Investigation of the Influence of Ultrafine Grinding and Ultrasonic Field on the Intensification of the Processes of Extraction of Iron Oxides from Ash and Slag Waste of Energy

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Abstract. The possibility of extracting iron oxides from ash and slag wastes of the Primorskaya GRES power plant - (Luchegorsk, Primorsky Kray) was investigated. The positive influence of the energy of ultrasonic vibrations with frequency of 22 kHz and power at 1 kW, on the increase in the percentage of extraction of iron from ash and slag waste from thermal power is presented. The optimal time of exposure to ultrasound radiation on ash and slag pulp is determined. A comparison of the magnetic separation of ash and slag waste (ASW) without additional grinding with magnetic separation with preliminary grinding of sample materials down to 90 and 40 microns is carried out. The positive effect of preliminary grinding of the ASW sample on the efficiency of the subsequent magnetic separation is presented. With double magnetic separation of the ASW sample in a magnetic field of 300 T and preliminary crushing of the sample to a particle size class of less than 40 microns, a concentrate was obtained with an iron oxide content of 52.9; the extraction rate of iron oxides equaled 84.2%.

1. Introduction
Ash and slag waste from energy industry enterprises accumulated over the past years has a negative impact on the environment. The main danger is the ingress of effluents from ash dumps into the ground, leading to the flow of pollutants (heavy metals, radioactive elements, etc.) into groundwater, and with them into rivers and water bodies and, ultimately, into human food. When acid rains fall, the processes of ash and slag waste dissolution intensify, as a result of which an even greater volume of substances hazardous to human life flow into various water bodies [1, 2].

At the same time, ash and slag waste can be partially or completely recycled and serve as a source of a number of metals and elements [3-5]. Burned coals, being natural sorbents, contain admixtures of many valuable elements, including rare earth and precious metals. When burned, their content in ash increases 5–6 times and may be of industrial interest [6–8]. As studies by the third-party authors show, ash and slag waste contains a significant amount of iron oxides (from 5 to 15% depending on the types of source coals [9]), which can be extracted into a concentrate with an iron content of about 50%. Although the content of metals in ASW from thermal power plants is somewhat lower than in natural ores, ASW after their enrichment can become a new source of raw materials for enterprises of ferrous and non-ferrous metallurgy. The recent “raw material hunger” [10], associated with the gradual depletion of traditional natural resources for many metallurgical plants, only enhances the role of this field.
The iron concentrate is obtained from the ash and slag waste from CHPs via the method of magnetic separation based on the difference in the magnetic properties of the separated materials. However, the content of magnetite in ASW is several times lower than in the natural material. In power plant waste, iron is in a more oxidized (non-ferromagnetic) state. The degree of oxidation, defined as the Fe2O3 / FeO ratio, is 3.6-5.0 for ASW, and for iron ore of various deposits, this indicator ranges from 0.6-3.1. As a result, the magnetization of ash and slag waste is several times lower than in natural ore [11]. Therefore, for an effective separation of the iron oxides from ASW, multiple magnetic separation in a strong magnetic field may be required.

Since the ash particles, moving around within the magnetic field of the separator, are exposed to not only magnetic, but also mechanical forces, the degree of iron extraction from the ASW depends on the strength of the magnetic field, the size of the particles and the nature of the medium in which the movement occurs. As a result, wet separation is more efficient than dry separation, and grinding of ASW allows obtaining a concentrate with a higher iron content [12].

A promising method for obtaining mineral raw materials is acoustic (ultrasonic) treatment, the use of which increases the efficiency of the processes of emulsifying substances, dispersing, cleaning surfaces, degassing, extracting, grinding and many others. When ultrasound passes through liquid and liquid-dispersed media, the phenomenon of cavitation and a number of accompanying effects - radiation pressure, electrical discharges, microflows, etc. This is a mechanism for the local concentration of a relatively low average acoustic field energy in very small volumes, which leads to the creation of extremely high energy densities [12, 13].

