

## Assessment of the batch flotation results of finely sized colemanite in terms of gangue minerals and the importance of desliming

Şafak Gökhan Özkan <sup>1,2</sup>, Martin Rudolph <sup>2</sup>

<sup>1,2</sup> Turkish-German University, The Institute of the Graduate Studies in Science and Engineering, Department of Robotics and Intelligent Systems, 34820, Beykoz, Istanbul, TÜRKİYE

<sup>2</sup> Helmholtz-Zentrum Dresden-Rossendorf, Helmholtz-Institute Freiberg for Resource Technology, Chemnitz Straße. 40, 09599 Freiberg, GERMANY

Corresponding author: [safak.ozkan@tau.edu.tr](mailto:safak.ozkan@tau.edu.tr) (Şafak Gökhan Özkan)

**Abstract:** It is aimed in this article to re-evaluate the necessity for the desliming process prior to froth flotation of finely sized colemanite samples from the Hisarcik region at the Emet borate deposits of Türkiye. For this purpose, previous batch flotation conditions and quantitative flotation results were considered by not only taking the previous findings targeting only B<sub>2</sub>O<sub>3</sub>% grade and recovery into account but also other main components or gangues (i.e. As<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, and SiO<sub>2</sub>) by using today's modern laboratory characterization analyses such as XRD and MLA and data evaluation methods. The current assessment proved that newly constructed particle size distribution results showed even 10 min of grinding would be more relevant, especially if the slimes would be ultimately problematic for flotation. The desliming process is certainly necessary and relatively effective as much of the gangue mineral components could be removed easily at minus 20 µm before the flotation process. However, the desliming might also cause B<sub>2</sub>O<sub>3</sub> losses in slimes, and this time, the final mass and assay data should be assessed for further grade-recovery calculations. Although AP825, a sodium alkyl sulphonate, was a very effective collector for colemanite flotation in previous studies, the current assessment revealed that CA927, a sulphosuccinamate (anionic type collector), might be considered as an alternative collector. However, when it was successful in getting the targeted B<sub>2</sub>O<sub>3</sub>% content, selectivity against gangue constituents, such as As<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> in the current conditions cannot support this idea. This paper shows the necessity of controlling optimum grinding time for targeted liberation particle size, the importance of the desliming process in order to avoid losses of valuable components in fines, and the consideration of gangue components in all products at all particle size fractions during colemanite flotation when evaluating the overall recovery.

**Keywords:** colemanite, borate, flotation, desliming, mineral liberation analysis (MLA)

### 1. Introduction

The list of critical raw materials (CRMs), which have frequently been re-updated by the European Union in recent years, includes a wide range of minerals, materials, elements, and metals linked to all industries at all supply chain stages, such as lithium, copper, graphite, cobalt, borates, etc., given the developments regarding the increase in demand for energy and battery storage (Anon, 2023). The ever-increasing quality of life and development in the modern world depends on the production efficiency and technology of many of these critical raw materials. For example, a smartphone, a space satellite, or a medical diagnostic device can contain up to 50 different types of metals, contributing to its small size, lightweight, and functionality. Environmental issues, especially clean technologies, solar panels, wind turbines, electric vehicles, and energy-efficient lighting, are also closely linked to the mining and technology development of critical raw materials that cannot be replaced.

Boron which is one of the important CRMs is the 51<sup>st</sup> most common element present in the earth's crust at an average concentration of about 3 ppm as borates or boron silicates. Elemental boron, B is unique in that is the only non-metal in Group 13 (IIIA) of the Periodic Table. Boron atomic number 5,

atomic weight of 10.81 g, and specific gravity 2.0-2.5 g/cm<sup>3</sup> is a dark brown powder in the amorphous form and a yellowish-brown, hard, brittle solid in the monoclinic crystalline form. Its melting point is very high (2,300°C). It is the only electron deficient non-metallic element with a great affinity for oxygen and it has more similarity to carbon and silicon than to other elements in Group 13.

Although there are at least 150 minerals known to contain boron, few of the boron minerals have been mined as an ore in nature. These minerals generally have alkali anions such as sodium (Na), calcium (Ca), and magnesium (Mg) with hydro-borates. The most common boron minerals that are currently utilised in any quantity for their borate contents are borax (tincal), colemanite and ulexite. In Table 1, the principal boron minerals are shown with their chemical compositions and approximately B<sub>2</sub>O<sub>3</sub>% weight contents (Özkan, 1994).

