

Article

Application of a Simple Pretreatment in the Process of Acid Leaching of Electric Arc Furnace Dust

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Abstract: Experimental investigations of the effectiveness of the pretreatment of the acid leaching process of a representative sample of electric arc furnace dust (EAF dust) and characterization of a representative sample were performed. As EAF dust is globally defined as hazardous solid industrial waste, and it contains a high percentage of zinc and other useful components, in this paper, the application of pretreatment will be carried out in order to increase the efficiency of the further process of acid leaching, all with the aim of using EAF dust as a secondary raw material for metal valorization. The pretreatment involved the application of a simple process of leaching the EAF dust sample with distilled water. Under the following defined optimal leaching conditions: time—30 min, ratio of solid to liquid phase—1:10, temperature—ambient, and stirring speed—500 rpm, a reduction in the content of water-soluble components was achieved, namely, 83% Cl, 92% K, 74% Na, 14% Cd, 15% Ca, and 19% F. The application of the pretreatment enables significantly better filtration of the suspension and increases the leaching efficiency of zinc and other useful components in the further process of leaching EAF dust with a sulfuric acid solution, compared with the process of direct acid leaching of EAF dust.

Keywords: pretreatment; EAF dust; water leaching; hazardous waste



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1. Introduction

The basic method of recycling waste iron is in the steel production process by melting iron in electric arc furnaces. Across the world, the steel production process from secondary raw materials (waste iron) using electric arc furnaces is becoming more common. One of the steel mills in which this process is performed is located in the Republic of Serbia. The main sources of secondary raw materials for steel production are waste construction materials, old cars, appliances, and household waste, which means that the waste can contain a large number of metals, plastics and rubber, glass, paint, oil, and even salts [1–9]. Besides that, fluxes (additives) of limestone or lime, reductants, such as brown coal or coke, and oxygen and/or natural gas are injected into electric arc furnaces in addition to scrap iron [2–4,10–17].

An electric arc furnace reaches a temperature of 1600 °C, during which the elements from the iron melt evaporate and the main intermediate product of the process is generated—electric arc furnace dust (EAF dust) [2,3,7,8,15–18].

The generation of EAF dust occurs because volatile elements, such as zinc, lead, and cadmium, in the batch mostly evaporate in the early stages of the operation, and eventually, each of them passes into the gas phase. Other elements, such as iron, chromium, nickel, and manganese, are generated from gaseous products during the refining period, mainly because of oxygen injection. The emitted volatile components are quickly converted into the corresponding oxides in contact with air after the combustion and cooling of the exhaust

gases [1,2,4,18–21]. The oxides in the slag mixed with the metal vapors and generated dust are removed from the furnace by the rising hot gas flow and then enter the gas treatment system along with the gas flow, which is oxidized, vulcanized, or chlorinated in the dust collection system, forming EAF dust that is collected in gravity or electrostatic dust collectors and bag filters [2,5,12,16,20,22–25]. Purified gas containing small amounts of nitrogen oxides and ozone meets environmental standards and is released into the atmosphere [23–26].

Generally, during the production of one ton of crude steel, about 10–20 kg of EAF dust is generated [27]. To a large extent, the composition of EAF dust depends on the electric arc furnace operating conditions, the characteristics of the scrap iron charged to the furnace, the operating period, and the specification of the produced steel, and it is also specific to each plant [1,12,17,18,23–25,27–34].

The dust from an electric arc furnace contains significant amounts of zinc and solid iron oxides, as well as variable amounts of lead, calcium, manganese, nickel, copper, cadmium, magnesium, silicon, chromium, fluorine, chlorine, etc. [6,7,9,12,25,29–31,35–37]. The zinc content of EAF dust varies between 2 and 40% [5,22,26,27]. The presence of higher zinc content in EAF dust is a consequence of its widespread use to protect steel from corrosion or as a component of scrap brass [3,38,39]. Zinc in EAF dust is present in oxide phases such as $ZnFe_2O_4$ and ZnO , iron as Fe_3O_4 and Fe_2O_3 , calcium as CaO and $CaCO_3$, lead, copper, chromium, and nickel can appear in the form of oxides, chlorides, sulfides or sulfates, and chlorine in the form of alkali metal chlorides, while fluorine occurs in the form of lead and zinc fluorides [8,23,24,28,40,41].

Because of the fact that it contains heavy metals, EAF dust is a toxic product. In many countries of the world, it is considered a hazardous solid industrial waste, and therefore, it represents a problem for disposal in an environmentally friendly way because it has the potential to cause long-term environmental pollution [21,24,29,31–33,35–37].

This type of hazardous waste requires additional attention and the application of appropriate treatment in order to protect the environment and human health. The treatment of this type of hazardous waste can be performed by hydrometallurgical, pyrometallurgical, or combined process [3,6,9,20,28,30,42,43]. Apart from the application of appropriate treatment of EAF dust, primarily in order to protect the environment from the negative impact of this hazardous waste, this material can also be used as a secondary raw material for zinc recovery, with adequate economic profit [8,31,32,41].

The application of the hydrometallurgical process of the treatment of secondary and waste materials is an economical option for metal recovery, even at their low concentrations in raw materials. Hydrometallurgical treatment is carried out at relatively low temperatures (below 100 °C) using aqueous solutions containing cheap chemicals, without the generation of toxic and volatile solid particles or gases, and the process itself is economical even with a small production volume. The hydrometallurgical process includes the following three technological phases: leaching (acidic, alkaline, or ammoniacal), purification of the solution, and metal/compound recovery [44–53], which is presented in Table 1 as a literature overview of the application of the hydrometallurgical treatment of secondary and waste materials containing zinc.

This paper describes experimental investigations of a simple pretreatment process in the process of the acid leaching of EAF dust. The investigation results have shown that this pretreatment has significant advantages. This pretreatment greatly reduces the content of certain elements, i.e., water-soluble components from the initial EAF dust sample. This leads to a better filtration of the suspension, an increase in the leaching efficiency of zinc and other useful components from the EAF dust, and a lower consumption of leaching reagent. In addition to efficiency and economic profitability, the application of the proposed pretreatment is also reflected in the fact that its application reduces the amount of this type of hazardous waste by 6% of the initial amount.

Table 1. Literature overview of the application of hydrometallurgical processes for zinc extraction from secondary and waste materials containing Zn.