The experience of using methods for intensifying the processes of extracting valuable components from natural and technogenic raw materials shows that for solving each technological problem, there are optimal parameters of acoustic effects, which make it possible to achieve the maximum effect, taking into account the specifics of the processed raw materials. For example, in the results of studies [14] on the study of the possibility of mechano-physical activation of semi-coke ash using ultrasound are presented. Ultrasonic treatment was conducted in aqueous suspensions based on the non-reagent treatment of elastic mechanical vibrations on the structure and properties of the material. As a result of ultrasonic treatment with a frequency of 35 kHz with an intensity of 3 W/cm² on the suspension for 8-10 minutes at a temperature of 65-68°C, it is possible to significantly increase the output percentage of the valuable elements during the subsequent sulfuric acid leaching of ash. The work [16] describes the technology of purification from iron oxides of quartz glass sands. Mechanical scrubbing of the sand did not provide sufficient complete removal of oxides, although it was carried out for 30-40 minutes. Using ultrasound with a frequency of 20 kHz and an intensity of 3 W/cm², the authors of the study were able to reduce the processing time to 2-5 minutes and remove oxides more accurately. In turn, the imposition of an ultrasonic field with a frequency of 35 kHz with a power of 1.5 W/cm² in a sulfuric acid solution with a concentration of 5% provides an intensification of the leaching process by 40 times for chalcopyrite, 26.5 times for sphalerite, and 20 times for pyrite [15].

Thus, the presented work is aimed at studying the possibilities of intensifying the processes of iron oxides extraction from ASW from the power industry enterprises by exposing samples to an ultrasonic field with a frequency of 22 kHz to 35 kHz.

2. Materials and methods

As a target of research, we have used samples of ash and slag wastes taken from the landfill of Primorskaya GRES power plant (Luchegorsk). Sampling of ASW was carried out at different depths from 0.5 m to 1.5 m from the surface of the ash landfills. The distance between the sampling points ranged from 20 to 100 m. The coordinates of the sampling were fixed using the navigation system.

Point samples were taken on sample plots from one or more layers, or horizons. Samples were taken diagonally at 7-10 points at equal distances, in the amount required to obtain the initial sample. A combined sample was formed from the point samples, compiled by mixing point samples (at least five) taken at the same site. The mass of the spot sample was at least 500 g. The mass of the combined sample was 5 kg.
The determination of the elemental composition of the ASW samples and the content of iron oxides in the samples was conducted via X-ray fluorescence analysis using an energy-dispersive X-ray fluorescence spectrometer "EDX-800HS" (Japan).

Mineralogical analysis was performed using a Zeiss LSM 510 Meta confocal microscope (Germany).

The study of the granulometric composition of the initial sample was carried out on laboratory vibrating sieves and on the vibratory sieve shaker Analysette 3 from Fritsch, the sieves were cleaned using an ultrasonic bath Laborette 17 from Fritsch.

In order to disperse ash and slag samples down to size classes less than 90 microns and less than 40 microns, a dry ball mill MSHP-06 (Russia) was used.

Laboratory studies of the influence of ultrasonic cavitation (US-cavitation) on the efficiency of extraction of iron oxides from ASW were carried out according to the following scheme (see Figure 1):

- The original bulk samples of ash and slag waste were subjected to preliminary flotation treatment at the FML-12 unit (Russia) and subsequent calcination for the complete removal of the underburned coal.

- Large samples of 30 kg each were taken from the ASW material that had undergone preliminary preparation. The mass of enlarged samples was determined based on the technical characteristics of the used magnetic separator PBM-P-25-10 (Russia), which requires a sample weighing at least 15 kg for effective operation.

- The enlarged ASW sample was divided into two equal parts (15 kg each).

- One part of the sample was sent directly to magnetic separation on PBM-P-25-10, with a magnetic field strength of 300 T and a rotation frequency of 30 rpm.