Table 1. The most common boron minerals (modified from Özkan, 1994; 2023)

Mineral	Common Formula	Chemical Composition	Content (B <sub>2</sub> O <sub>3</sub> %)	Content (H <sub>2</sub> O %)
Colemanite	Ca <sub>2</sub> B <sub>6</sub> O <sub>11</sub> . 5 H <sub>2</sub> O	Ca[B <sub>3</sub> O <sub>4</sub> (OH) <sub>3</sub> ]. H <sub>2</sub> O	50.81	21.91
Ulexite (Boronatrocalcite)	NaCaB <sub>5</sub> O <sub>9</sub> . 8 H <sub>2</sub> O	NaCa[B <sub>5</sub> O <sub>6</sub> (OH) <sub>6</sub> ]. 5 H <sub>2</sub> O	43.07	35.57
Probertite (Kramerite)	NaCaB <sub>5</sub> O <sub>9</sub> . 5 H <sub>2</sub> O	NaCaB <sub>5</sub> O <sub>7</sub> (OH) <sub>4</sub> . 3 H <sub>2</sub> O	49.10	25.64
Tincal (Natural Borax)	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> . 10 H <sub>2</sub> O	Na <sub>2</sub> (B <sub>4</sub> O <sub>5</sub> ) (OH) <sub>4</sub> . 8 H <sub>2</sub> O	36.51	47.24
Kernite (Rasorite)	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> . 4 H <sub>2</sub> O	Na <sub>2</sub> [B <sub>4</sub> O <sub>6</sub> (OH) <sub>2</sub> ]. 3 H <sub>2</sub> O	51.00	26.10
Priceite (Pandermitite)	CaB <sub>10</sub> O <sub>19</sub> . 7 H <sub>2</sub> O	Ca <sub>2</sub> B <sub>5</sub> O <sub>7</sub> (OH) <sub>5</sub> . H <sub>2</sub> O	49.87	18.27
Hydroboracite	CaMgB <sub>6</sub> O <sub>11</sub> . 6 H <sub>2</sub> O	CaMg[B <sub>3</sub> O <sub>4</sub> (OH) <sub>3</sub> ] <sub>2</sub> . 3 H <sub>2</sub> O	50.53	26.15

Colemanite is the preferred calcium-bearing borate hydrate. It is slightly soluble in water, although it dissolves readily in acid. It is an oxide-type mineral classified as a sparingly soluble ionic salt. Colemanite flotation requires the use of collectors such as naphthenic acids, fatty acids, alkyl sulphates, alkyl sulphonates, or hydrocarbon oils (vapour oils, kerosene, and fuel oils) but limited information is available in the open literature (Yarar, 1971; Crozier, 1992).

The solubility of colemanite has been investigated by several researchers, while these data are cited in the literature as one part in 1,100 parts and at 20-25 °C as 0.19% CaO plus 0.285% B<sub>2</sub>O<sub>3</sub> neither of which is a clear statement (Yarar, 1971). Surface chemistry based processes are largely influenced by mineral solubility. Therefore, more recent and reliable solubility data of colemanite has been determined as 0.8 g/dm<sup>3</sup> by Yarar (1985) and 1.0 g/dm<sup>3</sup> by Celik et al. (1992) both of which show the results of theoretical studies.

When colemanite is dissolved in water, it will release a number of species into solution like other sparingly soluble minerals, therefore the surface of colemanite will be charged with a certain electric potential. This surface charge generation is caused by solids concentration in the solution in connection with the pH value. The zero point of charge (zpc) of colemanite in the presence of 2.10<sup>-3</sup> mol/dm<sup>3</sup> NaCl was determined to be approximately 10.5 - 10.7 in agreement with Yarar (1985) and Celik et al. (1992). The relationship between the zeta potential of colemanite and its flotation response could be helpful in the determination of surfactant uptake by colemanite.

In the literature, Yarar (1988) reported a contact angle for colemanite mineral is 43° with 5x10<sup>-3</sup> mol/dm<sup>3</sup> sodium oleate (NaOL) solution. The significance of contact angle arises from the fact that it is a measure of surface wettability; θ > 0 indicates a hydrophobic solid. The contact angle values of θ > 10° generally indicate that particles can form bubble-particle contacts strong enough to resist turbulence in a conventional flotation cell.

Colemanite flotation may be achieved by means of only collectors and frothers, although it is known that modifiers for pH regulating, activators such as metallic sulphates especially CuSO<sub>4</sub> and BaSO<sub>4</sub>, depressants for silica and calcite such as Na<sub>2</sub>SiO<sub>3</sub> and starch and flocculants for desliming of clay minerals prior to flotation are also used. For example, depressants for arsenic sulphides, calcite, and clay minerals are suggested by companies such as Allied Colloids with Procol series DA911, DA914, DA1099, and DN895 and Henkel with Acrol F22 and Suspendol PPK (Yarar, 1971; 1973; 1979; Crozier, 1990; 1992).

Colemanite flotation collectors are cited as sodium dodecyl benzene sulphonate, sodium oleate, dodecyl amine hydrochloride, naphthenic acids, hydrocarbon oils (vapour oils, kerosene, fuel oils), and some commercial reagents of which chemical compositions could depend upon either those main chemicals above or not be known exactly such as Leviron, Soap production disposal, Emigol, Coco Amine Acetate, Aerosols-OS, Cyanamid Aero Promoter series 801R, 825, 830, 845, 851, 853, 857, and 727, Hoechst Flotisor series FS2, S72, V3960, V4083, V4085, Arkomon SO, and F2874, Henkel OMC series 111, 5020, 5050P, and collector FS/R, Allied Colloids Procol series CA540 and 927, DP1 series 4396 and 4619J. It should be noted that a few of these commercial collectors have been tested by some researchers. Desirable results could not be obtained when each collector was used alone, and the necessity of collector combinations was recognised. There is an interesting point that while some of these collectors are anionic like sodium dodecyl benzene sulphonate, others are cationic like dodecyl amine hydrochloride (DAH). This shows the unique property of colemanite in terms of floatability with both types of collectors (Ayok and Tolun, 1976; 1978; 1979; Kose et al., 1988).

