



RESEARCH ARTICLE

RECOVERY OF BORON WASTES WITH INORGANIC ACID

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ABSTRACT

Approximately 73-74% of boron mineral reserves all over the world are located in the Western Anatolian region of Turkey, in the provinces of Eskişehir, Kütahya, Balıkesir, and Bursa. The most intense minerals extracted from these fields are colemanite, ulexite, and tincal minerals, respectively. The general principle in the recovery of these minerals is to extract the raw ore and obtain it by size reduction processes of concentrates in high grade. In addition to concentrates, boric acid ( $H_3BO_3$ ) production is also possible in Balıkesir and Kütahya. The production of this acid in question includes a series of processes from dissolution with sulfuric acid at high temperatures to crystallization. In this study, except for the aforementioned high concentrations, boron recovery from clay waste, which is high in boron content and is collected from plant waste ponds, is investigated. For this purpose, ulexite-colemanite-containing slime wastes of the Bigadiç (Balıkesir) Plant were dissolved in 7% solids at 60°C in different sulfuric acid concentrations (1-6%  $H_2SO_4$ ) and were obtained with 83-97%  $B_2O_3$  recovery. When the boron wastes are evaluated, issues such as the discovery and establishment of a new waste pond/dam will be solved, the valuable content in the wastes will be recovered, and the environmental problems of boron and other contents in clay will be eliminated. This article includes information about the characterization of the structure by considering the dissolution of boron and other compounds, as well as obtaining boric acid from inorganic acid and boron wastes.

**Keywords:** Boron waste, Colemanite, Ulexite, Inorganic acid, Sulfuric acid

1. INTRODUCTION

Boron (B), which is the first element of group 3A of the periodic table, is found in compounds with oxygen bonds due to oxygen affinity, although it is in the content of more than 150 minerals, with the properties indicated in Table 1. While the use of boron in Turkey dates back to ancient times, it is known that it was used in the production of tiles in Anatolia in the 1860s [1, 2].

**Table 1.** General properties of elemental boron [1, 2].

Properties	
Chemical symbol	B
Atomic number	5
Atomic weight	10.81-10.82
Melting point	2200°C
Boiling point	2250°C

The boron deposits located in Western Anatolia, the easternmost part of the Aegean Expansion Province, consist of two parts. The first part is the Izmir-Balıkesir Transfer Zone (IBTZ), which includes the Bigadiç, Sultançayır, and Kestelek basins, and the second part is the northern part of the Menderes Massif, where the Selendi and Emet basins are located. The Kırka boron deposit, located within the volcano-stratigraphic sequence, contains a completely different geological environment further east [3, 4].

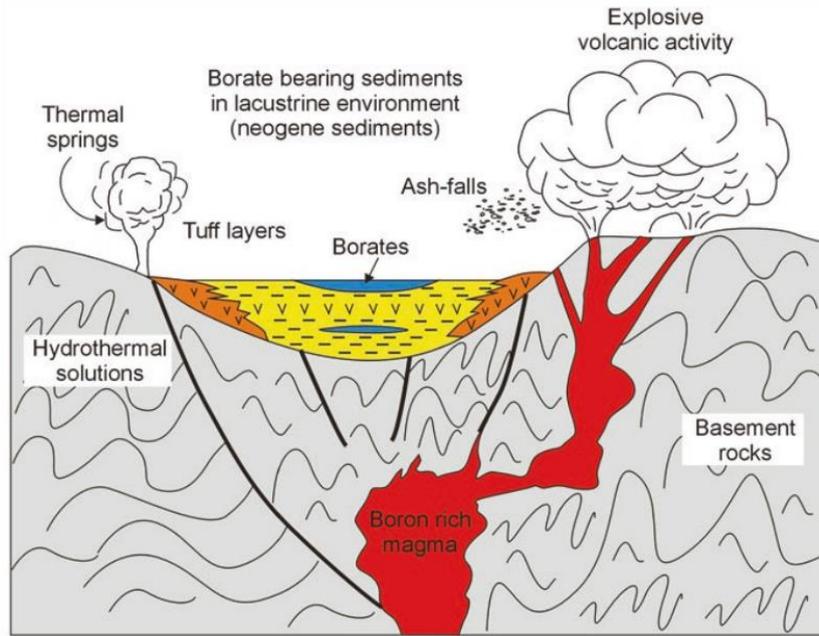
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Borate deposits consist of three main groups: skarn (containing silicate and iron oxide), magnesium oxide (marine evaporitic sediment), and sodium-calcium borate hydrates (playa lake) group. Five formation steps are required for playa-lake volcano-sedimentary boron deposits. These occur as follows [3, 5] (Figure 1):