- The second part of the sample was preliminarily exposed to ultrasonic cavitation in an ultrasonic technological apparatus UZAP-1/22-OPST (flow-through type, radiation frequency - 22 kHz, power - 1 kW, radiation intensity - at least 15 W/cm²). The radiation exposure time ranged from 1 to 30 min. After exposure to ultrasonic cavitation, each sample was also sent for magnetic separation via PBM-P-25-10. When studying the effect of the ultrasonic cavitation on the process of wet enrichment of ash and slag waste by the content of iron oxides, to obtain pulp (a mixture of water with ASW), the S: L ratio (1:12) was chosen, corresponding to the average ratio in the line that removes ash and slag from boilers TPP to the ash landfills. In the process of ultrasonic irradiation, the pulp was stirred, simulating its real state when passing through the ultrasound emitter.

- The samples obtained after passing through the magnetic separation were subjected to elemental and mineralogical analysis. In the magnetic fraction, the content of iron oxides was determined via the XRD method.
Laboratory studies of the effect of ultrafine grinding on the efficiency of extraction of iron oxides from ash and slag waste were carried out according to the following scheme (Fig. 2):

- The magnetic fraction of ASW, which has undergone a single magnetic separation in accordance with the diagram in Fig. 1 (in the variant without ultrasonic cavitation), was dried, after which a 45 kg sample was taken from the obtained material.
- The selected sample of the magnetic fraction of ash and slag waste was divided into three equal parts (15 kg each).
- One part of the sample was sent directly to magnetic separation via PBM-P-25-10, with a magnetic field strength of 300 T and a rotation frequency of 30 rpm.
- The second part of the sample was preliminarily subjected to disintegration to a particle size class of less than 90 microns on a dry grinding ball mill MSHP-06, after which it was sent for magnetic separation on PBM-P-25-10, with a magnetic field strength of 300 T, a rotation frequency of 30/min.
- The third part of the sample was preliminarily subjected to disintegration to a particle size class of less than 40 microns on a dry grinding ball mill MSHP-06, after which it was sent for magnetic separation on PBM-P-25-10, at a magnetic field strength of 300 T and a rotation frequency of 30/min.
- The samples obtained after passing through the magnetic separation were subjected to elemental analysis. In the magnetic fraction, the content of iron oxides was determined via the XRD method.

**Figure 1.** Scheme of laboratory studies of the ultrasonic cavitation effect on the efficiency of iron oxides extraction from ASW.
Results
As shown by the studies of the initial samples of ash-and-slag waste, the main ash-forming chemical compounds are silicon, aluminum and iron (Table 1). The studied samples also contain macro-amounts of calcium, potassium, magnesium, sulfur and titanium. All samples were found to contain trace amounts of chromium, copper, nickel, cobalt, vanadium, tin, zinc and tungsten. The iron content in the samples ranges from 6.0 to 8.5%.

The results of mineralogical analysis show that the studied ASW samples have high contents of iron oxides (about 7%), mainly in the form of magnetite, partly in the form of intergrowths of quartz and magnetite (Table 2).

The results of the performed granulometric analysis show that ash particles with a particle size of less than 0.25 mm account for more than 75% of the total sample volume (Table 3). The major part of the studied sample is represented with fine and finely dispersed floating material.

In fact, the studied ASW represent technogenic deposits of valuable components, resource reserves of which are constantly replenished. Efficient processing of this class of waste with a complex chemical composition is possible only when ash and slag waste is separated into mineral monofractions.