Yarar (1971) utilised the Hallimond Tube and Modified Partridge-Smith Cell for his experiments for colemanite flotation at different times. In these tests, some note-worthy findings were determined such as the selection of a convenient reagent system by using zeta potential and contact angle measurement in terms of the floatability of colemanite. For example, when sodium dodecyl benzene sulphonate and sodium oleate were utilised, both of them could lead to flotation of colemanite with 100% recovery and at equal concentrations, while sodium oleate was a more powerful collector than sodium dodecyl benzene sulphonate which was more selective towards colemanite.

According to Celik et al. (1992) micro flotation test results, the zeta potential of colemanite varied considerably with changes in solids concentration due to the dependence of  $\text{Ca}^{2+}$  ion release with the amount of solid added. The addition of sodium dodecyl sulphonate (SDS) and dodecyl amine hydrochloride (DAH) marginally affected the zeta potential of colemanite with pH. The sharp changes corresponded to the solubility limit of calcium dodecyl sulphonate and that of dodecyl amine respectively. Anionic (SDS) and cationic (DAH) surfactants floated colemanite in the same concentration region corresponding to the formation of hemi micelles. Flotation of colemanite with SDS showed a decrease with increasing pH indicating the role of electrostatic interactions in the system. Flotation of colemanite with DAH at  $5 \cdot 10^{-5}$  mol/dm<sup>3</sup> exhibited a plateau at pH 10 where maximum amounts of ion molecular complexes formed and then remained approximately constant due to precipitation of amine above this pH.

To summarize, froth flotation investigations for colemanite, which is a valuable borate mineral for the Turkish economy began in the early 1970s with a few scientists' enormous efforts by using mainly manual characterization, concentration tests, data evaluation methods, and personal experiences in Türkiye. The main parameters used during flotation and the relatively compared results are outlined in Table 2 in detail. There have been a lot of research studies on colemanite flotation since the early 1990s in light of those previous efforts despite the limited laboratory facilities. For this purpose, this study aimed to assess and re-evaluate the previous batch flotation conditions and results including desliming conditions prior to flotation of finely sized colemanite and to take the previous findings not only targeting  $\text{B}_2\text{O}_3\%$  grade and recovery into account but also other main components or gangues (i.e.  $\text{As}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ , and  $\text{SiO}_2$ ) by using today's modern laboratory characterization analyses and data evaluation methods.

## 2. Materials and methods

### 2.1. Source and sample preparation

The previously supplied representative colemanite ore and crystal samples were taken from the Bottom Section of the Hisarcik open pit mine in Türkiye in 1992. Prior to previous flotation testing, some preliminary work was done on the raw colemanite samples for material characterization to find out what the samples contained as principal and gangue minerals and in which particular particle size range. The sample preparation was followed by identification techniques such as X-ray diffraction analysis, complete chemical analysis, and particle size distribution tests.

Approximately 30 kg of colemanite ore sample, sized at minus 200 mm, was first crushed with a primary jaw crusher to obtain the samples minus 40 mm, then quartered twice. One-fourth of the sample

was saved for the records, the rest was crushed to minus 8 mm through a secondary jaw crusher. Those archived samples have been kept in safe storage for about 30 years and also used in this present investigation. Some of the above mentioned characterization studies were also performed on these archived samples and the results were compared to each other for making further comments.

Table 2. Experimental conditions of colemanite flotation studies (Modified from Özkan, 1994; 2023)