Type of Treated Waste Material	Hydrometallurgical Process Phases			References
	Leaching Process (Reagent Used)	Solution Purification Process	Extracting Metals/Zn Compounds Process	
EAF dust	1.5 M H ₂ SO ₄	-	-	[21]
EAF dust	-	Chemical precipitation of Fe, solvent extraction of Zn	Electrowinning process (EW)	[44]
EAF dust	6 M NaOH	-	-	[50]
EAF dust	First phase 1.0 M H ₂ SO ₄ Second phase 1.5 M H ₂ SO ₄	-	-	[51]
EAF dust	First phase water leaching, Second phase 100 g/L (NH ₄) ₂ CO ₃	Cementation	Precipitation of ZnCO ₃ , calcination ZnCO ₃ to ZnO	[19]
EAF dust	16 different reagents (acid, base, etc.)	-	-	[38]
Two filter dusts and zinc ash	25% H ₂ SO ₄	Chemical precipitation of Fe	Electrowinning process (EW)	[47]
EAF dust	5 M NaOH	Chemical precipitation	Electrowinning process (EW)	[52]
EAF dust	2 M NaOH, KOH, and LiOH	-	-	[8]
Zinc ash	20 % H ₂ SO ₄	Chemical precipitation of Fe	Electrowinning process (EW)	[45]
Zn smelting slag	2 M H ₂ SO ₄	Chemical precipitation	-	[53]

2. Experimental Part

2.1. Materials

The subject of investigation in this paper is hazardous solid industrial waste, i.e., dust from an electric arc furnace from a dust collection system, more precisely, from a bag filter, from a steel mill in the Republic of Serbia. A sample of the EAF dust was taken from the steel production process for the purpose of experimental investigations. Sampling of the EAF dust was carried out from the disposal site in the steel mill area, randomly, from the jumbo bags in which EAF dust was disposed.

2.2. Sample Preparation

By mixing the sample on the foil, the EAF dust sample was homogenized, and then, the representative sample was sampled using the quartering method.

For physical and chemical characterization, a representative sample of the EAF dust was taken in triplicate, in amounts of 0.5, 1.0, and 2.0 kg. The samples were then dried in a dryer at a temperature of 105 °C for 24 h.

For the examination of the morphology of the EAF dust sample, two dry representative samples in amounts of 5.0 g each were prepared by dipping in epoxy resin, after which they were ground and polished with silicon carbide and then polished with a diamond suspension of dimensions 6, 3, and 1 µm. After steaming with gold in a gold steaming chamber, the samples were analyzed with a Scanning Electron Microscope with Energy Dispersive X-ray Spectroscopy (SEM–EDS, JSM IT 300LV, JOEL, Tokyo, Japan).

Two dry representative samples in the amount of 1.0 g each were prepared by comminution in an agate mortar, in order to determine and monitor the phase composition of a representative sample of the EAF dust using X-ray diffraction analysis (XRD, RigakuMini-Flex 600, Tokyo, Japan).

2.3. Methods of Characterization of the EAF Dust Representative Sample

2.3.1. Physical and Chemical Characterization

The physical characteristics of the initial representative EAF dust sample included the determination of the pH value of the sample, bulk density, moisture, and density of the sample.

For metal content determination, a representative sample of the EAF dust was dissolved in 4 acids (HCl, HClO₄, HNO₃, and HF), and then the resulting solutions were analyzed. The concentrations of Co, Fe, Na, Mn, Sn, Bi, As, Cr, Mo, Al, P, Ni, Cu, Sb, Ca, Pb,

Cd, Si, K, and Mg were determined using an Inductively Coupled Plasma Atomic Emission Spectrometer device (ICP–AES, Spectro CirosVision, Borchen, Germany). Concentrations of metals with relatively higher values, such as Zn, Fe, Na, Mn, Pb, Cu, Ni, Ca, Ag, Mg, and K, were confirmed using an Atomic Absorption Spectrophotometer (AAS, PerkinElmer PinAAcle 900F, Waltham, MA, USA). As the concentration of Zn in a representative EAF dust sample was the highest compared with all other metals, a more precise concentration of Zn was determined volumetrically by titration with ethylenediaminetetraacetic acid using methylthymol blue as an indicator. The sulfur content was determined using a Carbon and Sulfur Analyzer (CSA, Horiba EMIA–920V2, Osaka, Japan). For the determination of chlorine, fluorine, and pH values, solutions for analysis were obtained by suspending a representative sample of the EAF dust in demineralized water at a ratio of 1:10, shaking, and filtering. The concentrations of chlorine and fluorine in the solutions were determined with a Spectrophotometer (SF, HACH DR 3900, Loveland, CO, USA). The pH value was measured with a pH meter (WTW inoLab pH 7310, Burladingen, Germany).

2.3.2. Particle Size Distribution

Using the laser device MASTERSIZER 2000 (MALVERN Instruments, Malvern, UK) on a representative EAF dust sample, without any prior preparation, investigations of the granulometric composition of the sample were performed.

2.3.3. Mineralogical Characterization

Mineralogical characterization of a dry representative EAF dust sample included Scanning Electron Microscopy with Energy Dispersive X-ray Spectrometer (SEM-EDS) and X-ray diffraction (XRD) analysis.

An SEM-EDS microscope (JSM IT 300LV, JOEL, Tokyo, Japan) was used to examine the morphology and elemental mapping of a representative EAF dust sample, with an accelerator voltage of 20 kV.

To determine the phase composition of a representative EAF dust sample and the solid residue obtained after the water leaching process, as polycrystalline samples (powders), XRD analysis was applied using an X-ray diffractometer (RigakuMiniFlex 600, Tokyo, Japan), with a “D/teXUltra 250” high-speed detector and an X-ray tube with a copper anode. The recording conditions were as follows: angle range 3–90°, step 0.02°, and recording speed 10°/min. The voltage of the X-ray tube was 40 kV, and the current was 15 mA. The identification and semi-quantitative assessment of minerals were performed in PDXL 2 Version 2.4.2.0 software, and the obtained diffractograms were compared with the data from the COD database.

2.4. Pretreatment of the EAF Dust

The chemical and mineralogical compositions of EAF dust originating from the steel production process are individual, which means that their processing technology must be examined separately, depending on the goals to be achieved. In this paper, the EAF dust processing technology is based on a hydrometallurgical treatment, which includes a simple and inexpensive selective leaching process of water-soluble components, as a pretreatment for the next technological step—acid leaching.

Considering that zinc in EAF dust occurs mainly in the form of ZnO and ZnFe₂O₄, further research was planned on the acid leaching of the solid residue obtained after the pretreatment for zinc recovery. The leaching of zinc from ZnO can be easily carried out in both alkaline and acidic mediums; however, part of the zinc present in the form of ZnFe₂O₄ can only be leached in an acidic medium, with the application of more rigorous conditions of the leaching process (using a stronger concentration of acids and higher process temperature values), and for this reason, future investigations of the EAF dust treatment were to be conducted using an acid rather than a base [3,4,12]. In addition to the present zinc compounds, calcium, magnesium, potassium, sodium, and chlorine compounds are present in the dust, which, during the acid leaching process of EAF dust, increase the

consumption of sulfuric acid and also lead to the contamination of the pregnant leaching solution (zinc sulfate solution), from which it is possible to obtain zinc-based products. In this paper, as the first stage of the hydrometallurgical treatment of the EAF dust, the process of the selective leaching of the EAF dust was investigated. Considering that most of the mentioned compounds are soluble in water, for the process of the selective leaching of EAF dust, the leaching process with distilled water was investigated. The selectivity of the water leaching process is reflected in the fact that its application results in the removal of water-soluble components (calcium, sodium, potassium, chlorine, cadmium, fluorine) without dissolving other metals present in EAF dust [2,51,54,55].

