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Ispitivanje mogućnosti poboljšanja kvaliteta Pb-Zn šljake iz "Topionica" -Veles primenom magnetne separacije

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Abstract

Smelting slag from "Topionica" -Veles (Northern Macedonia) owned by the company "KEPS MONT GROUP" Skopje is a potentially valuable raw material. Tests have shown that this is an inhomogeneous raw material with significant contents of non-ferrous metals, primarily Pb, Zn, Cu, and silver is present from precious metals. Mineralogical tests have shown that the composition of man-made raw materials is very complex and consists of amorphous and mineral phases, ie the microscopic method showed the following composition: amorphous phase, lead alloys, zinc alloys, vistite (FeO), sphalerite, galena, cerusite, elemental silver, elemental copper, elemental iron, magnetite, spinel, rutile, hematite, troilite (FeS). The most common amorphous phase (glass matrix) of spinel, silicate and mixed (spinel-silicate) composition is in the sample, while vistite, which occurs in the form of skeletal separations in the glass matrix, is significantly less represented. Based on SEM analysis (scanning electron microscopy), these are Fe-Mn-Zn spinels. Lead and zinc alloys are the most common. Mineralogical analysis by size classes determined that useful and unuseful components of Pb-Zn slag "Veles" were released at a fineness of -0.1 + 0.00 mm, so that magnetic separation tests were performed on this size class. Magnetic separation was performed on two devices, a Davis wet analyzer and a dry disk magnetic separator. By magnetic separation on a Davis magnetic separator, a small degree of iron concentration in the MF (1,134) relative to the input was obtained, with low iron distribution in the magnetic fraction of 16.79%. Magnetic separation on a dry disk magnetic separator also yielded a low degree of iron concentration in the magnetic fraction with distribution of 55.38% and 44.62% in the non-magnetic fraction.

Keywords: *lead, zinc, slag, magnetic separation.*

Izvod

Šljaka iz "Topionice" -Veles (Severna Makedonija) u vlasništvu firme "KEPS MONT GROUP" Skoplje je potencijalno vrijedna sirovina. Ispitivanja su pokazala da se radi o nehomogenoj sirovini sa značajnim sadržajem obojenih metala, prvenstveno Pb, Zn, Cu, a od plemenitih metala prisutno je srebro. Mineraloška ispitivanja su pokazala da je sastav tehnogene sirovina vrlo složen i sastoji se od amorfne i mineralne faze, odnosno mikroskopska metoda pokazala je sljedeći sastav: amorfna faza, legure olova, legure cinka, vistit (FeO), sfalerit, galenit, cerusit, elementarno srebro, elementarni bakar, elementarno gvožđe, magnetit, spinel, rutil, hematit, troilit (FeS). U uzorku je najzastupljenija amorfna faza (staklasti matriks) spinelnog, silikatnog i mešanog (spinel-silikatnog) sastava, dok je znatno manje zastupljen vistit koji se javlja u obliku skeletnih izdvajanja u staklastom matriksu. Na osnovu SEM analize (skenirajuća elektronska mikroskopija) radi se o Fe-Mn-Zn spinelima. Najčešće su legure olova i cinka. Mineraloškom analizom po klasama krupnoće utvrđeno je da su korisne i nekorisne komponente Pb-Zn šljake "Veles" oslobođene pri finoći -0,1+0,00 mm, pa su na ovoj klasi krupnoće izvršena ispitivanja magnetske separacije. Magnetska separacija izvršena je na dva uređaja, Davisovom mokrom magnetskom analizatoru i suvom magnetskom separatoru sa diskom. Magnetskom separacijom na Davisovom magnetskom separatoru dobijen je

mali stepen koncentracije gvožđa u MF (1,134) u odnosu na ulaz, sa niskom raspodelom gvožđa u magnetičnoj frakciji od 16,79%. Magnetskom separacijom na suvom magnetskom separatoru sa diskom takođe je dalo nizak stepen koncentracije gvožđa u magnetičnoj frakciji sa raspodelom od 55,38% i u nemagnetskoj frakciji 44,62%.
Ključne reči: *olovo, cink, topionička šljaka, magnetska separacija.*