Conditions	Yarar, 1971	Ayok, 1976	Kose, 1988	Özkan, 1994
Feed Particle Size	-200 $\mu\text{m}$	-200 $\mu\text{m}$	-150 $\mu\text{m}$	-210 $\mu\text{m}$
Slime Particle Size	-53 $\mu\text{m}$	-71 $\mu\text{m}$	-53 $\mu\text{m}$	-20 $\mu\text{m}$
Slime/Feed Ratio	25%	30%	50%	15-20% with controlled grinding
Solids/Liquid Ratio	30%	25%	20%	20%
Deslimed Feed Grade	40% $\text{B}_2\text{O}_3$	40% $\text{B}_2\text{O}_3$	36% $\text{B}_2\text{O}_3$	40% $\text{B}_2\text{O}_3$
pH Regulation	Natural	Natural	Natural	Natural (7.5-8.0)
Collectors	-Naphthenic Acid +Kerosene (1.0+1.2 kg/ Mg) -Naphthenic Acid +Kerosene+AP825 (0.5+0.9+0.3 kg/Mg) -AP825+ Naphthenic Acid (0.5+0.085 kg/Mg) (5% emulsion)	AP825+Kerosene (2.0+0.25 kg/Mg) (5% emulsion)	AP825+Kerosene (1.6+0.4 kg/Mg) (5% emulsion)	-Cyanamid AP825 (a sodium alkyl sulphonate) (2.0 kg/Mg) (5% emulsion) -Allied Colloids CA927 (a sulphosuccinamate) (1.5 kg/Mg) (5% emulsion) -Henkel OMC5050P (a petroleum sulphonate), OMC111 (a modified sulphosuccinate) and OMC5020 (a partial sulphonated mixture of fatty material) were also tested at (2.0 kg/Mg) (5% emulsion)
Frothers	MIBC, Flotanol G	Flotanol G	Pine Oil (50 g/Mg)	AF70 (100 g/Mg)
Cond. Time	5 min	5-10 min	No	5 min
Flot. Time	10 min	10 min	10 min	5 min
<b>Outcomes</b>	<b>Yarar, 1971</b>	<b>Ayok, 1976</b>	<b>Kose, 1988</b>	<b>Özkan, 1994</b>
Conc. Grade	48% $\text{B}_2\text{O}_3$	48% $\text{B}_2\text{O}_3$	46% $\text{B}_2\text{O}_3$	47-49% $\text{B}_2\text{O}_3$
$\text{B}_2\text{O}_3$ % Recovery	90	95	88	80-90 with conventional and 90-95 with ultrasonically treated samples

The crushed samples were passed through a tertiary roll crusher in order to obtain convenient particle sizes for the grinding tests, for which a laboratory rod mill was used to grind samples into different particle sizes over different periods. All grinding tests were carried out in a rod mill whose operating parameters were kept constant as given below:

- Inner diameter of mill: 157 mm,
- Length of mill: 314 mm,
- L/D ratio: 2 / 1,
- Diameter of rods: 24 mm
- Length of rods: 266 mm,

- Number of rods: 10,
- Weight of rods: 10532 g
- Speed of mill: 100 rpm (75% of critical speed),
- Weight of Sample used approx. 1,000 g

In order to reach the desired liberation particle size, previously determined -210  $\mu\text{m}$  after detailed microscopic investigations, the optimum grinding time with a dry batch rod mill was determined to be 15 min by wet sieve analyses and drawing cumulative undersize graphs by 5 min intervals (Özkan, 1994).

## 2.2. Mineralogical and chemical analyses

For mineralogical analyses, X-ray diffraction techniques were used to determine the crystal structure of representative colemanite samples. The general plan was first to determine the d-spacing for each diffraction peak on the X-ray diffraction patterns and then to plot these on a rule. This rule was compared with a graph of reference samples. The reference samples cited in the PDF (Powder Diffraction File) number 33,267 of the ICDD (International Centre for Diffraction Data) were from Ryan, Death Valley, Inyo County of California in the USA and are exhibited at the Mineralogy-Geology Museum of Delft University, Netherlands. These samples were described as transparent and colourless and likely to exhibit the preferred orientation. Opaque grey samples from other localities gave an identical diffraction pattern.

After determining that the samples were almost entirely crystalline colemanite, it was necessary to determine what these samples contained as major elemental components. A crushed sample was ground to -106  $\mu\text{m}$  with a rod mill in order to obtain the required particle size for complete chemical analysis. The amount of ground sample was reduced to 100 g with a spinning-riffler, avoiding any contamination, and this representative colemanite ore sample was subjected to a complete chemical analysis for which as well as wet chemical analysis methods, such as gravimetric for sulphates, titration for CaO, B<sub>2</sub>O<sub>3</sub>, and sulphides, and absorption for carbonates, some instrumental analysis methods such as A.A.S. (Atomic Absorption Spectrometry for SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, MgO, TiO<sub>2</sub>, MnO<sub>2</sub>, SrO, and As<sub>2</sub>O<sub>3</sub>, and A.E.S. (Atomic Emission Spectrometry) for Na<sub>2</sub>O and K<sub>2</sub>O were employed. Complete chemical analysis results are presented in Table 3.

The results of the previous material characterisation tests revealed that the colemanite ore sample from the Hisarcik region at the Emet borate deposits of Türkiye has specific characteristics in terms of

Table 3. Complete chemical analysis results of representative colemanite samples (Özkan, 1994)