- The creation of a Playa-lake media,
- The enrichment of boron in this lake, which contains from andesite to rhyolite, and the deposition of either hydrothermal solutions or ash-fall sediments within the basin along the graben faults.
- Thermal resources near volcanism
- Conditions of an arid or semi-arid climate
- Alkaline pH conditions of lake water (varying between 8.5-11)



**Figure 1.** According to the generalized Playa lake sedimentation model, the formation of boron deposits in the Neogene basins in Western Anatolia [5]

Volcanics range from acid to base, where volcanic activities are very common, and pyroclastic rocks with sediments have been observed around the playa-lakes that form the borate deposits in Turkey [6].

Compared to the world, Western Anatolia has an important place with its reserves of around 73-74%. Colemanite (Kütahya-Emet, Balıkesir Bigadiç, Bursa Kestelek), ulexite (Balıkesir-Bigadiç), and tincal (Eskişehir-Kırka) minerals are the most abundant boron minerals in Turkey [6-10]. The densities of colemanite and ulexite minerals are 2.4 and 1.98 g/cm<sup>3</sup>, respectively [11]. The country rock minerals of boron ores are composed of carbonates and silicates, and the densities of these minerals are 2.6-2.7 g/cm<sup>3</sup> [12]. The formulas and % B<sub>2</sub>O<sub>3</sub> amounts of the commercially important boron minerals found extensively in Turkey are given in Table 2 [8, 10, 13, 14].

**Table 2.** Properties of some commercially important boron minerals [8, 13].

Mineral name	Formula	B <sub>2</sub> O <sub>3</sub> , %
Colemanite	Ca <sub>2</sub> B <sub>6</sub> O <sub>11</sub> .5H <sub>2</sub> O	50.8
Ulexite	NaCaB <sub>5</sub> O <sub>9</sub> .8H <sub>2</sub> O	43.0
Tincal/Borax	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> .10H <sub>2</sub> O	36.5
Pandermite	Ca <sub>4</sub> B <sub>10</sub> O <sub>19</sub> .7H <sub>2</sub> O	49.8
Propertite	NaCaB <sub>5</sub> O <sub>9</sub> .5H <sub>2</sub> O	49.6

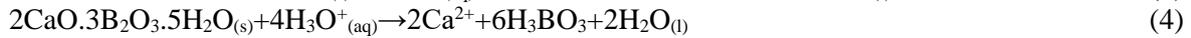
In general, the recovery principle of crude boron is based on physical recovery methods including size comminution and classification [15]. Some of the common names of crude boron products are known as tincal concentrate, ulexite concentrate, colemanite concentrate, ground colemanite. In addition, refined products are also known by names such as refined/calcined borax pentahydrate, refined/calcined borax decahydrate, boric acid, anhydrous boric acid, synthetic/calcined/refined colemanite, calcined tincal, calcined ulexite [7, 16]. Concentrated boron products are used in many industries such as glass-glass fiber, metallurgy-materials, agriculture, textile, cleaning, ceramics and medicine. In addition to these areas, refined boron products can also be used in many industries such as military, electronics, photography, cosmetics and aerospace [11].

Boric acid, which is one of the refined products, is obtained especially from the boric acid plants established in Emet (Kütahya) and Bandırma (Balıkesir) regions, especially with colemanite [17, 18]. Obtaining this acid from concentrated ores consists of a series of processes such as dissolving in sulfuric acid solutions at high temperatures (80-90°C), separation from impurities, filtration, cooling, evaporation, crystallization, and washing, and drying of crystals [19-21].

