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Synopsis

We present three technological scenarios for the recovery of valuable components from gangue, stored in the tailings dam at Kremikovtzi metallurgical plant in Bulgaria, into marketable iron-containing pellets. In the first approach the iron concentrate was recovered through a two-stage flotation process, desliming, and magnetic separation. In the second proposed process, the iron concentrate was subjected to four sequential stages of magnetic separation coupled with selective magnetic flocculation. The third route entails the not very common practice of magnetizing roasting, followed by selective magnetic flocculation, desliming, and magnetic separation. The iron concentrate was pelletized in a laboratory-scale pelletizer. Each technology has been assessed with regard to the mass yield of iron concentrate, the iron recovery, and the iron, lead, and zinc content in order to identify the most effective route.

Keywords

tailings reprocessing, magnetizing roasting, pelletization.

Introduction

Tailings are the commercially worthless fractions of an ore. Usually, they are stored in the most cost-effective way possible to meet specific environmental regulations. Converting tailings, through reprocessing, into a valuable material represents a profitable field for technological research for mining companies and a viable solution to environmental issues.

Here we used three technological scenarios for the recovery of valuable components from gangue, stored in the tailings dam at Kremikovtzi ore dressing plant in Bulgaria, into marketable iron-containing pellets. The tailings storage facility of the company, which has been closed down, was used for disposal of waste material originating from various manufacturing processes, such as granulated blast-furnace slag, coal-fired power station ash and slag, steelmaking slag, and by-products from ferroalloy and fuel production.

Many approaches have been developed for recovering iron from tailings (Ajaka, 2009; Rao and Narasimhan, 1985; Sakthivel *et al.*, 2010; Li *et al.*, 2010). Usually they couple magnetic separation with selective flocculation. Another method for the beneficiation of the iron ore tailings is magnetizing roasting, which is an energy-intensive process and as such seldom used. However, it offers the advantage of reduction of paramagnetic haematite to ferromagnetic magnetite, which can be recovered by wet low-intensity magnetic separation (Da Corte, Bergmann, and Woollacott, 2019). As shown below, magnetizing roasting could be very helpful if the extracted iron concentrate will be subjected to pelletizing.

Another technology that is being developed for iron recovery from tailings is the production of electrolytic iron. This appears to be an attractive way to produce iron with lower associated CO_2 emissions (Maihatchi *et al.*, 2020). It has the advantage of producing a product of extreme purity, and further of utilizing ores which are not suitable for smelting. The high production cost is a disadvantage, however.

The direct reduction of iron ore to iron using as reductant gas instead of coal is another potential process. The reduction of iron oxides using hydrogen produced via renewable energy has been intensively investigated as a future alternative to the commonly used carbon reducing agents. (Spreitzer and Schenk, 2019). A method for microbial recovery of iron from solid industrial waste has been patented (Hoffmann, Arnold, and Stephanopoulos, 1989). However, none of the 'green' technologies mentioned above can yet match the advantages of the popular blast-furnace technology for recovering iron from mine tailings.

Many researchers have investigated the utilization of iron tailings for production of building materials such as bricks, ceramic tiles, cement, or concrete, microcrystalline glasses, and underground backfill materials (Kuranchie, 2015; Sun, Ren, and Lu, 2010; Das, Kumar, and Ramachandrarao, 2000; Das et al., 2012; Tang et al., 2019, Li and Wang, 2008; Li et al., 2010). A little information about using iron ore tailings for pellet making can be found in the literature. Güngör, Atalay, and Sivrikaya (2011) describe the extraction of magnetite concentrate from tailings through low-intensity magnetic separation, which meets the specifications for pellet making, but the agglomeration process itself has been not carried out. Roy, Das, and Mohanty (2007) present a technology for beneficiating low-grade iron ore slime, sampled in Chitradurga, India, for producing pelletgrade concentrate, together with a detailed investigation of the mineralogical and microstructural characteristics of the iron ore bulk sample and the flotation concentrate. However, pelletizing was not included in the work. In this paper, we aim to present a complete study on processing mine tailings to marketable iron-containing pellets. Three different technological scenarios have been investigated so that the most suitable route can be identified.

Materials and methods

Raw material from the upper 2.5 m horizon of the tailings was sampled, dried at 105°C, classified by hand sieving at 2 mm aperture, and analysed for chemical (Tables I) and mineralogical composition (Figure 1). The chemical composition of the sample, the intermediate products, and the final iron concentrate from each of the three process flow sheets were characterized using classical chemical analytical methods.