Table 1. The average content of the main chemical elements in the initial samples of ASW from the Primorskaya GRES landfill (according to RFA data).

| Element | Si   | Al  | Fe  | Ca  | Mg  | K   | S    | Ti  | Ba  | Mn  | Sr  |
|---------|------|-----|-----|-----|-----|-----|------|-----|-----|-----|-----|
| Content, % | 59.56 | 24.71 | 7.52 | 2.98 | 1.76 | 1.4 | 0.79 | 0.6 | 0.22 | 0.18 | 0.12 |
Table 2. The mineralogical composition of the initial samples of ASW from the Primorskaya GRES landfill.

| No | Minerals | Fractions, mm | Total |
|----|----------|---------------|-------|
|    |          | -2.5+0.5 | -0.5+0.2 | -0.2+0.063 | -0.063+0 | %     |
| 1  | Magnetite (crystalline, fragments of crystallites, fused crystals) | 0.6918 | 1.2211 | 4.3214 | 1.5551 | 7.7894 |
| 2  | Magnetic spheres | - | 1.2748 | 4.8210 | 1.7662 | 7.8620 |
| 3  | Brown coal, rarely stone coal | 8.6042 | 4.3226 | 3.0297 | 3.5289 | 19.4854 |
| 4  | Magnetic slag | 0.3828 | 3.8957 | 15.4475 | - | 19.7260 |
| 5  | Porous clay slag | 5.7230 | 16.9504 | 5.3238 | - | 27.9972 |
| 6  | Clay (white, gray) | 0.3794 | 0.0748 | - | - | 0.4542 |
| 7  | Clay with inclusions of magnetite spheres | 0.3694 | - | - | - | 0.3694 |
| 8  | Marcasite, pyrite | - | - | 0.0004 | - | 0.0004 |
| 9  | Feldspar | - | 0.0060 | - | - | 0.0060 |
| 10 | Quartz | 0.6015 | 4.9263 | 5.3558 | - | 10.883 |
| 11 | Quartz, feldspar | - | - | - | 2.4805 | 2.4805 |
| 12 | Amphiboles | - | 0.0236 | 0.0309 | - | 0.0545 |
| 13 | Rhyolites, rhyodacites, diorites | 0.8668 | - | 0.0010 | - | 0.8687 |
| 14 | Splices: | | | | | |
| 15 | Magnetite + quartz | 0.2149 | - | - | - | 0.2149 |
| 16 | Quartz + magnetite | - | 0.4591 | 0.0010 | - | 0.4601 |
| 17 | Coal with magnetite inclusions | 0.0201 | - | 1.3265 | - | 1.3466 |
| 18 | Accessory: zircon | - | 0.0010 | 0.0010 | - | 0.0020 |
| **Total, %** | 17.8539 | 33.1554 | 39.6600 | 9.3307 | 100.00 |

Table 3. Results of granulometric analysis, after wet sifting of the ASW sample.

| Particle size | 4 mm | 2 mm | 1 mm | 0.5 mm | 0.25 mm | 0.125 mm | 0.063 mm | <0.063 mm | Total, % |
|---------------|------|------|------|--------|---------|----------|----------|----------|---------|
| Quantity, % of the dry weight | 1.52 | 6.84 | 7.51 | 5.26 | 17.55 | 31.22 | 23.50 | 6.61 | 100.00 |

The results of the laboratory tests on the effects of ultrasound exposure on the efficiency of extraction of iron oxides in wet magnetic separation are presented in Table 4 and Fig. 3-4. The study of samples for the content of iron in them (by the XRD method) was carried out after processing the samples on a magnetic separator, in accordance with the scheme shown in Fig. 1. Used in the experiments processing ("magnetization") with the determination of the iron content was carried out each time after exposure to ultrasonic cavitation.

Table 4. Results of laboratory tests to study the effect of the ultrasonic cavitation effect on the efficiency of iron oxides extraction from ASW during wet separation.

| Time of ultrasonic impact | Initial sample | 1 min | 5 min | 10 min | 15 min | 20 min | 25 min | 30 min |
|---------------------------|----------------|-------|-------|--------|--------|--------|--------|--------|
| Fe content in samples, %  | 45.55          | 45.60 | 46.00 | 46.70  | 47.40  | 47.90  | 48.10  | 48.12  |
| Increase in Fe content, % | 0.00           | 0.050 | 0.400 | 0.700  | 0.700  | 0.500  | 0.200  | 0.014  |
| Increase in Fe content, % from the previous quantity | 0.000 | 0.110 | 0.877 | 1.522  | 1.499  | 1.055  | 0.418  | 0.029  |
The tests conducted show that the ultrasound treatment of the ash and slag pulp made it possible to increase the content of iron oxides in the magnetic concentrate from 45.55 to 48.12%. Analyzing the data obtained, it can be concluded that under the conditions selected for the experiment (radiation power 1 kW), in order to obtain an optimal enrichment result, the processing of ash and slag waste should be carried out within no more than 30 minutes.