The two-stage separation of H<sub>2</sub>SO<sub>4</sub>, which is used for boric acid production, by dissolving into its ions in water is given below [20-24] (1-2):



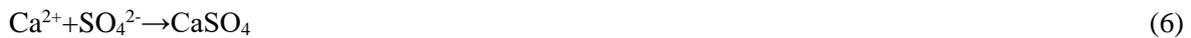
The equilibrium constant (2) for the second reaction is  $K_a = 0.012$ . It is suggested that the following reactions will occur when the minerals ulexite [22, 23] (3) and colemanite [24] (4) are added to this solution:



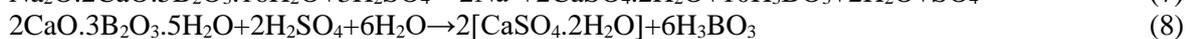
When Ca<sup>2+</sup> concentrations reach a limiting value determined by the product of solubility (K<sub>sp</sub>) (5);



The ion [SO<sub>4</sub><sup>2-</sup>] obtained by the 2nd reaction of sulfuric acid creates the following reaction (6).



Thus, a solid precipitate is obtained [21-24]. Finally, the dissolution reactions of sulfuric acid are as follows for boron minerals ulexite [22]; [23] (7) and colemanite minerals [8, 19, 20, 24-28] (8):



In Turkey, after the recovery of crude boron products, a large amount of high-grade  $B_2O_3$ -containing waste is collected in waste ponds/dams. While the amount of waste from these areas in 2001 was around 2 million tons [2], it is estimated that the 4 waste ponds in the Balıkesir/Bigadiç region recently exceeded this amount [20].

Recently, many different methods have been applied in the evaluation of boron wastes. Some of these evaluations can be made in the form of the recovery of boron contents in the waste, the use of all the contents, or other (except boron) contents in the waste in different sectors such as ceramics and building [2, 7, 29-31]. One of the most appropriate evaluations is the use of the remaining structures in appropriate industries after the recovery of boron from wastes. Therefore, this strategic mineral can be brought into the economy, and the remaining products such as clay can be used in other industries, as well as the environmental impact of waste can be eliminated [32].

In this context, in this study, ulexite-colemanite-containing wastes in the waste ponds of Etimaden Plant Balıkesir/Bigadiç Boron Operation Directorate between 1980-1995 have been evaluated. For this purpose, it was investigated whether the boron contents of these fine-grained processed wastes can be recovered from inorganic acids by dissolving them with sulfuric acid. Therefore, a preliminary evaluation will be carried out in order to reveal all the processes in the recyclability of this and other boron wastes. Thus, the study aims to make predictions about the industrial-scale recycling process.

## **2. MATERIALS AND METHODS**

### **2.1. Characterization Studies**

In the study, representative samples taken from Etimaden Bigadiç Boron Mining Directorate waste ponds, leaching process liquids and solid samples of waste were analyzed in Bigadiç Research laboratories.

In this context, chemical analyzes (using the melting method) for feed (main waste) sample and leaching wastes were carried out with the Rigaku ZSX Primus XRF (X-ray fluorescence) device.

For particle size distribution analysis, analyzes were carried out using the laser diffraction method with the Malvern Mastersizer 3000 device. Analyzes were obtained in an aqueous environment by taking the average of 3 samples.

Additionally,  $B_2O_3$  analyzes were repeated using the titration method. LOI (loss on ignition) analyzes were carried out using the gravimetric method.

Experiments were carried out in the Sample Preparation Laboratory at the Central Research Laboratory of Niğde Ömer Halisdemir University (NOHU). Dissolution of the samples was done in a fume hood with a heated magnetic stirrer.

### **2.2. Reagents**

The acid used during the experiments is Tekkim (Turkey) 95-98%  $H_2SO_4$  (sulfuric acid). The acid solutions diluted with distilled water between 1-6.1% were adjusted to a 7% solids ratio, and then processes of dissolution were carried out.

### 2.3. Samples

For the experiments, the samples obtained from four waste ponds in Bigadiç (Balıkesir) depending on their quantity (around 60-65 kg in total) were dried. The dried sample (approximately 30 kg) was blended, divided in a way that it would be approximately 2 kg by coning and quartering method, and packaged. Experimental samples of about 100-120 g were obtained by re-dividing these packages.