Components	Weight (%)	Method Used	Precision ( $\pm$ %)
B <sub>2</sub> O <sub>3</sub>	46.90	Volumetric	0.20
CaO	20.50	Volumetric	0.20
LOI* or H <sub>2</sub> O(at max. 800°C)	22.51	Mass	0.10
SiO <sub>2</sub>	3.77	A.A.S.	1.50
As <sub>2</sub> O <sub>3</sub>	2.00	A.A.S.	0.70
SrO	1.55	A.A.S.	0.70
MgO	1.18	A.A.S.	1.00
Al <sub>2</sub> O <sub>3</sub>	0.86	A.A.S.	1.50
CO <sub>3</sub>	< 0.50	Absorption	2.00
Fe <sub>2</sub> O <sub>3</sub>	0.31	A.A.S.	0.70
TiO <sub>2</sub>	0.21	A.A.S.	1.00
SO <sub>4</sub>	0.25	Gravimetric	0.50
K <sub>2</sub> O	0.19	A.E.S.	0.70
Na <sub>2</sub> O	0.06	A.E.S.	0.70
SO <sub>3</sub>	0.02	Volumetric	3.00
MnO <sub>2</sub>	< 0.01	A.A.S.	0.70

\*LOI: Loss on ignition

mineralogical and chemical content which directly or indirectly affect the mineral processing tests due to crushability, grindability, and liberation degree prior to application of a particular concentration method.

Material characterization also showed that this ore contains almost pure colemanite crystals, although some arsenic and iron sulphides and clay minerals were observed during the sample preparation tests. Interpretation of the identification tests clearly showed that the target mineral which would be recovered during the subsequent flotation could be either colemanite or other observed gangue minerals, i.e. orange and red coloured arsenic sulphides or brownish black clay minerals which could be seen in the fine size fractions, especially during the sieve analysis and desliming stage.

Consequently, it was found that the boron content of the fine material sharply decreased at  $-20\ \mu\text{m}$ , and this fraction was therefore considered as slimes. Thus, all concentration tests would be affected by the slime content which would decrease the final recovery due to desliming losses. This unavoidable effect was minimised by a controlled-grinding procedure which was achieved by altering the grinding parameters such as time, amount of sample, and characteristics of grinding media.

### 3. Current results and comparison with previous findings

In Table 2, the main parameters used during colemanite flotation and the relatively compared results from all available previous research were outlined. In this article, the results of some repeated laboratory characterization analyses on the same feed sample, i.e. X-Ray Diffraction (XRD) and Mineral Liberation (MLA), and a comparison of grade-recovery curves under several parameters are given in detail by using modern data evaluation methods. These methods are also employed in order to understand the necessity of desliming before flotation and to reveal the possible effects of  $\text{B}_2\text{O}_3$  losses and contamination of the products due to gangue minerals which were very finely sized and distributed in feed.

#### 3.1. Particle size distribution

From previous investigations (Özkan, 1994), the optimal grinding time for the crushed feed sample was determined as 15 min to reach the liberation particle size of  $-210\ \mu\text{m}$  according to separate cumulative undersize graphs. In present investigations, the same comminution data sets were re-assessed and compared to each other by drawing cumulative undersize plots according to time intervals as shown in Fig. 1.

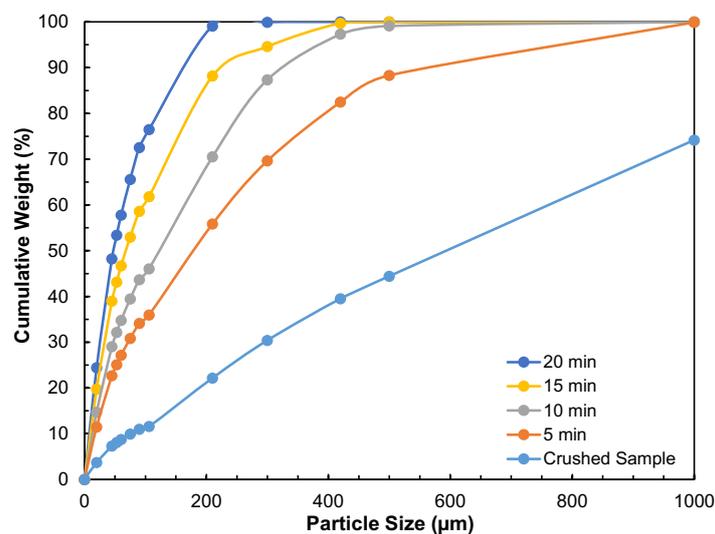


Fig. 1. Re-constructed particle size distribution graphs (Modified from Özkan, 1994)

Fig. 1 shows some important particle size average values such as  $d_{50}$  and  $d_{80}$  for different grinding time intervals. While  $d_{50}$  values seem to be 175-120-70-45  $\mu\text{m}$ ,  $d_{80}$  values are determined as 395-255-175-120  $\mu\text{m}$  for 5-10-15-20 min grinding times, respectively. When the particle size distribution curves in Fig. 1 are investigated in detail, there might be a problem of overgrinding if 15 min of grinding for

liberation is chosen as optimal. It is also seen that 15 min may be considered as too long if the liberation is desired at  $-210\ \mu\text{m}$ . In other words,  $d_{50}$  value is  $-70\ \mu\text{m}$ , and  $d_{80}$  value is  $-175\ \mu\text{m}$  if 15 min of grinding time is accepted as optimal. Perhaps even 10 min of grinding might be relevant for the liberation at  $210\ \mu\text{m}$ . Especially if the slimes are ultimately problematic for further flotation tests, the previous measurements and evaluations should be re-assessed by using novel analytical sieving methods, also considering new particle size distribution approaches.