The influence of time, a ratio of solid and liquid phases, and temperature on the leaching efficiency of water-soluble components from the EAF dust was investigated by experimental laboratory investigations. All experiments were conducted on the EAF dust samples in amounts of 50.00 g each, mixed with distilled water in appropriate proportions, in the reaction vessel using a magnetic stirrer with automatic speed and temperature control. After the experiments were completed, the suspension was vacuum-filtered. The solid residues, after being washed with distilled water, were dried in a dryer for 24 h at 80 °C. The obtained pregnant leaching solutions were chemically analyzed for the content of Na, Cl, K, Cd, Ca, F, Zn, Fe, Cu, Cr, Ni, and Pb, and the leaching efficiencies of the monitored elements were calculated based on the obtained results. All experimental investigations on the influence of different parameters on the leaching efficiency of water-soluble components were carried out in triplicate in order to achieve repeatability of the results. The solid residue obtained under optimal water leaching conditions was comminuted in an agate mortar, homogenized, and prepared for XRD and chemical analysis.

2.4.1. Influence of the Leaching Time on the Leaching Efficiency of the EAF Dust

The influence of the leaching time of 10, 15, 30, 45, and 60 min on the leaching efficiency of soluble components from the EAF dust in distilled water was investigated. The experiments were performed under the following conditions: S:L ratio = 1:5, stirring speed: 500 rpm, and temperature: ambient (~20 °C).

2.4.2. Influence of the Solid and Liquid Phase Ratio on the Leaching Efficiency of the EAF Dust

The investigations of the influence of solid and liquid phase ratios of 1:5, 1:10, 1:15, and 1:20 on the leaching efficiency of soluble components from the EAF dust in distilled water was carried out under the following constant conditions: leaching time: 30 min, stirring speed: 500 rpm, and temperature: ambient (~20 °C).

2.4.3. Influence of the Temperature on the Leaching Efficiency of the EAF Dust

The influence of temperature on the leaching efficiency of the water-soluble components from the EAF dust in distilled water was performed under the following conditions: leaching time: 30 min, stirring speed: 500 rpm, and ratio S:L = 1:10. The investigated temperatures were as follows: ~20 °C (ambient), 30, 40, 50, and 60 °C.

2.5. Leaching of the Solid Residue Obtained after the Pretreatment

Given that zinc in EAF dust occurs in the form of $ZnFe_2O_4$ in addition to ZnO , in order to destroy these crystal structures and perform zinc recovery, the solid residue obtained after the applied pretreatment was leached with a more aggressive leaching reagent, i.e., sulfuric acid. A literature survey showed that H_2SO_4 solutions are generally used for the acid leaching of EAF dust [6,14,21,38,39,45,47,51,56].

After the applied pretreatment, in experimental laboratory investigations, the influence of four different process parameters, including the time of the acid leaching process, the concentration of sulfuric acid, the ratio of the solid to liquid phase, and the temperature of the process, was investigated on the leaching efficiency of the EAF dust components in the acid leaching process. The leaching solution was prepared using distilled water

and analytical grade H₂SO₄ in the desired concentrations. The amount of solution was calculated based on the desired ratio of the solid to liquid phases. All experiments were carried out in triplicate on samples of the solid residue of the EAF dust obtained after the performed pretreatment under the defined optimal conditions. After separating the phases by filtration, the obtained pregnant leaching solutions and dried solid residues were chemically analyzed for the content of Zn, Fe, Pb, Cd, Na, K, Ca, Cu, and Ni.

3. Results and Discussion

3.1. Characterization of a Representative EAF Dust Sample

3.1.1. Physical and Chemical Characterization

The results of the physical characterization of a representative EAF dust sample are as follows: pH value: 7.15, bulk density: 686 kg·m⁻³, moisture: 1.60 wt.%, and density: 4.446 g·cm⁻³.

The pH value of a representative EAF dust sample of 7.15 indicates that the EAF dust is a material with slightly basic characteristics, and thanks to adequate disposal, the moisture content of the EAF dust sample is low (below 2 wt.%).

The results of the chemical characterization of a representative EAF dust sample, as well as the analytical methods used, are presented in Table 2.

Table 2. Chemical composition of a representative EAF dust sample and analytical methods used.

Element	Content, wt. %	Analytical Method ¹
Zn	32.38	
Fe	28.28	
Mn	2.29	V
Cu	0.19	
Pb	1.11	
Bi	<0.01	ICP-AES
Co	0.0018	
Ni	0.014	
Cr	0.40	
Mo	0.0028	
S	0.44	CSA
P	0.11	
As	0.045	
Ba	0.090	
Li	0.0032	
Sb	0.014	
Sn	0.035	
Sr	0.0062	
Ca	3.05	ICP-AES
Ti	0.083	
Cd	0.032	
Al	0.77	
V	0.0097	
Na	0.92	
K	0.74	
Mg	0.57	
Cl	2.50	SF
F	0.023	
Ag	0.0073	AAS

¹ V—volumetry; CSA—Carbon and Sulfur Analyzer; ICP-AES—Inductively Coupled Plasma Atomic Emission Spectroscopy; SF—Spectrophotometry; AAS—Atomic Absorption Spectrophotometry.

Based on the results obtained by chemical analysis (Table 2), it can be concluded that the analyzed sample of EAF dust contains a large number of elements, which confirms the complexity of EAF dust as a material. In addition, the obtained results are in agreement with the literature [4,6,7,12,23,28]. The highest abundance in the sample is zinc with a content of 32.38%, followed by iron with 28.28%, while other elements, such as Cl, Ca, Mn, Pb, Ni, Cu, Cr, Cd, Na, K, F, Ti, P, and others, are less abundant in the analyzed EAF dust sample.

3.1.2. Particle Size Distribution

The particle size distribution in a representative EAF dust sample is shown graphically in Figure 1. Based on the obtained results of the granulometric analysis, it was determined that 70 wt.% of the sample consists of particles of size $<31\ \mu\text{m}$, which indicates the possibility of a difficult filtering process during the hydrometallurgical process of the EAF dust treatment.

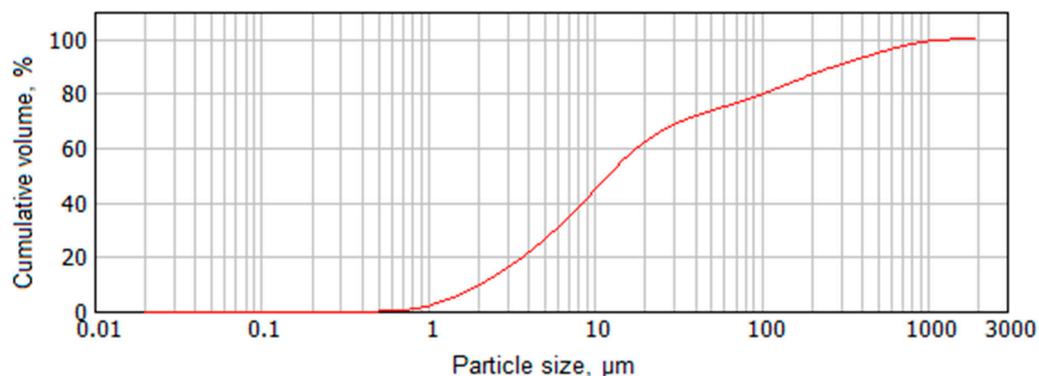


Figure 1. Particle size distribution in a representative EAF dust sample.