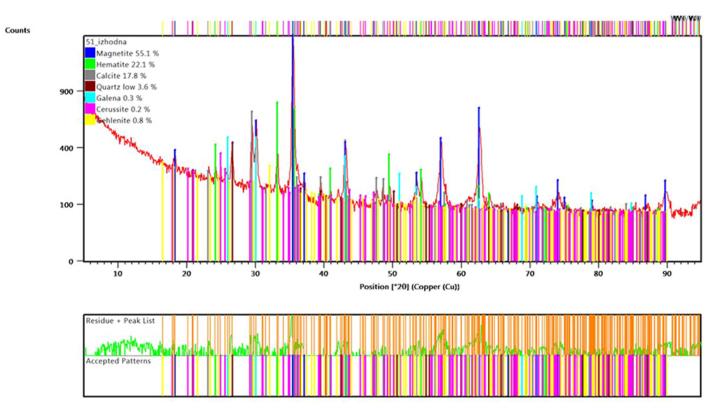
Mineralogical characterization

The main mineral constituents of the iron ore tailings, were determined using XRD with a Cu radiation source. The analytical parameters were step size 0.05°, scan time step size 43.6 seconds, and angle interval 5–95° 20. As shown in Figure 1, the main minerals in the tailings were magnetite, haematite, and calcite.

Recovery of iron concentrates through two stages of flotation, desliming, and magnetic separation

The first flow sheet applied for processing the tailings comprised sample grinding, flotation of lead, zinc, silver, and organic components, a second (reverse) process of flotation, desliming, and separation of the iron particles at 1800 G magnetic field intensity into an iron concentrate. The sample was milled for 12 minutes to less than 40 µm. The contents of iron, lead, zinc, silver, and carbon in the sample after each operation are shown in Figure 2.

Table I Chemical composition of the head sample				
(a)		(b)		
Component	Amount	Component	Amount	
Iron	45.72 (wt.%)	Total organic carbon	5.98 (wt.%)	
Lead	1.75 (wt.%)	Total inorganic carbon	1.39 (wt.%)	
Silver	53.00 (g/t)	Petroleum products	56.00 (mg/kg)	
Zinc	0.84 (wt.%)			
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Figure 1-XRD pattern of the iron ore tailings

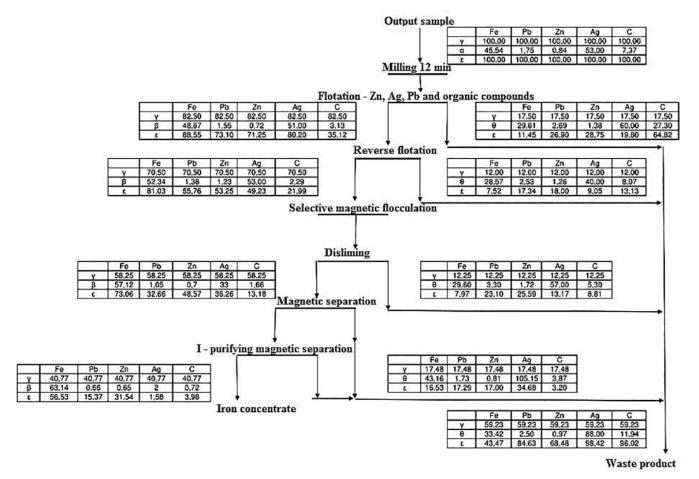


Figure 2—Recovery of iron concentrate through two stages of flotation, coupled with desliming and magnetic separation. (a) Concentration of the corresponding component in the output sample in weight %, (%) mass yield of the corresponding product in weight %, (%) concentration of the valuable component in the concentrate in weight %, (%) concentration of the corresponding component in the waste product in weight %, (%) recovery of the corresponding component in the extracted concentrate in weight %

Recovery of iron concentrate through four stages of magnetic separation, coupled with selective magnetic flocculation in impulse magnetic field

The second technology used for processing the tailings is presented on Figure 3. The flow diagram includes four stages of magnetic separation with selective magnetic flocculation after the second separation stage.

Recovery of iron concentrates after beneficiation by magnetizing roasting

Magnetizing roasting was applied to beneficiate the iron ore tailings, followed by selective magnetic flocculation, desliming, and magnetic separation. The raw material was initially milled for 12 minutes in a ball mill and then subjected to a magnetizing roast (Figure 4). Figure 5 presents the flow sheet.