The experiments were carried out under 60 minutes leaching time, 60°C leaching temperature, 7% solids ratio, 1500 rpm stirring speed, and approximately 1-2-3-4-6 H<sub>2</sub>SO<sub>4</sub> concentration conditions. At the end of the experiment, the solution was filtered, and the solid part was washed several times and dried. Solid (secondary waste) and liquid samples were sent to the Etimaden Bigadiç plant analysis laboratory, and analyzed (Figure 2).

The feed sample (FS) in which the test was performed is the main waste sample with 11.43% B<sub>2</sub>O<sub>3</sub> content. After the experiments, secondary waste samples were designated with the code "WS" and these samples were numbered 1-2-3-4-5 according to their acid concentrations (1-2-3-4-6%).



**Figure 2.** Leaching process (left image), filter and leaching liquid (medium image), and the resulting filter cake (right image)

The reason why the 7% solids ratio was chosen for the experiments is that mixing cannot be achieved at higher solid ratios due to the clay structure of the sample. The experiments were carried out in 2000 ml beakers. In experiments to be carried out with solid ratios lower than this solid ratio, the solid (waste) sample required for analysis cannot be obtained. In addition, it was thought that lower solid ratios could cause difficulties in the analysis and could harm the industrial-scale prediction of the study.

## 3. RESULTS & DISCUSSION

### 3.1. Particle Size Distribution

The particle size distribution of the test sample was analyzed by laser diffraction method since it was a slime-sized sample taken from the waste pond. Analysis results were determined as  $d_{90}= 81.9 \mu\text{m}$  and  $d_{50}= 16.5 \mu\text{m}$  in Figure 3.

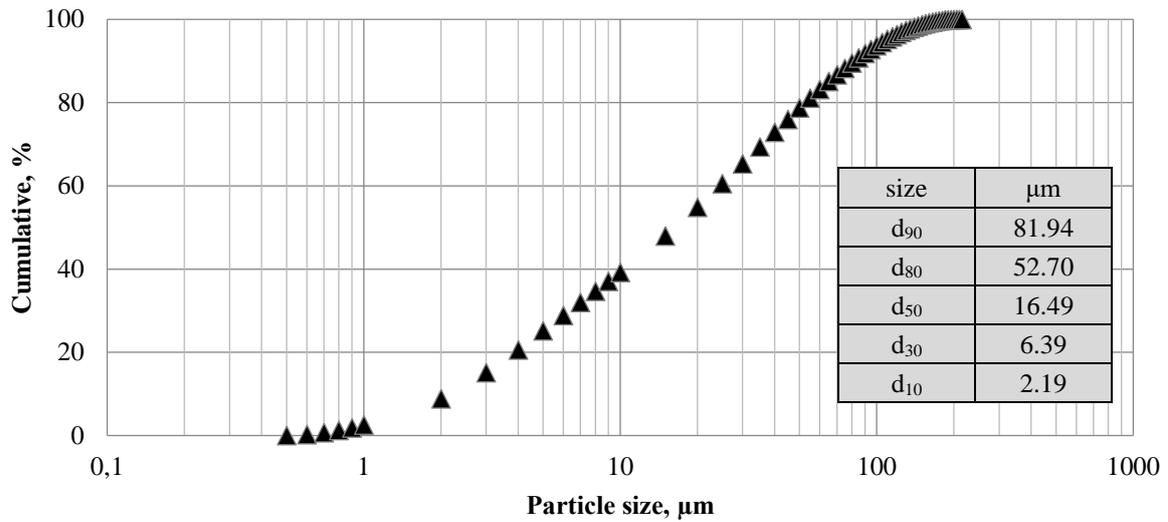


Figure 3. Particle size distribution of waste sample

### 3.2. Dissolution Results

Chemical analysis results of feed sample and leaching process waste samples are given in Figure 4-5 and dissolution results are given in Figure 6-7. Figure 2 shows the analysis results of Na<sub>2</sub>O, Fe<sub>2</sub>O<sub>3</sub>, SrO, and Al<sub>2</sub>O<sub>3</sub>. Especially Na<sub>2</sub>O (1-0.05%) and Al<sub>2</sub>O<sub>3</sub> (4-1%) contents decreased as a result of the experiments. It indicates the presence of Na<sub>2</sub>O ulexite mineral. The other two compounds did not change much.