If the cumulative undersize grade and distribution data from the previous sieving tests are evaluated together, the following graphs can be drawn as seen in Fig 2.

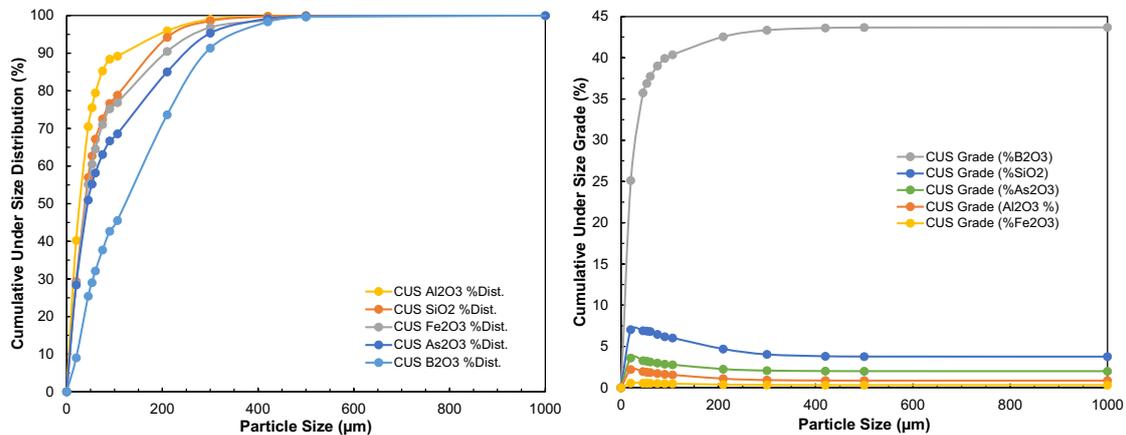


Fig. 2. Re-constructed cumulative undersize grade and distribution graphs (Modified from Özkan, 1994)

Fig. 2 shows that although the B<sub>2</sub>O<sub>3</sub> grade is uniformly distributed at relatively large size fractions, the gangue constituents spread into very fine sizes. It is noteworthy to emphasize that clayish constituents behave together in finer size fractions, the targeted B<sub>2</sub>O<sub>3</sub> values seem to be at the large size fractions and connected to As<sub>2</sub>O<sub>3</sub> data. Previous evaluations showed that if the sample is deslimed at a certain particle size, then B<sub>2</sub>O<sub>3</sub>% grade and distribution can be increased before any further concentration tests. Furthermore, gangue minerals can be easily removed as they are distributed at finer size fractions before flotation. All of above information paved the way that if the feed is ground to  $-210\ \mu\text{m}$  and then deslimed at  $-20\ \mu\text{m}$ , further concentration tests could be planned accordingly. However, if the grinding and desliming are performed at the above conditions, this might cause large amounts of B<sub>2</sub>O<sub>3</sub> losses in fine size fractions as well as finely sized gangues, such as clays, iron, silica, and arsenic containing minerals might also cause slime coatings problems during further concentration tests due to overgrinding.

### 3.2. Microscopy, X-ray diffraction (XRD), and mineral liberation analyses (MLA)

The feed colemanite sample archived for several years was firstly macroscopically checked against aging and possible oxidation thoroughly before using the proper sample preparation method for microscopic and X-ray diffraction analyses. After observing no ageing, damage, corrosion, or oxidation, it was decided to continue further characterization. Fig. 3 shows the photos of the raw sample, prepared, polished, and resin-embedded sections.

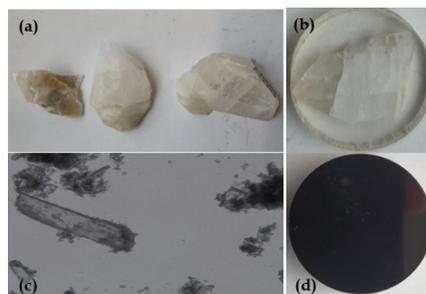


Fig. 3. Raw and prepared colemanite samples for characterization analyses (a) raw crystals (b) prepared thin sections (c) prepared fine size fractions under the microscope (50 $\times$ ), and (d) resin embedded fine size fraction

After obtaining properly prepared representative samples, the investigation continued by using PANalytical Empyrean model X-ray diffraction analysis device according to phase identification (qualitative XRD) based on the ICDD PDF-4+2020 database. PANalytical HighScore 3.0.4 program (semi-) quantitative phase identification and Profex 3.10.2 software (using Rietveld refinement) were also used with PIXcel3D-Medipix3 1x1 detector. Fig. 4 shows the XRD analyses results.