Introduction

An industry with a minimum amount of waste is a challenging, but also a vital strategy for the 21st century. This philosophy profoundly reshapes our thinking about resources and production, where waste is no longer waste but a secondary resource and is a key part of a sustainable life cycle. This encourages industry, government and society as a whole to redesign our practices to avoid waste disposal and to take advantage of useful waste components.
Slag from "Topionica" -Veles (Northern Macedonia) owned by the company "KEPS MONT GROUP" Skopje, according to the previously performed physical-chemical and mineral characterization is a potentially valuable raw material. Mineralogical and chemical analysis showed that it is an inhomogeneous raw material with significant contents of non-ferrous metals, primarily Pb, Zn, Cu, and of precious metals, Ag-silver is present [1]. Also, analyzes have shown that despite the presence of very useful elements and minerals, slag from "Topionica" -Veles has a very variable content of useful metals, ranging from a wide range of several% of non-ferrous metals to over 10%, also silver content is very variable from a few g/t to a few tens of grams per ton of man-made raw material. As in the sample, according to previous mineralogical tests [2], some components of the amorphous phase in which the iron in the melting process passed, spinels and elemental iron observed in the sample are present from the magnetic components, it was decided to perform the magnetic separation process on two Davis devices. wet analyzer and dry disc magnetic separator [3, 4, 5].

Sample preparation and sampling for characterization and technological investigation

The sample of "Veles" Pb-Zn slag after physical-chemical and mineralogical characterization was crushed to the size required for technological tests in laboratory operating. The whole sample was crushed to a size of 100% -2 mm, a size suitable for a laboratory mill with balls "Denver", in which the conditions for the release of useful components from slag were determined. Then, the initial ore sample was homogenized and samples of mass m = 1 kg were taken from it, on which grinding experiments were performed [7, 8]. The scheme of sample preparation for technological processing is given in Figure 1 [1].

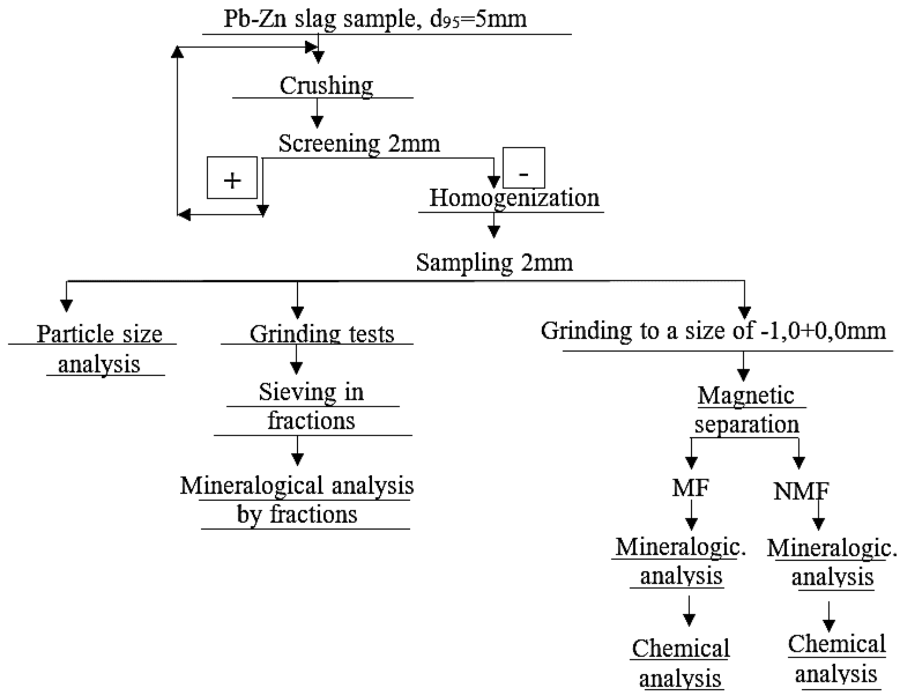

Figure 1. *Written scheme of "Topionica" -Veles Pb-Zn slag preparation for technological testings*

Characterization of the representative sample

Physico-chemical and mineralogical tests of Pb-Zn slag "Smelter" -Veles were performed in order to obtain data that would be the basis for further technological tests to define the procedure for processing and cleaning of Pb-Zn slag "Smelter" - Veles, and obtaining commercial products, primarily the separation of non-ferrous metals and silver.