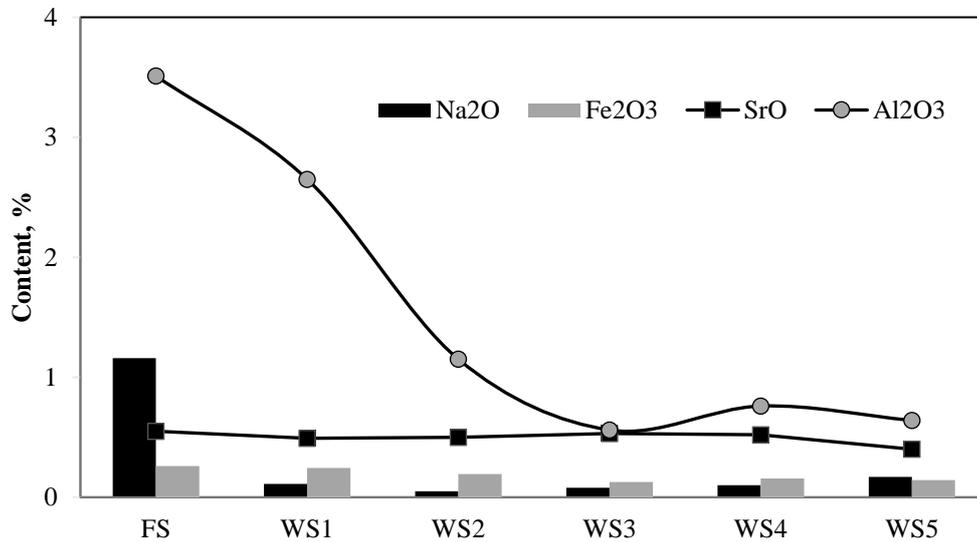
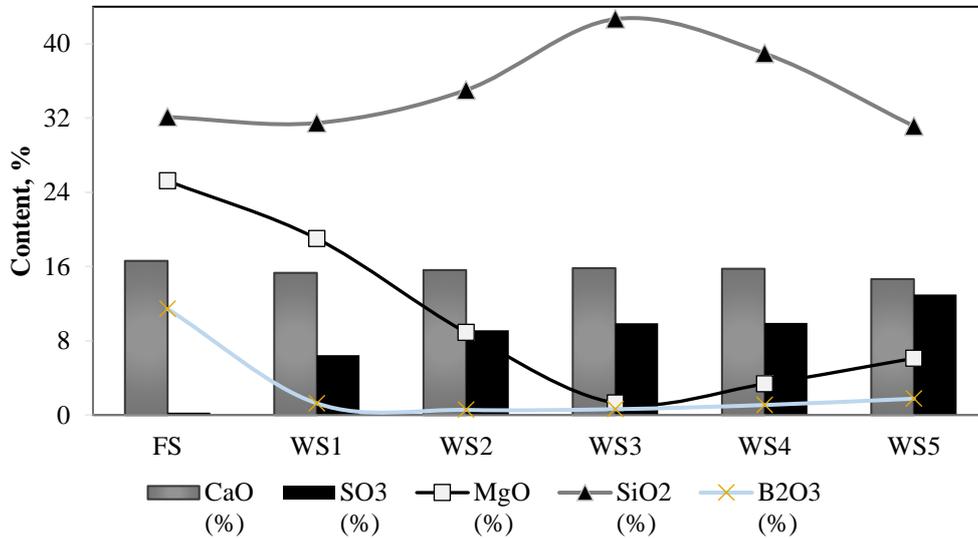


Figure 4. Chemical analyses result of feed and waste samples 1 (Na<sub>2</sub>O, Fe<sub>2</sub>O<sub>3</sub>, SrO, and Al<sub>2</sub>O<sub>3</sub>, %)