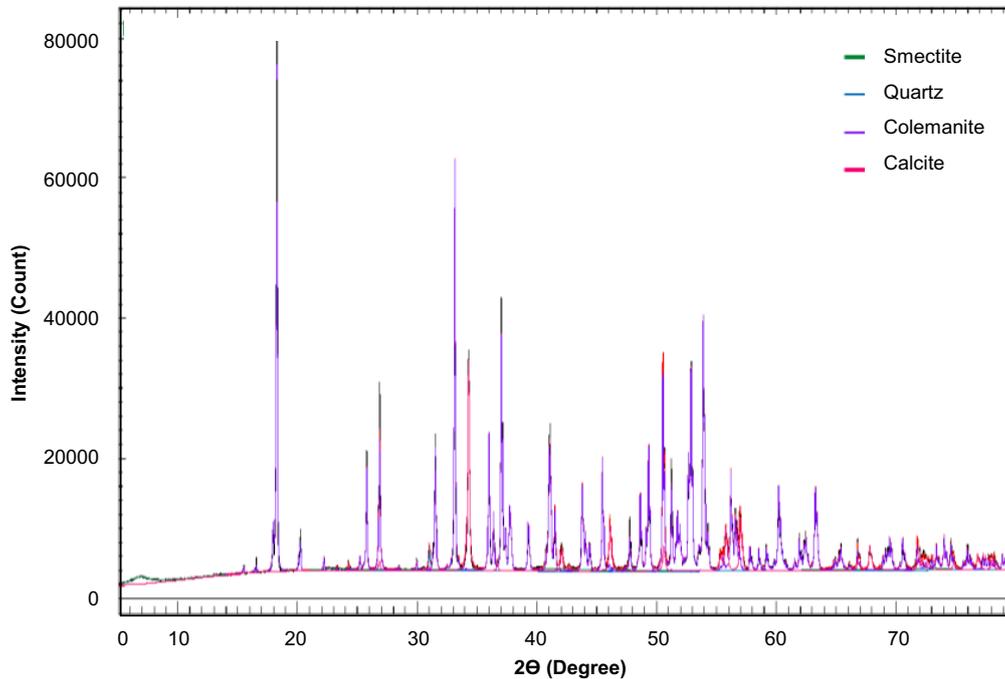


Fig. 4. The current XRD analysis results

Although previous XRD analyses showed colemanite peaks together with some arsenic sulphides and clay minerals due to manual access to all XRD pattern databases in the 1990's, the current results detected colemanite, calcite, quartz, and smectite peaks, and the absence of arsenic minerals. In this assessment work, some novel characterization methods were used to determine whether those above listed constituents really exist in the feed. Especially MLA measurements may reveal their form, quantity, and associations with each other in feed and in which particle size where enough liberation and desliming could be achieved. Then, Mineral Liberation Analyses (MLA) were also conducted to reveal different mineral associations and their relations inside current colemanite crystals. The device model is FEI Quanta 650 MLA-FEG with Bruker Quantax X-Flash 5030 EDS-Detectors using MLA-Suite software. Fig. 5 shows the processed SEM images (a), modal mineralogy (b), mineral locking for colemanite (c), the particle size of the sample, (d) particle size distribution of main minerals, and (e) data from the MLA measurements.

When the MLA analysis results are assessed together with XRD analysis data and compared with the previous findings and evaluations from 1994, there are certain differences in terms of mineral characterization concerns. For example, the current sample contains a significant amount of colemanite, calcite, talc, and quartz minerals, and their mineral particle size distributions differ from each other as seen in Fig. 3. There is also a small amount of unknown phases which may have been previously estimated and evaluated as the existence of iron and arsenic minerals. Tables 4 and 5 present the calculated assay and elemental distribution data according to different mineral source data.

When the present findings by characterization analyses are evaluated in detail, the results of previous mineralogical and chemical analyses showed some misleading information in terms of gangue minerals' associations. Although the previous XRD and chemical analyses were consistent with each other, especially showing the target  $B_2O_3$  content as correctly as possible, there is now a small amount of unknown phases that may have been previously estimated and evaluated as the existence of arsenic and iron minerals inside the feed. Besides, there is no clear evidence of the existence of arsenic minerals inside the current colemanite sample.