The obtained results of the particle size distribution of the EAF dust sample are in agreement with the literature [3,28,45].

3.2. Mineralogical Characterization

3.2.1. SEM-EDS Analysis

The morphological properties of a representative EAF dust sample were examined by SEM-EDS analysis. The investigations showed that the sample consists of agglomerates of irregular shapes, as well as spherical particles that differ in size (Figure 2). The obtained results are in agreement with the results of Santos et al. [31] and Khattab et al. [42].

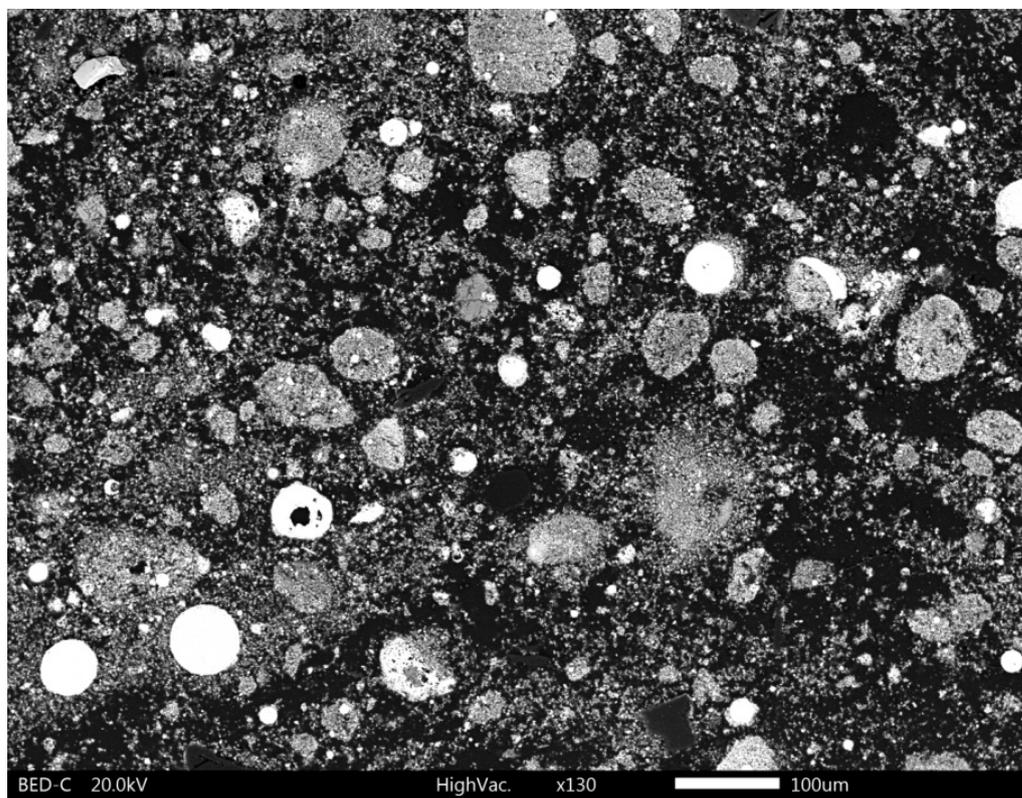
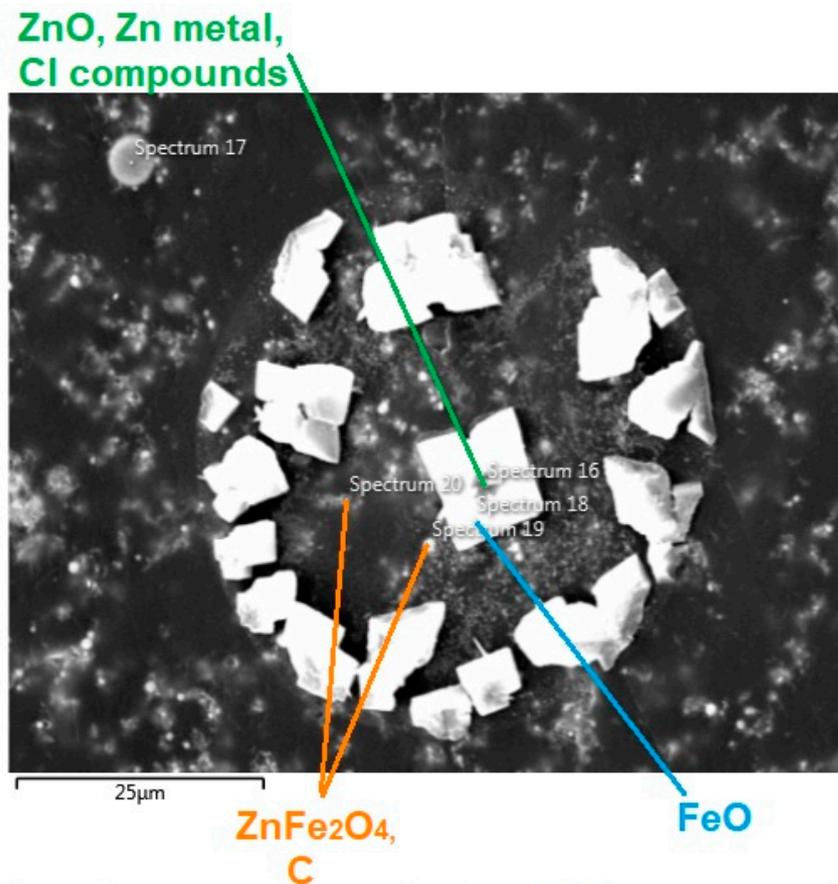


Figure 2. SEM-EDS analysis of a representative EAF dust sample.

Figure 3 presents an SEM-EDS magnified image of an EAF dust particle. Figure 3 shows a particle whose composition includes compounds of sodium, calcium, potassium, and chlorine. Also, iron oxides, zinc compounds, i.e., zinc ferrite and zinc oxide, as well as metallic zinc, were found. The sample also contains carbon that enters the dust following the consumption of the graphite electrodes used in the batch melting process in the electric arc furnace. In Figure 3, the spectrum related to the corresponding mineral phases is shown in the corresponding colors, and the results are given in wt.%. Because of the action of high temperature in the melting process of secondary raw materials in the electric arc furnace, during which EAF dust is formed, and then, its cooling in the output gas flow, EAF dust particles can burst, which is the case with the spherical particle presented in Figure 3.



	Spectrums (wt.%)				
	Spectrum 16	Spectrum 17	Spectrum 18	Spectrum 19	Spectrum 20
Fe	1.30	36.70	0.51	20.42	4.28
Zn	2.66	4.03		10.22	6.72
C	28.13	20.40	44.09	41.03	66.37
O	1.48	30.17	2.02	23.06	14.48
Na	20.91	1.08	21.18	2.88	4.17
Mg		0.59			
Al		0.57			
Si		2.49			
Cl	45.30		32.20	1.36	3.41
K					0.09
Ca		1.33		0.22	0.20
Mn	0.22	2.65		0.80	0.28
Total	100.00	100.00	100.00	100.00	100.00

Figure 3. SEM-EDS image of an EAF dust particle at 1500× magnification.