Particle size distibution

The granulometric composition of the initial sample of Pb-Zn slag was performed for the needs of technological tests after crushing to 2 mm ggk. The granulometric composition of the Pb-Zn slag sample is shown in Table 1.

Table 1. Particle size distribution of Pb-Zn slag from „Topionica“ Veles

Grain size, mm	M, %	↓∑ M, %	↑∑ M, %
-2.00 + 1.60	0.54	0.54	100.00
-1.60 + 1.19	8.20	8.74	99.46
-1.19 + 0.80	24.86	33.60	91.26
-0.80 + 0.63	19.20	52.80	66.40
-0.63 + 0.40	25.03	77.83	47.20
-0.40 + 0.30	11.04	88.87	22.17
-0.30 + 0.20	6.53	95.40	11.13
-0.20 + 0.15	2.14	97.54	4.60
-0.15 + 0.10	0.85	98.39	2.46
-0.10 + 0.00	1.61	100.00	1.61
Ulaz	100.00		

The mean grain diameter of the Pb-Zn slag sample was found to be d_{50}=0.6504 mm, while the d_{95} was 1.314 mm.

Chemical characterization of the raw sample

Table 2 shows the components obtained by silicate analysis and Table 3 shows the useful metal contents in the sample as well as the sulfur content.

Table 2. Basic components content of the Pb-Zn slag from „Topionica“ Veles initial sample (silicate analysis)

Comp.	SiO_2	Al_2O_3	CaO	MgO	Na_2O	K_2O	Fe_2O_3	TiO_2	G.Ž.
Cont.,%	17.43	7.43	12.39	2.135	0.391	0.565	47.68	0.503	poveć. mase. 5.98

Table 3. Useful metals and sulfur content in the Pb-Zn slag from „Topionica“ Veles initial sample

Component	Pb, %	Zn, %	S, %	Ag, ppm
Content	2,24	7,10	2,10	27,53

Mineralogical characterization of the raw sample

The most common phase is the amorphous phase (glass matrix) of spinel, silicate and mixed (spinel-silicate) composition, while whistle, which occurs in the form of skeletal separations in the glass matrix, is significantly less represented. Based on SEM analysis, these are Fe-Mn-Zn spinels. Lead and zinc alloys are the most common. Based on SEM analysis, these alloys are predominantly with copper. The grains of these phases range up to 100 µm, and appear almost exclusively as inclusions or inclusions, and in the best case simple to complex fusions with a glass matrix of elemental iron and vistitis, while those larger than 100 µm are mostly free or in the form of simple fusions. The presence of visible and "invisible" (structural) silver has not been determined in zinc alloys. Lead alloys almost always occur in the form of regular spheres. Unlike zinc alloys, lead alloys have the presence of visible, but also "invisible" (structural) silver, which was confirmed by SEM analyzes. Based on these analyzes, only a small part (about 10%) has silver with a content of up to 1%. Silver is oval in shape, even in the form of smaller wires whose dimensions go up to 5 µm. Except in these alloys, elemental silver, but also copper, appear in the glass matrix and whistle in the form of small inclusions that rarely exceed 2-3 µm (silver) and 7-8 µm (copper). It is important to note that elemental silver often occurs in cracks in the host's grain. Galena and sphalerite are in a subordinate position in relation to Pb and Zn alloys, and they are almost exclusively in the form of simple to complex fusions with these alloys. Ceruzite is traceable and is exclusively related to sphalerite in the form of simple and complex adhesions or inclusions. Apart from vistite, magnetite, hematite, troilite and spinel, iron also occurs in elemental form, but also as pyrite and arsenopyrite in a significantly smaller volume. Microphotographs are given below in Figures 2, 3, 4 and 5.

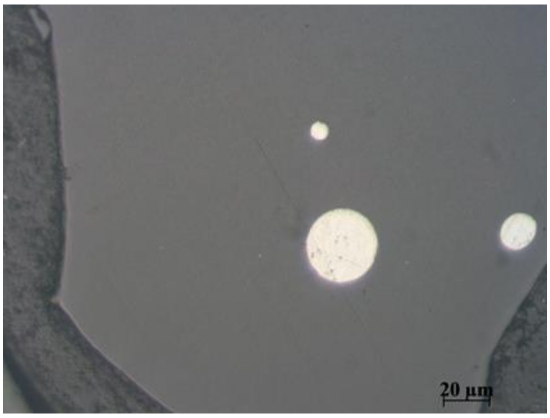

Figure 2. *Inclusions of spherical lead alloy in the glass matrix. Reflected light, air, II Nikoli.*