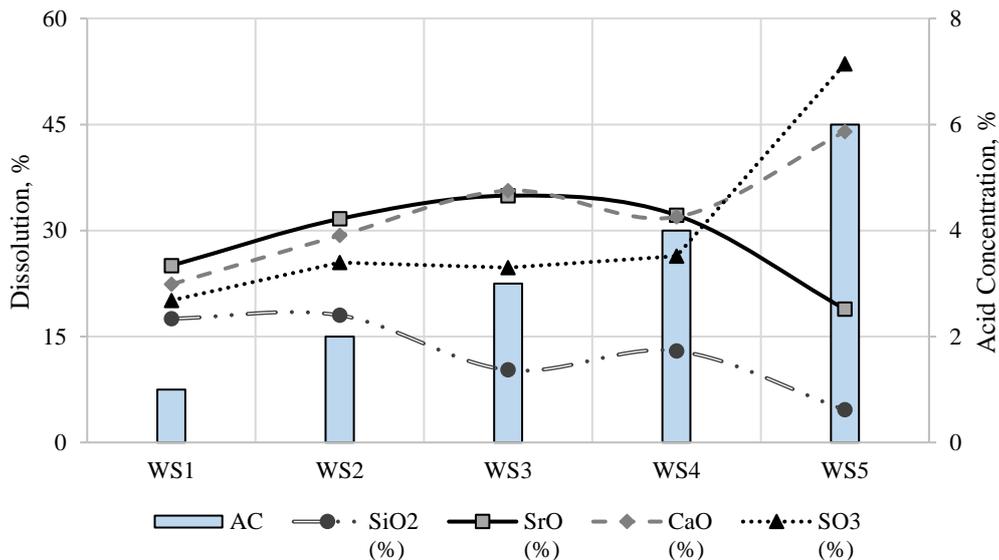
In Figure 5, CaO, SO<sub>3</sub>, MgO, and SiO<sub>2</sub> analyses are observed. The increase in the amount of SO<sub>3</sub> is directly related to the sulfuric acid. It was determined that the CaO analysis values (around 16%) progressed steadily, the SiO<sub>2</sub> content increased towards WS3 and then decreased towards the same point

as in the feed sample. MgO, on the other hand, was observed to be too low to be detected in the WS3 experiment (24-1%) and then increased to around 4-8%. B<sub>2</sub>O<sub>3</sub> is approximately 11.5% and changes to 0.5-1.8% at the end of leaching processes.



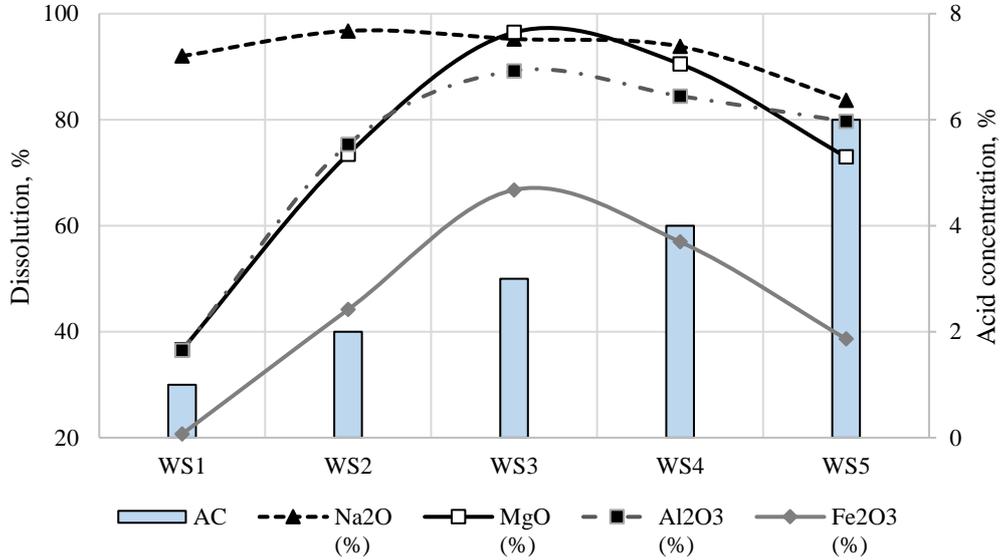
**Figure 5.** Chemical analyses result of feed and waste samples 2 (CaO, MgO, SO<sub>3</sub>, SiO<sub>2</sub>, and B<sub>2</sub>O<sub>3</sub>, %)