3.2.2. XRD Analysis

The X-ray diffraction analysis, the results of which are presented in Figure 4, identified several crystalline phases in a representative EAF dust sample. Three zinc-based phases were identified as follows: zincite (ZnO), franklinite (ZnFe_2O_4) and simonkolleite ($\text{Zn}_5(\text{OH})_8\text{Cl}_2 \cdot \text{H}_2\text{O}$), and magnetite (Fe_3O_4), as the iron phase. Magnetite and zincite are the phases that are more abundant in the sample (33% and 36%, respectively), and Ruiz et al. [19], Pickles [1], Wu et al. [5], Miki et al. [7], Silva et al. [32], and Halli et al. [38] also came to this conclusion. There is less franklinite (20%) than magnetite and zincite in the analyzed sample, while simonkolleite is the least abundant (11%).

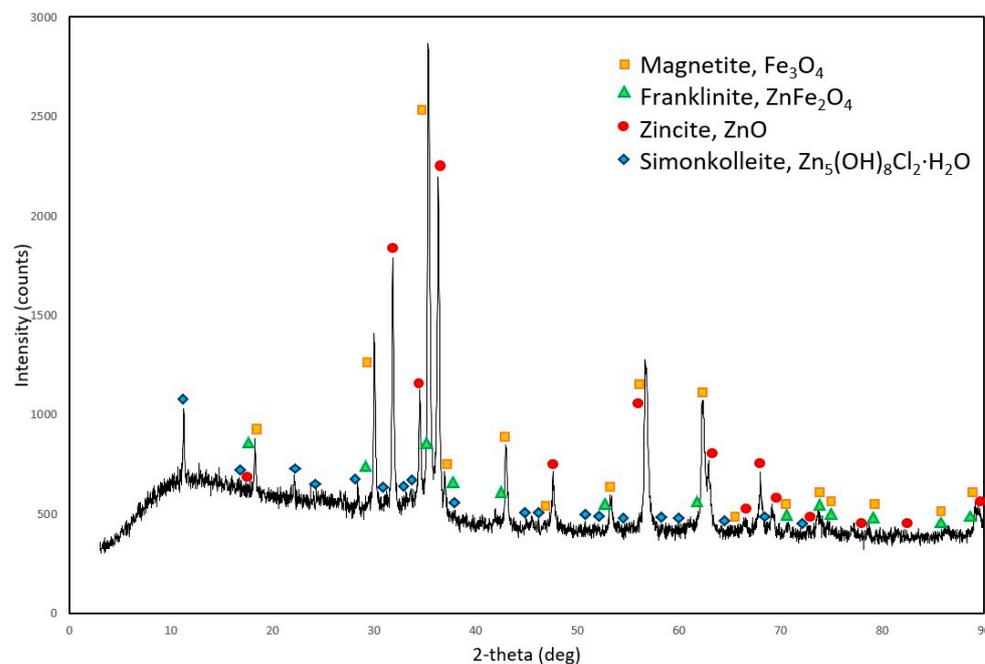


Figure 4. Diffractogram of a representative EAF dust sample.

Zinc is found in higher concentrations in EAF dust most often because of its widespread use to protect steel from corrosion or originated from scrap brass. The presence of zinc bound in the form of simonkolleite can be explained by the presence of protective paints on waste materials used as raw materials for steel production, or it can be present as a corrosion product. Because of the complex chemical composition of the EAF dust (Table 2), the existence of other, less common crystalline phases in the sample is possible. However, they are not clearly identified because they are present in amounts less than 1 wt.% and cannot be detected by the XRD analysis.

3.3. Pretreatment of the EAF Dust

Based on the results obtained by experimental investigations of the influence of certain parameters on the leaching efficiency of water-soluble components from the EAF dust, it can be concluded that the leaching efficiency of water-soluble components can be classified into three groups. The leaching efficiencies of Na, K, and Cl (first group) suggest that these elements have the highest leaching efficiencies.

In the case of Cd, Ca, and F (the second group), similar trends in leaching efficiencies were observed, suggesting that these three elements have similar characteristics. In the third group, Zn, Fe, Pb, Cu, and Ni can be classified as components that are relatively insoluble, which indicates that the compounds of these elements are found in forms that are difficult to dissolve in water. Montenegro et al. [51], Chen et al. [54], and Bruckard et al. [2], in their papers, also achieved good results in the removal of chlorine from EAF dust using the water leaching process.

3.3.1. Influence of Leaching Time on the Leaching Efficiency of the EAF Dust

By analyzing the obtained results, it was observed that the duration of the process has no great influence on the solubility of water-soluble components. The duration of the water leaching process of 30 min was chosen as the optimal parameter, given that the leaching efficiency of chlorine as the main water-soluble component (Cl content in the initial sample is the highest and is 2.5%) is 69.31%. The Na and K leaching efficiencies do not change significantly after 15 min of the leaching process and are 71.06%, 73.26%, and 72.70% for Na and 89.12%, 90.46%, and 90.92% for K, respectively. The leaching efficiency of Cd, Ca, and F ranges from 10 to 22%, respectively, with Cd 10.23–11.21%, Ca 11.06–15.32%, and F 16.81–22.15% (Figure 5). The leaching efficiency of other elements such as Zn, Fe, Pb, Cu, and Ni are negligible (<1%) and therefore not shown graphically.

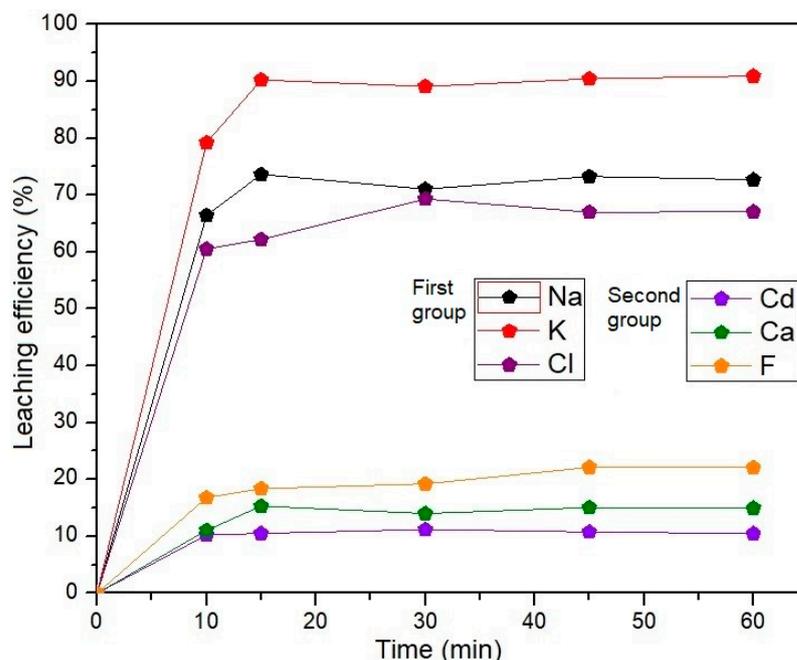


Figure 5. Influence of leaching time on the leaching efficiency of the EAF dust.