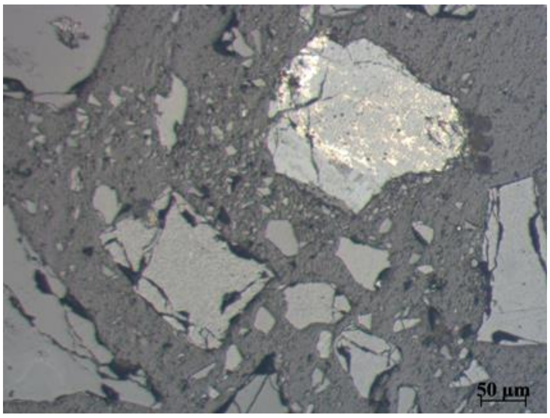

Figure 3. *Complex intergrown of Pb-Zn alloys with sphalerite, galena and cerusite (dark gray).. Reflected light, air, II Nikoli*

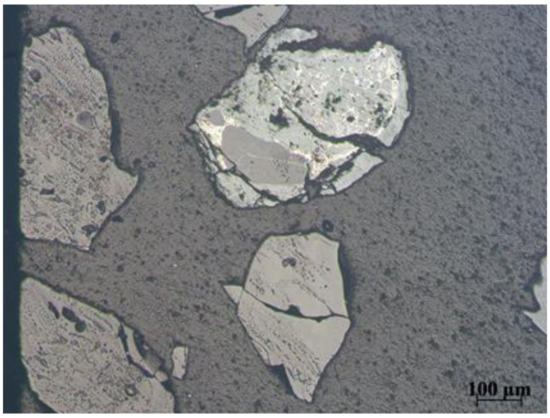

Figure 4. *Complex intergrown of Pb-Zn alloys with sphalerite, galena and vistite. Reflected light, air, II Nikoli*

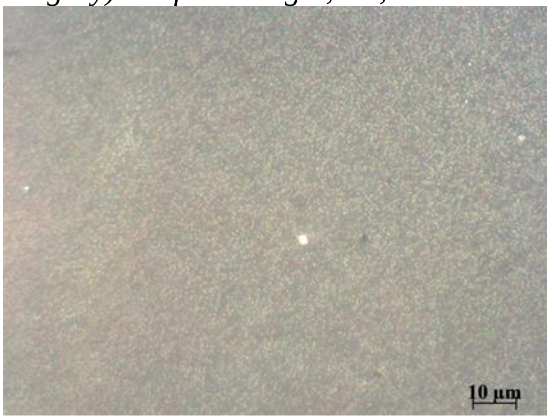

Figure 5. *Inclusion of elemental silver (bright white dot) in the glass matrix. Reflected light, air, II Nikoli*

Technological laboratory investigation

As part of the technological tests, the sample was first prepared for testing. Then, as part of technological tests, grinding tests were first performed, which were supposed to determine the conditions for the release of useful components from inert and harmful ones. In the second phase, the methods and procedures of concentration were examined, i.e. tests of the magnetic separation of useful components in separate products were performed.

Grinding experiments and mineralogical analysis of size classes have determined that useful components from Pb-Zn slag "Veles" are free at fineness of grind -0.1 + 0.00 mm. Based on this fact can be expected that such crushed technogenic raw material should be subjected to the process of magnetic separation, ie concentration, and in that way harmful and useless components should be removed. By applying this separation procedure, finished products, concentrates with increased metal content in relation to the input and with their satisfactory utilization should be obtained.

Magnetic separation should remove most of the elemental iron, magnetite and vistite, as well as the amorphous phases of the spinel and ferrite composition that contains iron in its structure. The sample was divided into two parts and one was used to perform magnetic separation experiments on a Davis analyzer, and the other part of the sample was used to perform experiments on a dry magnetic disk separator.

Magnetic separation of Pb-Zn slag "Veles" on a Davis magnetic analyzer

A sample of Pb-Zn slag from Veles was treated on a Davis magnetic analyzer, whereby the magnetic fraction (MF) and the non-magnetic fraction (NMF) were obtained. Chemical analysis of the following elements was performed on the magnetic separation products: Pb, Zn, Cu and Fe. The obtained results of chemical analysis together with product masses are arranged and presented in the form of material balance in Table 4.