As can be seen from the results of elemental analysis (see Table 5), ultrasonic treatment has a positive effect on the extraction of titanium, generally increasing the efficiency of magnetic separation.

### Table 5. The elemental composition of the ASW magnetic fraction at the different duration of ultrasonic impact.

| Content, % | Duration of ultrasonic impact, min |
|------------|-----------------------------------|
|            | 0       | 1       | 5       | 10      | 15      | 20      | 25      | 30      |
| Fe         | 45.55   | 45.60   | 46.00   | 46.70   | 47.40   | 47.90   | 48.10   | 48.12   |
| Si         | 27.16   | 27.13   | 26.94   | 26.57   | 26.22   | 25.92   | 25.83   | 25.72   |
| Al         | 17.30   | 17.29   | 17.14   | 16.95   | 16.69   | 16.57   | 16.46   | 16.70   |
| Ca         | 6.14    | 6.13    | 6.09    | 6.00    | 5.93    | 5.86    | 5.84    | 5.62    |
| Mn         | 1.62    | 1.62    | 1.61    | 1.59    | 1.57    | 1.56    | 1.55    | 1.55    |
| S          | 1.04    | 1.04    | 1.04    | 1.02    | 1.02    | 1.02    | 1.03    | 1.07    |
| P          | 0.54    | 0.54    | 0.54    | 0.53    | 0.53    | 0.52    | 0.52    | 0.52    |
| K          | 0.22    | 0.22    | 0.21    | 0.21    | 0.21    | 0.21    | 0.21    | 0.21    |
| Ti         | 0.16    | 0.16    | 0.16    | 0.16    | 0.16    | 0.17    | 0.18    | 0.21    |
| Ba         | 0.10    | 0.10    | 0.10    | 0.11    | 0.11    | 0.11    | 0.11    | 0.12    |
| Cr         | 0.06    | 0.06    | 0.06    | 0.06    | 0.06    | 0.06    | 0.06    | 0.06    |
| Sr         | 0.05    | 0.05    | 0.05    | 0.05    | 0.05    | 0.05    | 0.05    | 0.06    |
| Cu         | 0.03    | 0.03    | 0.03    | 0.03    | 0.03    | 0.03    | 0.04    | 0.04    |
| Zr         | 0.02    | 0.02    | 0.02    | 0.02    | 0.02    | 0.02    | 0.02    | 0.01    |

With regard to the mineralogical composition of samples subjected to ultrasonic and electromagnetic irradiation, it can be concluded that the transformation proceeds according to certain mineral associations (Table 6). Therefore, after exposure to ultrasound in the samples of the magnetic fraction,
the content of magnetic spheres increased by 2%, in turn, the amount of magnetic slag decreased by 2%,
intergrowths of a complex complex composition (quartz + magnetite + coal) were no longer recorded.
Such results can be interpreted from the point of view of the peculiarities of the mechanism of the effect
of ultrasound radiation (in particular) cavitation on mineral complexes. The shock impact of ultrasonic
cavitation of a certain power could destroy the associations of dissimilar mineral complexes along the
spikes, which influenced the change in the mineral composition of the samples of the ASW magnetic
fraction.