Chen et al. [54], in their work, investigated the influence of the leaching time from 5 to 60 min with an S:L ratio of 1:10 on the leaching efficiency of Cl. A 74% reduction in chloride content was achieved in 40 min.

3.3.2. Influence of the Solid and Liquid Phase Ratio on the Leaching Efficiency of the EAF Dust

Investigations of the influence of the solid and liquid phase ratio on the leaching efficiencies of water-soluble components of the EAF dust showed that with an increase in the S:L ratio above 1:5, a higher leaching efficiency of Na, K, and Cl is achieved (Figure 6). The S:L ratio from 1:10 to 1:20 has no major influence on the chlorine leaching efficiency, which ranges from 83 to 84%, sodium from 74 to 77%, or potassium from 92 to 97%, while with an increase in the S:L ratio at 1:25, there is a slight decrease in the leaching efficiencies of potassium to 94% and chlorine to 80% with sodium remaining at 77%. It can be observed in Figure 6 that the leaching efficiencies of Cd, Ca, and F range from 10 to 20%, respectively, with Cd 10.23–19.51%, Ca 14.02–17.11%, and F 19.22–21.24%. The leaching efficiencies of the other elements (Zn, Fe, Pb, Cu, and Ni) are not shown graphically because their values were very low. Based on the above, the solid and liquid phase ratio of 1:10 was determined as the optimal parameter for further investigations.

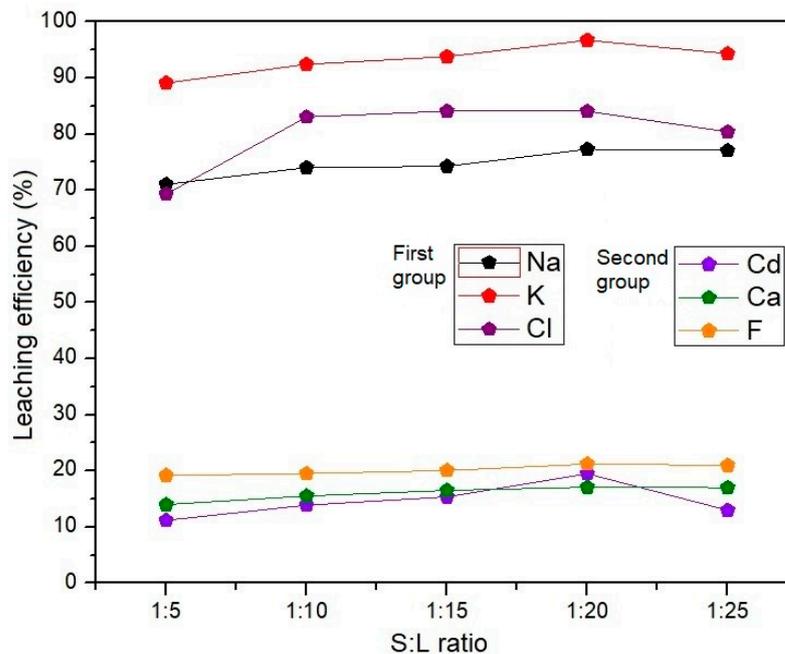


Figure 6. Influence of the solid and liquid phase ratio on the leaching efficiency of the EAF dust.

3.3.3. Influence of Temperature on the Leaching Efficiency of the EAF Dust

The results of the investigation showed that the leaching efficiency depends on the temperature in the process of the pretreatment of the EAF dust. With increasing temperature, the leaching efficiency slightly decreases in the case of the results obtained for sodium, chlorine, and potassium, where the leaching efficiency decreases from 74% to 68% for Na, from 83% to 81% for Cl, and from 92% to 83% for K (Figure 7). Based on the presented results, it can be concluded that with an increase in the temperature of the process, from ambient to 30 °C, the fluorine leaching efficiency increases from 19% to 50%, and with a further increase in temperature, its leaching efficiency decreases up to 42%. As for calcium, its leaching efficiency of about 15% remains constant for all investigated temperature values. The cadmium leaching efficiency decreases significantly with increasing temperature. Elements such as Zn, Fe, Pb, Cu, and Ni are not significantly affected by temperature. As it is known, the solubility of solid substances depends on their nature, the nature of the solvent, and the temperature. For most solids, solubility increases with increasing temperature, some substances change their solubility slightly, while the solubility of certain solids decreases with increasing temperature. For a detailed analysis and application of the stability/solubility diagram, it is of particular importance to know the chemical forms and species in which the water-soluble components, which, in our work, are the EAF dust components, are found. The leaching efficiency, i.e., dissolution in water, of monitored elements depends on the form in which they can be found in EAF dust [8,23,24,28,40,41]. For example, lead, iron, zinc, and copper in sulfide and sulfate forms are insoluble or slightly soluble in water, sodium and potassium in chloride forms are soluble in water, but chlorine in the form of a compound with zinc ($Zn_5(OH)_8Cl_2 \cdot H_2O$) is not soluble in water. The results of our research (Figure 7) indicated that the ionic species present in the studied EAF dust sample belong to species that dissolve better in water at lower temperature values. Based on the obtained results of the investigation the influence of process temperature on the leaching efficiency of water-soluble components from the EAF dust, ambient temperature, more precisely, a temperature of 20 °C, was chosen as the optimal parameter.

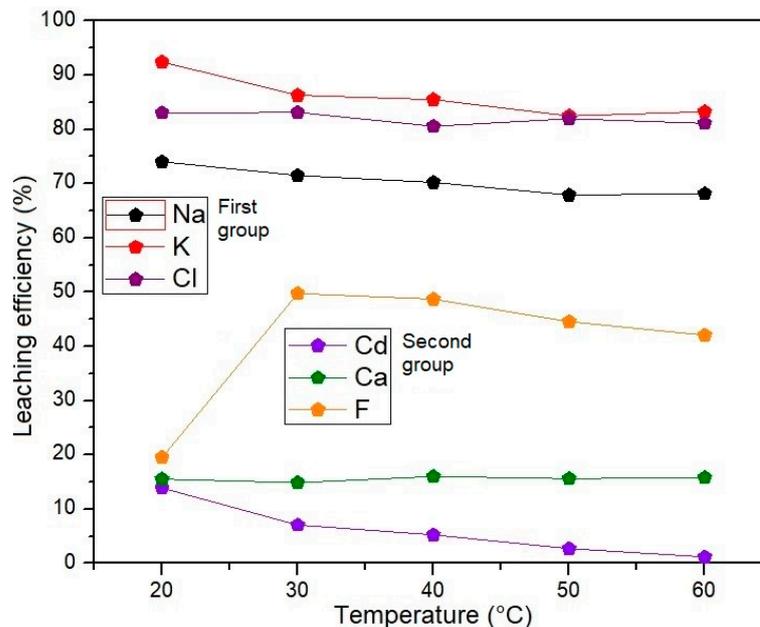


Figure 7. Influence of temperature on the leaching efficiency of the EAF dust.