Table 4. Magnetic concentration balance of Pb-Zn slag on a Davis analyzer

Product	M,%	Pb, %	Zn, %	Cu,%	Fe,%	IPb, %	IZn, %	ICu, %	IFe, %
MF	14.81	1.030	3.480	0.620	34.830	9.14	7.36	20.42	16.79
NMF	85.19	1.780	7.610	0.420	30.010	90.86	92.64	79.58	83.21
Feed	100.00	1.669	6.998	0.450	30.724	100.00	100.00	100.00	100.00

From the balance of the magnetic separation experiment on the Davis magnetic analyzer, at a magnetic induction of B = 0.4T, it can be seen that the separation of the magnetic and non-magnetic phases is not good. The relative mass fraction of the magnetic fraction is 14.81%, while the mass fraction of the non-magnetic fraction is 85.19%. Interestingly, the content of non-ferrous metals in the magnetic fraction is relatively high 1.03% Pb, 3.48% Zn and 0.62% Cu. The distribution of lead in MF is 9.14%, the distribution of zinc in this fraction is 7.36%, while the distribution of copper is 20.42%. The distribution of non-ferrous metals in the non-magnetic fraction are relatively large: Pb in NMF is 90.86%, Zn in NMF is 92.64%. The low concentration of iron-Fe in the magnetic fraction (34.83% Fe in MF) relative to the input and the non-magnetic fraction where the iron content is about 30% (30.724% Fe in the input, and 30.010% Fe in NMF), can be explained by that iron is probably present in the vitreous phase, spinels, franclinite, elemental iron, and vistite. Depending on the magnetism of these forms, one part passed into the magnetic fraction, while one part remained in the non-magnetic fraction. Thus, iron was concentrated in the magnetic fraction with a small degree of concentration of 1,134 in relation to the input, ie the content of Fe in the MF fraction increased in relation to the input by 13.4%. The utilization of iron in the magnetic fraction is low 16.79%, which means that its loss in the non-magnetic fraction (NMF) is 83.21%.

Magnetic separation of Pb-Zn slag "Veles" on a dry magnetic separator whit disc

A sample of Pb-Zn slag from Veles was treated on a dry magnetic separator whit disc, whereby the magnetic fraction (MF) and the non-magnetic fraction (NMF) were obtained. Chemical analysis of the following elements was performed on the magnetic separation products: Pb, Zn, Cu and Fe. The obtained results of chemical analysis together with product masses are arranged and presented in the form of material balance in Table 5.

Table 5. Magnetic concentration balance of Pb-Zn slag on a dry magnetic separator whit disc

Product	M,%	Pb, %	Zn, %	Cu,%	Fe,%	IPb, %	IZn, %	ICu, %	IFe, %
MF	53.11	1.380	6.440	0.490	32.720	42.90	48.97	56.92	55.38
NMF	46.89	2.080	7.600	0.420	29.860	57.10	51.03	43.08	44.62
Feed	100.00	1.708	6.984	0.457	31.379	100.00	100.00	100.00	100.00

From the balance of the magnetic separation experiment on the Davis magnetic analyzer, at a magnetic induction of B = 0.7T, it can be seen that the separation of the magnetic and non-magnetic phases is not good. The mass fraction of the magnetic fraction in this experiment is far higher than in the previous experiment 53.11%. The content of non-ferrous metals in the magnetic fraction is relatively high 1.38% Pb, 6.44% Zn and 0.49% Cu. There was an increase in the content of Pb and

Zn in the magnetic fraction. The content of copper (Cu) in the magnetic fraction of 0.49% is higher than in the input, where the content of Cu is 0.457%. The presence of Zn-zinc in the magnetic fraction can be attributed in part to the existence of franclinite, ZnFeO4 or amorphous forms that are chemically similar to franclinite. The Zn content in the magnetic fraction of the magnetic separator with a disk of 6.44% is much higher than in the previous magnetic separation test (3.48% Zn in MF on a Davis magnetic analyzer) and only slightly lower than the Zn content in the input sample where it is 6.984 %.