In Figure 6, CaO and SrO progressed to near solubility values up to WS4. In WS5, CaO increased more (44%), while SrO decreased in the opposite direction (19%). This situation of CaO is due to its precipitation in the form of CaSO<sub>4</sub>, generally turning into gypsum in sulfuric acid dissolution processes. SO<sub>3</sub> increased due to the increase in sulfuric acid concentration. When compared to other compounds, the SiO<sub>2</sub> solubility varies between 5-18%, indicating that it is the least soluble compound.



**Figure 6.** According to acid concentration (AC), %, dissolution of waste samples 1 (SiO<sub>2</sub>, SrO, CaO, and SO<sub>3</sub>, %).

Figure 7 examines the dissolution of the compounds found in 2. The solubility of Na<sub>2</sub>O is in the range of about 84-97%, and while the acid concentration is the highest, the solubility is the lowest. Other compounds were particularly elevated in the WS3 experiment and then decreased to WS2 levels.

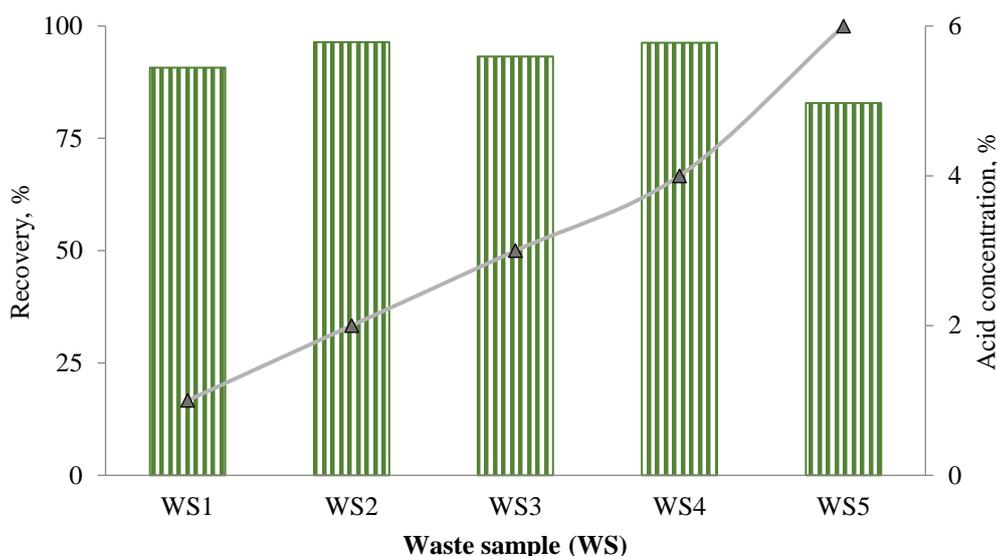


**Figure 7.** According to acid concentration (AC), %, dissolution of waste samples 2 (Na<sub>2</sub>O, MgO, Al<sub>2</sub>O<sub>3</sub>, and Fe<sub>2</sub>O<sub>3</sub>, %)

### 3.3. Recovery Results of B<sub>2</sub>O<sub>3</sub>%

The %B<sub>2</sub>O<sub>3</sub> recovery efficiencies were calculated with the solid waste and dissolved liquid analyses obtained as a result of the dissolution experiments in which different amounts of sulfuric acid were investigated and shown in Figure 8. These calculations are based on %B<sub>2</sub>O<sub>3</sub> in solid (waste) analysis results and B (ppm) element in liquid concentrate analysis.

According to the acid leaching results, %B<sub>2</sub>O<sub>3</sub> recovery yields were obtained with approximately 83-97%. Although these efficiencies tend to increase in general, it is observed that the recovery decreases in the experiment at the 6% concentration, where the amount of acid concentration is the highest. It is possible that this situation may be caused by reasons such as the reaction cannot occur after a certain acid concentration is exceeded, and the possibility of this inorganic acid reaction being reversible.