In experimental investigations of chlorine extraction, Bruckard et al. [2] achieved 99% extraction of chlorides at ambient temperature at a time of 60 min.

Investigating the parameters of the selective leaching process of the EAF dust in order to remove water-soluble components, it can be concluded that the optimal parameters for the pretreatment of the EAF dust are as follows: time—30 min, solid and liquid phase ratio—1:10, temperature—ambient, and stirring speed—500 rpm.

It can be concluded that the leaching of the EAF dust with water represents a convenience for the further process of acid leaching. By applying the pretreatment of the EAF dust, a reduction in the mass of the solid residue obtained after leaching with distilled water is achieved by about 6% compared with the initial mass of the EAF dust sample.

3.3.4. XRD Analysis of the Solid Residue Obtained after the Pretreatment

The solid residue obtained after the pretreatment of the EAF dust, under optimal parameters, was chemically analyzed, and its XRD analysis was performed. The diffractogram of the obtained solid residue is presented in Figure 8.

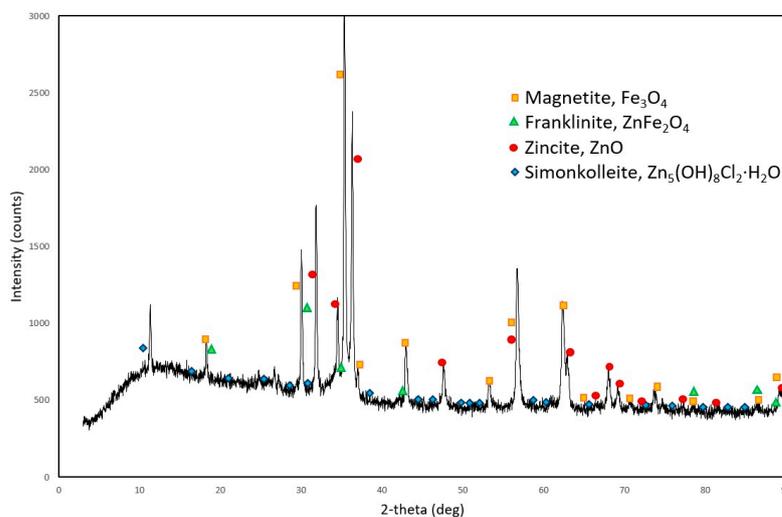


Figure 8. Diffractogram of the solid residue obtained after the pretreatment of the EAF dust.

By comparing the XRD analysis of the solid residue obtained after the pretreatment, under optimal leaching parameters (shown in Figure 8), and the XRD analysis of a representative initial EAF dust sample (Figure 4), it can be concluded that after the pretreatment of a representative sample of the EAF dust, there is no change in the content of the main mineralogical phases in the EAF dust sample, i.e., zincite, franklinite, magnetite, and simonkolleite. Comparing the XRD diffractograms in Figures 4 and 8, it can be seen that there are no changes in the crystal phases, i.e., destruction of crystal structures of magnetite, franklinite, zincite, and simonkolleite. Therefore, it can be said that the basic components of these crystal structures shown before and after the pretreatment investigated in this section (primarily Zn and Fe) will be able to be leached using an aggressive leaching reagent—sulfuric acid.

The results of the chemical analysis of the obtained solid residue confirmed high leaching efficiencies of Na—74%, K—92%, and Cl—83%, lower leaching efficiencies of Cd—14%, Ca—15%, and F—19%, and almost no leaching efficiencies of Zn—0.09%, Fe—0.0001%, Pb—0.0086%, Ni—0.006%, and Cu—0.003% after the applied pretreatment, which was expected. Also, the results of the chemical analyses indicate significant leaching efficiencies of toxic and chemically aggressive elements—Cd, Cl, and F.

3.4. Leaching of the Solid Residue Obtained after the Pretreatment

In agreement with the previously obtained results of low levels of leaching efficiencies of Zn, Fe, Pb, Cu, and Ni in the process of the pretreatment of the EAF dust, in this section, attention will be devoted to the leaching of these elements.

In order to increase the leaching efficiencies of monitored elements, primarily zinc, the process of acid leaching was investigated using concentrations of sulfuric acid from 0.1 M to 1.5 M for a period of 5 to 60 min at an S:L ratio of 1:4, ambient temperature, and a stirring speed of 500 rpm. Based on the results of the chemical analysis of the solid residues obtained after acid leaching, it can be concluded that with an increase in the concentration of sulfuric acid, a greater zinc leaching efficiency occurs, that is, a smaller percentage of zinc remains in the solid residue, as shown in Figure 9, which is in agreement with the investigations by Kukurugya et al. [6] and Shawabkeh et al. [56]. It can also be seen in Figure 9 that at all tested concentrations of sulfuric acid, the zinc leaching efficiency increases with time and that the most zinc is leached in the first 15–20 min, and after that, the leaching efficiency almost does not change. With increasing acid concentration, the zinc leaching efficiency increases from 5% to 65%.

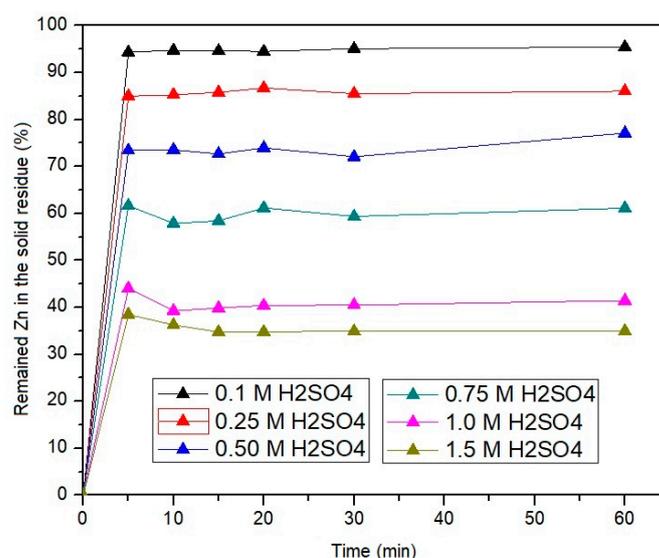


Figure 9. The percentage of the remaining Zn in the solid residue as a function of time and H₂SO₄ concentration.

The leaching efficiencies of the other monitored elements after the acid leaching process with a concentration of H_2SO_4 of 1.5 M also increase up to a time of 15–20 min and then stagnate and slightly decrease with further leaching time, which can be seen in Figure 10. The highest leaching efficiencies for Cd, Na, K, and Cu in this process are about 79%, 100%, 74%, and 60%, respectively. The leaching efficiencies of Fe, Pb, Ca, Ni, and F range from 0.2 to 22%, and they could be increased by correcting the pH value of the suspension during the leaching process, which will be the subject of our future research.