In the case of a magnetic separator with a disk, the mass participation of the magnetic fraction is 53.11%, so the utilization (loss) of non-ferrous metals in it is considerable. Thus, the distribution of lead in MF is 42.90%, the distribution of zinc in this fraction is 48.97%, while the distribution of copper in the magnetic fraction of the magnetic separator with disk is the largest of all non-ferrous metals 56.92%, because copper in this experiment concentrates in this fraction. The distribution of non-ferrous metals in the non-magnetic fraction are lower, so the lead in NMF is 57.10%, but the degree of Pb concentration in relation to the input is 1.218 times, which is a low degree of concentration, ie the Pb content in the NMF fraction increased 21.8%. the distribution of zinc in NMF is 51.03% but the degree of concentration of Zn in relation to the input is 1.088 times, which is a very low degree of concentration for zinc, ie the content of Zn in the NMF fraction increased in relation to the input by 8.8%. It is interesting to note that the degree of zinc concentration in the magnetic separator with disk in the non-magnetic fraction in relation to the initial sample is 1.088.

The low concentration of iron-Fe in the magnetic fraction (32.72% Fe in MF) relative to the input and the non-magnetic fraction where the iron content is about 30% (31.379% Fe in the input, and 29.86% Fe in NMF), can be explain that less magnetic minerals with lower iron content, such as spinels, and franclinite consisting of different divalent and trivalent cations (in this case slag Fe and Zn) also pass into the magnetic fraction. If a strong magnetic field separates franclinite in which the Zn content is significant and therefore the Zn content in the MF increases, it is logical that weaker magnetic minerals with lower Fe content are released in such a magnetic field and therefore the Fe content in the MF magnetic fraction of the magnetic separator with disk. Thus, iron was concentrated in the magnetic fraction with a small degree of concentration of 1,043 in relation to the input, ie the Fe content in the MF fraction increased in relation to the input by 4.3%. The distribution of iron in the magnetic fraction is 55.38%, which means that its loss in the non-magnetic fraction (NMF) is 44.62%.

Conclusion

Magnetic separation on the Davis analyzer at B = 0.4T of the "Veles" slag sample with a size of -1.0 + 0.0 mm showed that non-ferrous metals were separated in the magnetic fraction to a small extent (the content of non-ferrous metals in MF is 1.03% Pb, 3.48% Zn and 0.62% Cu). In this way, there was a loss of non-ferrous metals in the magnetic fraction, and in addition, their presence spoils the quality of this fraction in which iron (Fe) should be concentrated. At the same time, there was a low concentration of Fe in magnetic fraction of 34.83% Fe. It is important to note that there is a concentration of copper (Cu) in the magnetic fraction, which is not good from the point of view of the quality of this product.

On the other hand, in the non-magnetic fraction of NMF, there was a weak concentration of Pb and Zn, in relation to the input, while in this fraction there was a decrease in the content of Cu in relation to the input. It should also be noted that the Fe content in NMF is a significant 30.01%, which is a small decrease compared to the Fe content in the feedstock of 30.724%.

Based on all these data, it can be said that the Davis magnetic analyzer did not successfully separate Pb-Zn slag, ie the NMF did not obtain a significant concentration of non-ferrous metals relative to the input, even the copper content in this fraction was reduced compared to its content at the entrance.

Also, a significant concentration of iron in relation to the input was not achieved in the magnetic fraction.

In general, it can be said that by applying a stronger magnetic field (B = 0.7T) in the process of Pb-Zn slag separation, there was no serious separation of non-ferrous metals from iron-bearing components.

Namely, in the magnetic fraction, non-ferrous metals were separated to a significant extent (the content of non-ferrous metals in MF is 1.38% Pb, 6.44% Zn and 0.49% Cu). In this way, there was a significant loss of non-ferrous metals in the magnetic fraction, and in addition, their presence spoils the quality of this fraction in which, by the nature of things, iron (Fe) should be concentrated. At the same time, there was a weak concentration of Fe in MF of 32.72% Fe. It is important to note that there was a concentration of Cu copper in the magnetic fraction, which is not good from the point of view of the quality of this product.

On the other hand, in the non-magnetic fraction of NMF, there was a weak concentration of Pb and Zn, in relation to the input, while in this fraction there was a decrease in the content of Cu in relation to the input. It should also be noted that the Fe content in NMF is significant by 29.86%, which is a small decrease compared to the Fe content in the feedstock of 31.379%.

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