Process conditions

Flotation was carried out at a pulp solids concentration of 33% and pulp pH in the range 8.5–9.0, adjusted using water glass. Potassium isobutyl xanthate was used as the collector for the first flotation stage, and potassium oleate for the second (reverse) flotation.

Magnetic separation was performed at 0.18 T field strength. The magnetizing roasting was carried out for 60 minutes at temperature of 700° C in the presence of coals as a reducing agent.

Pelletizing experiments

A lot of experimental work has been done to establish the optimal conditions under which iron pellets with an iron content over 60.00%, a lead content less than 0.01%, and zinc content of 0.09% or less can be produced. Initially, the pellets were produced manually by varying the size of the pellets, the binder concentration (bentonite), the firing temperature, the concentration and type of the coal used as a reducing agent, and the roasting time. A laboratory-scale pelletizer disc was used to form the pellets under the following operating parameters: iron concentrate moisture 12.5 wt.%, bentonite concentration 2.5 wt.%, pellets size 9–16 mm, pelletizer disc rotation speed 15 r/min, pelletizer disc inclination 60°, coal content 25 wt.%. After firing at 1100°C in a high-temperature furnace for 40 minutes, the pellets were cooled to 700°C in a reducing environment.

Results

The iron concentrate obtained *via* the first flow sheet (two stages of flotation, desliming, and magnetic separation) contained 63.14 wt.% iron, 0.66 wt.% lead, 0.65 wt.% zinc, 2 g/t silver, 2.15 wt.% silicon dioxide, 2.40 wt.% lime, 1.6 wt.%, manganese, 0.74 wt.% magnesium oxide, 0.046 wt.% copper, 0.50 wt.% alumina (aluminum trioxide), and 0.72 wt.% total carbon. The mass yield of the concentrate was 40.77 wt.%. The iron content meets the requirements for producing pellets with customized

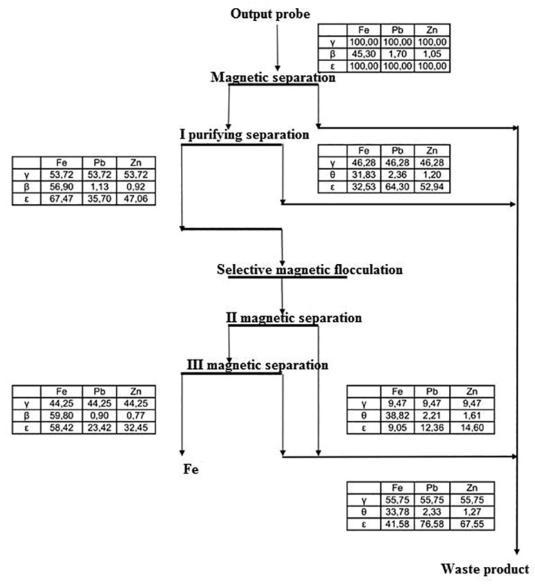


Figure 3-Extraction of iron concentrate after four stages of magnetic separation coupled with selective magnetic flocculation

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properties. We tried to extract the non-ferrous bearing minerals as a separate marketable product through further flotation experiments. Unfortunately, the results from the laboratory analysis have shown that a very small proportion of the silver-, lead-, and zinc-bearing minerals is in a form that can be successfully floated (Table II).

The mass yield of the iron concentrate after applying the second proposed approach was slightly higher than with the first one – 44.25 wt.% vs. 40.77 wt.% – but it contained less iron (59.80 wt.%), more lead (0.90 wt.%), and more zinc (0.77 wt.%). The iron recovery was 58.24 wt.%. The results show that magnetic separation coupled with selective magnetic flocculation yields an iron concentrate of good quality suitable for pelletizing. Furthermore, this technology is much easier to apply in industry than that presented in Figure 1.

Processing the tailings in accordance with the third proposed technological scenario resulted in a mass yield to the concentrate of 74.42 wt.% and an iron recovery of 88.00 wt.%. The concentrate contained 59.98 wt.% iron, 1.50 wt.% Pb, and 1.19 wt.% Zn. After desliming the waste product contained 16.11 wt.% iron, which is significantly lower than that obtained by the first approach (29.60 wt.%). Compared to the second scenario (γ = 44.25 wt.%, β = 59.80 wt.%, ϵ = 58.42 wt.%) the yield of the iron concentrate was 28.15 wt.% higher and the iron recovery to the concentrate 29.58 wt.% higher. The lead and zinc contents were almost double those obtained via the other two technologies. Since during pelletizing the concentrate will be subjected to firing at 1000°C in the presence of a reducing agent, in our case the carbon in the concentrate, the higher contents of lead and the zinc will be reduced.