**Figure 8.** According to acid concentration (AC), %, %  $B_2O_3$  Recoveries of waste samples

Similarly, when boron (38 ppm boron) from Qaron Lake waters was removed from the loaded resin using 10% sulfuric acid, boron elution efficiency was determined as 75.04% [33]. In different studies examining the solubility of colemanite [24] and ulexite [23], it was determined that the solubility decreased with the increase in acid concentration at a concentration of 0.5-2M sulfuric acid. The main reason for this can be explained as the increase in the formation of  $SO_4^{2-}$  ions per unit volume and the formation of solid  $CaSO_4$  and  $CaSO_4 \cdot 2H_2O$  in the environment as in Reaction 6 [23, 24, 34, 35]. After this stage, the final production of pure boric acid can be achieved from this solution, as in the Boric Acid Plants, after the purification stage, crystallization, and washing-drying processes [19-21].

Literature studies on Bigadiç sludge waste are generally on flotation studies, and positive results have been obtained in these studies [29, 36]. Some of the hydrometallurgical studies carried out are as follows:

As a result of a study in which a heap leaching experiment was performed with the same acid (4.7%  $H_2SO_4$ ), the %  $B_2O_3$  recovery efficiency was approximately 80% [20]. In the experiments investigated for these wastes, organic acids with citric acid at 8-12% solid ratios, 40-98°C temperatures, 20-66 minutes, and 0.15-0.45M acid concentrations, recoveries in the range of 60-95% were achieved [37, 38]. A result of approximately 90% was also possible in the studies conducted for the recovery of  $B_2O_3$  with acetic acid (2-22% concentrations) from wastes [11].

In this study, the solid ratio in dissolution experiments is an important parameter when considering industrial dimensions. Because the more solids dissolve with less liquid, the lower the chemical consumption, the lower the water consumption. For this reason, the fact that the experiments were carried out at a rate of 7% can be seen as negative. However, boron wastes swell in aqueous media due to their dense clay content and mixing becomes a problem. Additionally, it is a remarkable output that the experiments reached over 90%  $B_2O_3$  recovery efficiencies in a short time of 1 hour. These results are important in terms of their application both in Bigadiç wastes and in other boron plant wastes in Turkey. Thus, it will be possible to obtain boron from these wastes and to use the remaining content in different sectors and/or feed it back to the open pit.

The utilization of boron wastes will provide additional income by helping to obtain refined products such as boric acid from the wastes accumulated in the waste ponds/dams since the beginning of

production from their plants. In addition, it will be able to solve the location problem of ponds/dams and environmental issues related to boron and other content in wastes [20, 39, 40].

Consequently, for the success of this study on an industrial scale, pilot-sized, more applicable leaching tanks, and better mixing speeds are required. Therefore, by increasing the % solid ratio, it will be possible to achieve similar boric acid recoveries in which more solids can be processed with the same acid concentration (or near this value).

#### **4. CONCLUSIONS**

In this study, the high boron content (11.5% B<sub>2</sub>O<sub>3</sub>) in Bigadiç slime waste was dissolved with sulfuric acid, an inorganic acid. The boron minerals in this waste sample indicated the presence of ulexite and colemanite minerals due to their chemical content of sodium, calcium, and boron. The remaining part of the sample consisted of a dense clay structure.

The optimum condition of the leaching experiments was obtained from the experiment where the acid concentration was 2%, and the B<sub>2</sub>O<sub>3</sub> leaching efficiency was determined as 96.38%. The reason for obtaining the lowest efficiency at 6.1% acid concentration was the increase in SO<sub>4</sub><sup>-2</sup> ions per unit volume. Therefore, solid CaSO<sub>4</sub> and CaSO<sub>4</sub>·2H<sub>2</sub>O were formed in the environment.

In conclusion, the study showed that Bigadiç and other boron plant wastes can be recycled. Correspondingly, it has been revealed that these valuable contents can be recovered, the remaining contents such as clay can be used in different areas, and the reusability of waste ponds and the search for space can be helped by eliminating their environmental impacts.

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#### **CONFLICT OF INTEREST**

The author states that there are no conflicts of interest regarding the publication of this article.

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