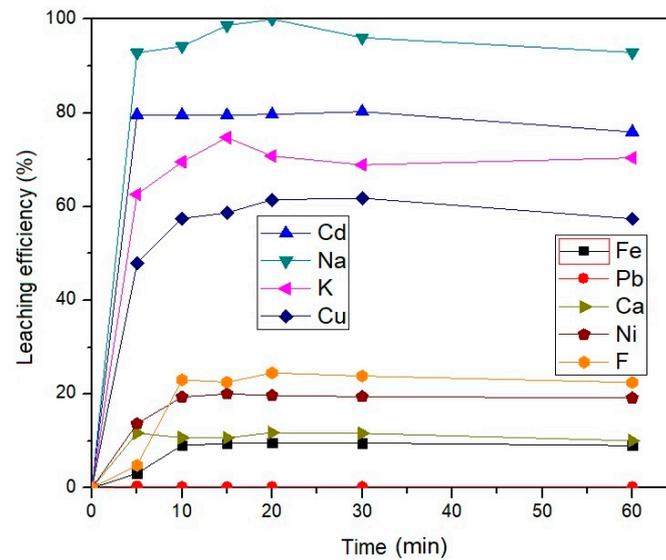


Figure 10. Leaching efficiency of monitored elements depending on time.

Figure 11 shows the influence of the S:L ratio on the leaching efficiency of Zn, Fe, Pb, Cd, Na, K, Ca, Cu, and Ni in 20 min at an H_2SO_4 concentration of 1.5 M. The results of the experiments showed that the leaching efficiencies of the monitored elements increase with an increase in the ratio of solid and liquid phases.

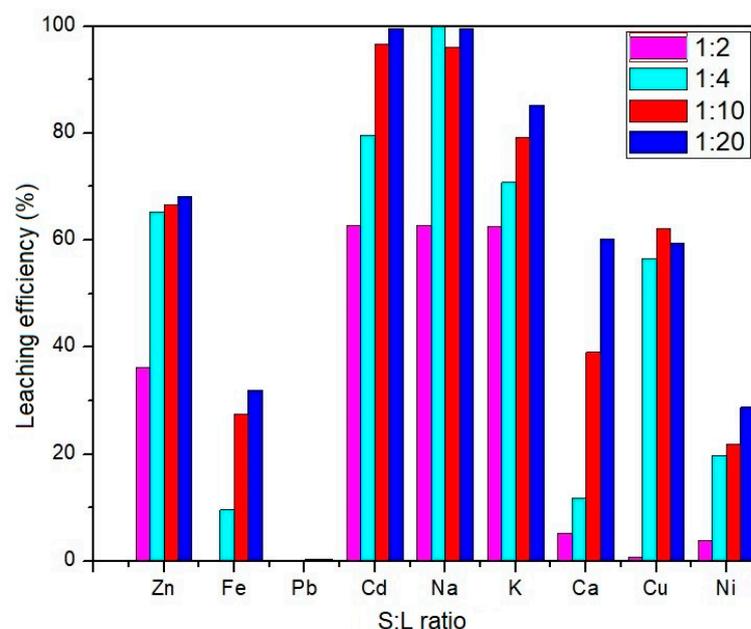


Figure 11. The leaching efficiencies of elements depending on the S:L ratio.

Regarding experimental investigations of the effect of temperature on the leaching efficiency of components from the EAF dust, the following temperatures were investigated:

ambient (~25), 40, 55, 70, and 85 °C. Similar conclusions were reached in works performed by Kukurugya et al. [6], Havlik et al. [14,39], Shawabkeh [56], and Hazaveh et al. [57], where they confirmed that the zinc leaching efficiency increases with increasing temperature in the acid leaching process.

In order to compare the results, an experiment was conducted in which a representative sample of the EAF dust was directly leached with 1.5 M sulfuric acid with an S:L ratio of 1:4, at ambient temperature, for 20 min. It was observed that by direct acid leaching of the EAF dust, the resulting suspension is very difficult to filter compared with the EAF dust sample, which involved the application of pretreatment before the acid leaching process. The achieved zinc leaching efficiency without pretreatment was 56%, which is 9% less than in the acid leaching process with the application of pretreatment. We can conclude that by applying pretreatment before the acid leaching process of the EAF dust, water-soluble components, i.e., compounds of potassium, sodium, calcium, and chlorine are selectively removed, thus leading to a reduction in the consumption of sulfuric acid in the acid leaching process. In this way, more sulfuric acid remains, which reacts with zinc from the EAF dust, thus achieving a higher level of zinc leaching efficiency in this process. Also, the obtained pregnant leaching solution then contains fewer contaminating components. The advantage is reflected in the fact that the future purification process of the pregnant leaching solution, i.e., the process of removing impurities from the solution, in order to obtain a commercial zinc-based product, will be less complicated and demanding.

4. Conclusions

After a detailed characterization of the EAF dust, which included physico-chemical and mineralogical characterization, as well as an investigation of the particle size distribution of a representative EAF dust sample originating from the Republic of Serbia, experimental investigations of the application of a simple and inexpensive pretreatment process for reducing the content of certain elements from the EAF dust were carried out. By investigating the water leaching process of the EAF dust, the following optimal leaching parameters were defined: process time—30 min, solid and liquid phase ratio S:L—1:10, temperature—ambient, and stirring speed—500 rpm. Under these leaching parameters, the reduction in the content of water-soluble components was as follows: Cl—83%, K—92%, Na—74%, Cd—14%, Ca—15%, and F—19%, while Zn, Fe, Pb, Cu, and Ni remained insoluble after the applied pretreatment. Because of the high content of zinc in the EAF dust (32.38%), it can also be used as a secondary raw material for the valorization of zinc. The advantage of the applied selective leaching of the EAF dust (pretreatment process) is reflected in the high selectivity and high leaching efficiency of water-soluble components, which, in the further process of the valorization of zinc and other useful components from EAF dust by acid leaching, will lead to a reduction in acid consumption. Given that EAF dust is globally characterized as hazardous solid industrial waste, the application of the pretreatment results in a reduction in the amount of hazardous waste by 6% from the initial amount, which is another advantage of this process that was further applied to investigate the acid leaching of the obtained solid residue. In the acid leaching experiments of the solid residue obtained after the applied pretreatment, investigations were carried out on the influence of the concentration of sulfuric acid as a leaching reagent, on the influence of the solid and liquid phase ratio, and on the influence of time and temperature on the leaching efficiency primarily of zinc but also of other useful components. The obtained results showed that the highest percentage of Zn is leached in 15–20 min and that the leaching efficiency increases with an increase in the solid and liquid phase ratio, at higher concentrations of sulfuric acid, as well as with an increase in temperature. Based on the compared results of the direct acid leaching experiments of a representative sample of the EAF dust, without pretreatment and with the application of pretreatment, it can be concluded that the zinc leaching efficiency increases by 9% with the application of pretreatment. It was also observed that by applying pretreatment before the acid leaching process, a suspension is obtained that is significantly better filtered compared with the suspension

obtained by the direct leaching of the EAF dust with sulfuric acid in the process of the further valorization of zinc and other useful components. In addition, it was observed that their leaching efficiencies increased. The possibility of achieving higher leaching efficiencies of useful components from EAF dust in the acid leaching process will be the subject of our further investigations.

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