The pellets produced from the iron concentrate obtained by magnetizing roasting (Figure 4) had an iron content of 68.0 wt.%, 0.007 wt.% lead, and 0.09 wt.% zinc. The chemical composition of the pellets is given in Table III.

Conclusion

The tailings stored at the Kremikovtzi ore dressing plant in Bulgaria can be reprocessed by one of three proposed flow sheets so as to produce metallized pellets of good quality. The first technology, which couples two-stage flotation with desliming,

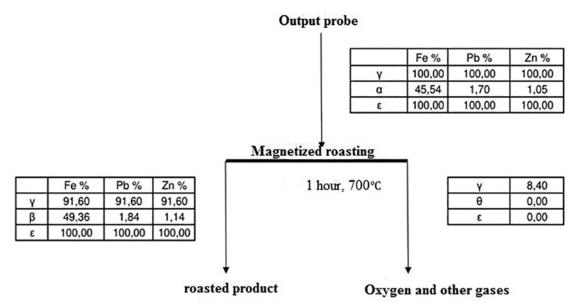


Figure 4—Beneficiation by magnetizing roasting

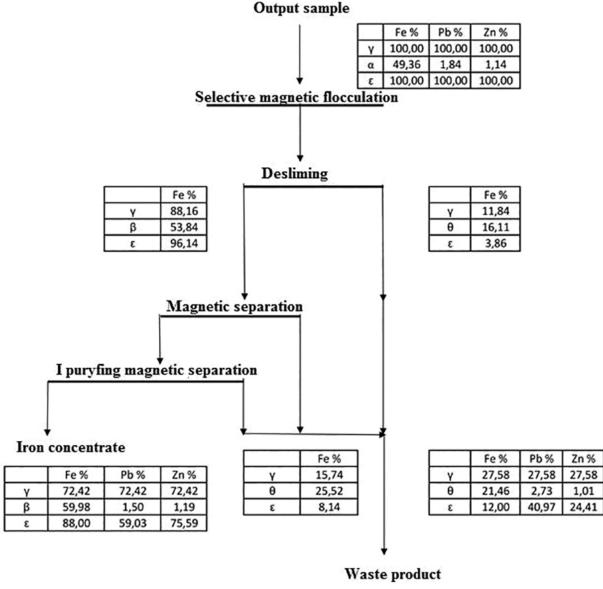


Figure 5—Recovery of iron concentrate after beneficiation by magnetizing roasting

Table II

Chemical composition of the waste product after additional flotation experiments

Element	Amount
Ag	40 wt.%
C	8.07 wt.%
Pb	2.53 wt.%
Zn	1.26 wt%

Table III

Chemical composition of the pellets produced from iron concentrate obtained by magnetizing roasting

Component	Amount
Ag	25 g/t
C	0.19 wt.%
Cu	0.04 wt.%
Fe	68.00 wt.%
MgO	1.53 wt.%
Mn	2.02 wt.%
CaO	7.92 wt.%
Pb	0.007 wt.%
SiO ₂	8.10 wt.%
Zn	0.09 wt.%

selective magnetic flocculation, and magnetic separation, offers the lowest iron recovery, and would be more complicated to apply on an industrial scale in comparison to the other two routes. Moreover, only the iron with strong magnetic properties can be extracted. The second technology, which couples four stages of magnetic separation with selective magnetic flocculation, can be used for production of pellet-grade concentrate, but it has the same disadvantage as the first route, namely only the iron with strong magnetic properties can be extracted. The third scenario offers a combination of magnetizing roasting followed by magnetic separation and desliming. This technology enables the recovery of iron with weak magnetic properties, but is more complex, more expensive, and would be more difficult to implement. The chemical composition analysis of the fired pellets shows that the contents of arsenic, copper, sulphur, phosphorus, chlorine, and fluorine are well below the standards. After further processing, the dust collected in the bag filters during pelletizing, which is rich in zinc and lead, can also used as a feed in nonferrous metals manufacturing. As further experiments, we plan to subject the product beneficiated through magnetizing roasting, to dry magnetic separation, and to fire the pellets in a rotary tube furnace.

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