9	1.4	13.6	16.5	-0.3	12.16

The chosen temperature interval is justified by the elimination of significant material overheating and, consequently, lower energy consumption during zinc distillation, as well as by the reduction of lead transfer into the vapor phase and its further co-condensation with zinc. Residues from zinc distillation from the

collective concentrate sample were formed into "tablets" using an industrial press and sent for X-ray fluorescence analysis. Figure 4 shows photographs of the residues after thermal vacuum treatment of the concentrate and the resulting zinc condensate at the bottom of a graphite crucible.

The bulk of the zinc at 900°C was condensed on the walls of the quartz retort in the colder zone from the location of the graphite crucibles (Table 4). X-ray fluorescence analysis showed that in the zinc obtained as a result of liquid-phase condensation (collected on the walls of the retort), the content of the main component is 98.21%, the main impurity elements, wt%: 0.15 - Pb; 0.15 - Mg; 0.13 - Al; 0.35 - Si; 0.2 - Cl. The content of other impurity elements is less than 0.02%.

The resulting sublimes of zinc condensate were remelted into an ingot (to average the composition) to determine the content of the main component (Zn) and impurity elements. Melting was performed in an induction vacuum furnace at atmospheric pressure of 480-520°C. The foam (ZnO) formed on the surface of the ingot was removed mechanically with a tantalum stick.

The resulting ingot is machined to give a smooth surface. A photograph of the obtained ingot of metallic zinc is shown in Figure 5. Table 5 shows the content of elements determined by X-ray fluorescence analysis.

Table 5
The content of elements in a metal zinc ingot obtained from sublimes during vacuum heat treatment of a sample of a collective concentrate of non-ferrous metals at t = 800 °C and t = 900°C

Element	Zn	Pb	Al	Si	Ni	S	Cd	F	Cl	Mg	Σof elements
wt. %	96.251	1.552	0.05	0.544	0.022	0.013	0.131	0.925	0.36	0.152	100.0

Table 6 shows the content of elements determined by the X-ray fluorescence analysis method in the residues from the thermal vacuum treatment of the collective concentrate of non-ferrous metals at temperatures of 800 °C and 900°C.

Table 6
The content of elements in the remains of the sample of concentrate No. 1 after the distillation of volatile components from them at t = 800 °C and t = 900°C

Content, wt. %											
Element	Cu	Zn	Pb	Al	Sn	Si	Ag	Mg	P	Fe	S
t=800°C	73.254	1.129	17.487	2.221	0.632	1.749	0.174	0.406	0.045	0.429	0.347
t=900°C	78.195	0.618	13.024	2.103	0.938	1.851	0.231	0.319	0.054	0.461	0.252
Element	Cl	K	Ca	Ni	Cr	Mn	Ti	Bi	Y	Mo	Sb
t=800°C	0.174	0.039	1.001	0.282	0.041	0.018	0.038	0.08	0.301	0.03	0.123
t=900°C	0.121	absent	1.251	0.216	0.082	0.042	0.074	0.047	0.121	absent	absent
* The sum of elements before normalization was: at t = 800 °C - 133.1%; at t = 900°C - 126,9%											

Table 7 shows the results of the X-ray phase analysis of the formed foam. Analysis performed on a D8 Advance (BRUKER) diffractometer, Cu radiation – K_α.

Results of X-ray phase analysis of the obtained foam			
Pattern #	Compound Name	Formula	S-Q
PDF 00-004-0831	Zinc, syn	Zn	62,4%
PDF 01-070-8070	Zincite, syn	ZnO	20,5%
PDF 00-004-0686	Lead, syn	Pb	14,2%
PDF 00-001-0649	Quartz	SiO2	3,0%

3.3. Material balance of zinc distribution by processed products

Material balance of zinc and main accompanying elements distribution under the products of vacuum-thermal treatment of collective non-ferrous metal concentrate is presented in Table 8.

Table 8

The material balance of zinc and main accompanying elements distribution among the products of vacuum heat treatment of material at zinc separation into from non-ferrous metal concentrate sample No.1

Name of products	Weight,	Output,	Content of		Distribution, %	Content of		Distribution, %	Content of		Distribution, %	Content of	
	Zn	Pb	Cu	Other elements									
	g	%	%	g		%	g		%	g		%	g
Loaded:													
Sample concentrate (+ 0.3-0.8 mm)	823.8	100	12.4*	102.15	100.00	14.78	121.76	100.00	68.07*	560.76	100.00	4.750	39
Received:													
Sublimates (obtained ingot)	99.4	12.07	96.251	95.67	93.66	1.552	1.54	1.27	0.00	0.00	0.00	2.197	2.
Residue from heat treatment	720.2	87.42	0.878	6.32	6.19	16.122	116.11	95.36	77.665	559.34	99.75	5.058	36
Total received:	819.6	99.49	-	102.00	99.85	-	117.65	96.63	-	559.34	99.75	-	38
Difference:	-4.2	-0.51	-	-0.15	-0.15	-	-4.10	-3.37	-	-1.42	-0.25	-	-0

4. Conclusions

It was found as a result of the study that the collective non-ferrous metal concentrate produced under the technology of dry mechanical processing of slag from incineration plants, sized +0.3-0.8 mm contains, wt %: 68.07 - Cu; 12.4 - Zn; 14.78 - Pb; 0.99 - Al; 1.2 - Sn; 0.15 - Fe, up to 2.0 kg/t - Ag. It is shown that in principle, it is possible to extract more than 93% of zinc from a sample of non-ferrous metals concentrate using an environmentally friendly reagent-free vacuum-thermal method into a separate product (zinc concentrate) containing more than 96% Zn. Valuable components (Cu, Pb, Ag) are concentrated in the evacuation residues and are suitable for further processing by classical methods. The environmental friendliness of vacuum-thermal technologies is determined by a significant decrease in the number of waste process gases, the sanitary cleaning of which is not difficult, by a decrease in the temperature of heat treatment resulting in the reduction of energy costs.

Declarations

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Figures

Figure 1

Photos of non-ferrous metal concentrate with +0.3-0.8 mm particle size

Figure 2

Schematic diagram of the laboratory vacuum unit 1 - tubular electric furnace; 2 - quartz reactor; 3 - graphite crucible (base); 4 - a set of crucibles with a channel for steam passage; 5 - a sample of the starting material; 6 - alundum support; 7 - rubber stopper.

Figure 3

The results of the study of the concentrate sample with the grain size of +0.3-0.8 mm on scanning electron microscope JEOL JXA-8230 (magnification x70)

Figure 4

Photographs of products obtained after stripping zinc from a sample of a collective concentrate of non-ferrous metals a) loading crucible with concentrate after zinc stripping at $t = 800^{\circ}\text{C}$; b) upper crucible with metallic zinc condensate; c) compressed concentrate in the form of a tablet for analysis after zinc stripping at $t = 900^{\circ}\text{C}$.

Figure 5

Photo of the obtained ingot of metallic zinc