Table 6. Mineralogical composition of the ASW magnetic fraction, before and after 30 min of
ultrasonic impact.

| No | Minerals                                      | Content (before ultrasonic impact), % | Content (after ultrasonic impact), % |
|----|----------------------------------------------|--------------------------------------|--------------------------------------|
| 1  | Magnetite (crystalline, fragments of crystals, fused crystals) | 17.21                               | 18.82                                |
| 2  | Magnetic spheres                              | 28.54                               | 30.54                                |
| 3  | Brown coal, rarely stone                      | 5.12                                | 4.83                                 |
| 4  | Magnetic slag                                 | 35.45                               | 33.27                                |
| 5  | Porous clay slag                              | 7.22                                | 6.87                                 |
| 6  | Clay (white, gray, red)                       | 0.44                                | 0.45                                 |
| 7  | Clay with inclusions of magnetite spheres     | 0.81                                | 0.80                                 |
| 8  | Quartz                                       | 2.52                                | 2.61                                 |
|    | Splices:                                     |                                      |                                      |
| 9  | Quartz + magnetite + coal                    | 0.45                                | 0.00                                 |
| 11 | Coal with magnetite inclusions               | 2.24                                | 1.81                                 |
|    | **Total:**                                    |                                      | 100.00                               |

In order to increase the efficiency of extraction of iron oxides from ash and slag waste, the effect of
ultrafine grinding was investigated in accordance with the scheme shown in Fig. 2. Brief results of the
research carried out are presented in (Table 7). As you can see, additional disintegration of ASW made it possible to significantly increase the content of iron oxides in the magnetic concentrate. The maximum content of iron oxides in the concentrate of 52.9% was achieved during repeated magnetic separation of the ash and slag waste sample from Primorskaya SDPP in a magnetic field of 300 T, with preliminary crushing of the sample to a particle size class of less than 40 microns., The total recovery of iron oxides into the concentrate from the original ash and slag samples, while it was 84.2% (Fig. 5).

Table 7. Results on the iron content in mineral concentrates of the ASW magnetic fraction from Primorskaya GRESS power plant, according to XRD data.

| No | Description of the sample                                      | Fe content, % |
|----|----------------------------------------------------------------|--------------|
| 1  | Initial iron-containing concentrate after primary separation of ASW from Primorskaya GRES | 45.8         |
| 2  | Iron-containing concentrate (Sm. 1) after secondary magnetic separation in a field with a strength of 300 T, without preliminary sample grinding | 48.8         |
|    | Iron-containing concentrate (Sm. 1) after secondary magnetic separation in a field with a strength of 300 T, with preliminary grinding of the sample to a size class of less than 90 microns | 52.2         |
| 3  | Iron-containing concentrate (Sm. 1) after secondary magnetic separation in a field with a strength of 300 T, with preliminary grinding of the sample to a size class of less than 90 microns | 52.9         |
| 4  | Iron-containing concentrate (Sm. 1) after secondary magnetic separation in a field with a strength of 300 T, with preliminary grinding of the sample to a size class of less than 40 microns | 52.9         |
Figure 5. Magnetic concentrate with an iron oxide content of 52.9%, obtained by 2-fold magnetic separation from ASW from the Primorskaya GRES landfill (Luchegorsk).

4. Conclusions
Laboratory studies have shown a positive effect of the energy of ultrasonic vibrations, with a frequency of 22 kHz and a power of 1 kW, on an increase in the percentage of iron extraction from ash and slag waste from thermal energy. The optimal time of exposure to ultrasound radiation on the research object is 30 minutes.

Additional disintegration of ash and slag material to a particle size of less than 90 microns made it possible to increase the content of iron oxides in the magnetic fraction from 48.8 to 52.2%. Grinding to a particle size of less than 40 microns showed a slight increase in the content of iron oxides from 52.2 to 52.9%.

The maximum recovery of iron oxides into the concentrate of the magnetic fraction of 84.2% was achieved via two-step magnetic separation of the ASW sample in a field of 300 T, with preliminary crushing of the sample to a particle size class of less than 40 microns.

The studies conducted have shown the possibility of increasing the efficiency of separation of the concentrate of minerals of the magnetic fraction from ASW by preliminary extraction of the mineral raw materials using ultrasonic cavitation and ultrafine grinding.

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