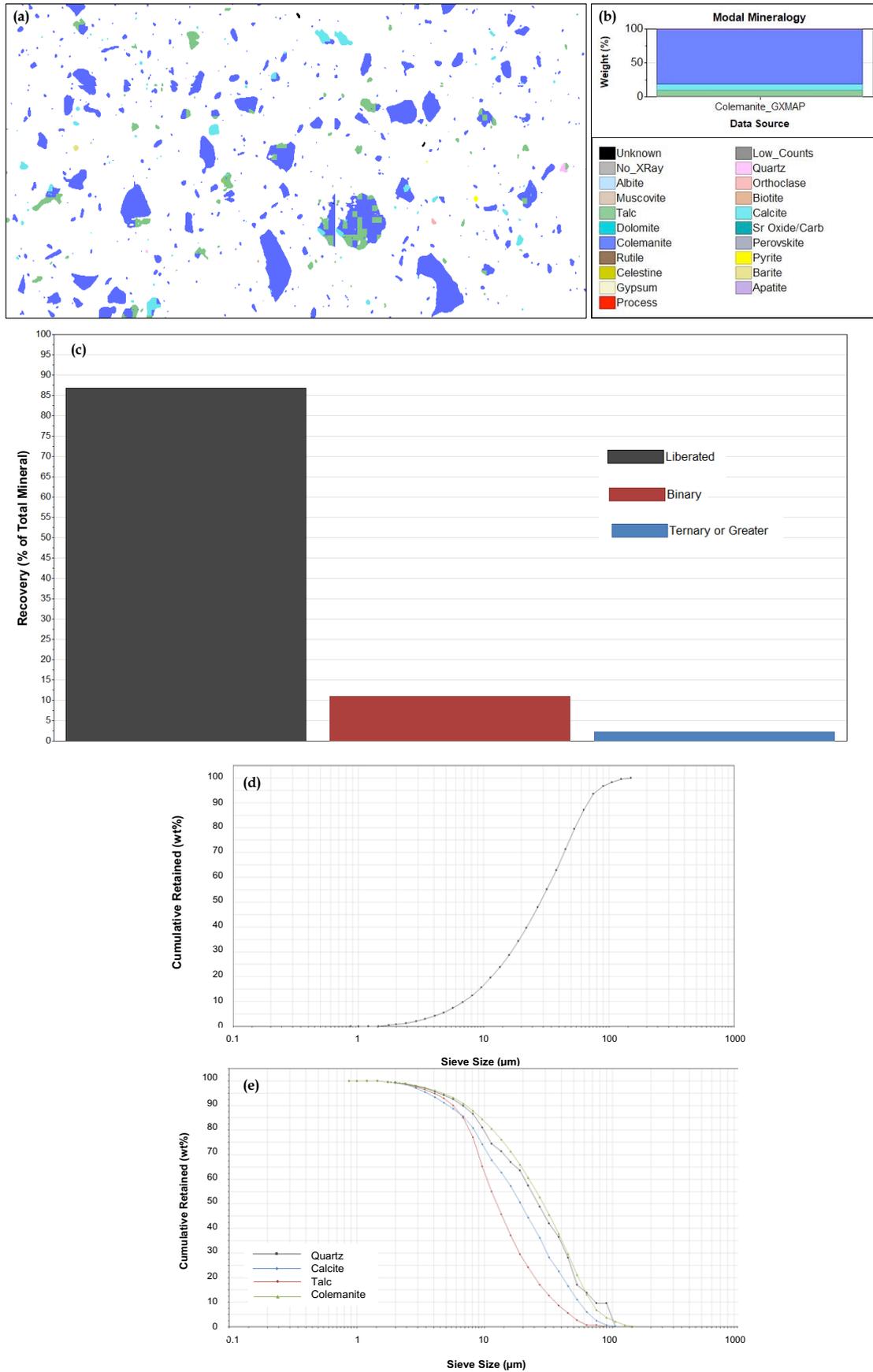


Fig. 5. The MLA analysis results (a) processed SEM image of the feed by MLA (b) estimated modal mineralogy by MLA (c) mineral locking for colemanite mineral by MLA (d) particle size distribution of main minerals by MLA, and (e) particle size distribution of the sample by MLA

Table 4. The calculated assay of the sample according to the MLA

Element	Weight (%)	Element	Weight (%)
Al	0.03995	Mg	1.72658
B	12.71762	Na	0.00237
Ba	0.01189	O	59.65479
C	1.09283	P	0.00093
Ca	19.33916	S	0.04690
Cl	0.00012	Si	3.03219
F	0.00423	Sr	0.08205
Fe	0.03566	Ti	0.02328
H	2.02380	Unknown	0.11176
K	0.05325	Total	99.99937

Table 5. The elemental distribution of the sample according to the mineral source by the MLA

Mineral/Element (%)	Al	B	C	Ca	F	Fe	K	Mg	S	Si	Sr	Ti
Quartz										12.36		
Albite	7.71			0.00						0.30		
Orthoclase	32.51						35.34			1.34		
Muscovite	0.43				0.19		0.16			0.01		
Biotite	59.35				98.31	68.85	64.50	3.09		2.44		
Talc								95.22		83.56		
Calcite			97.29	18.34								
Dolomite			2.63	0.25				1.69				
Sr Oxide/Carbonate		0.08								7.76		
Colemanite		100.0		81.27								
Perovskite				0.10								98.76
Rutile												1.24
Pyrite						31.15			27.20			
Celestine									59.03		92.24	
Barite									5.92			
Gypsum				0.02					7.85			
Apatite				0.01	1.50							
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

#### 4. Discussion and conclusions

In Table 2, the far right column shows the all parameters used in Özkan's previous colemanite flotation work (Özkan, 1994). The outline of those previous flotation results for only  $B_2O_3\%$  values at optimal collector consumption with and without the desliming stage is presented in Table 6.

Table 6 shows that colemanite flotation could not be achieved without the desliming stage. However, if  $-20\ \mu\text{m}$  particle size fraction is decanted, sieved, removed, or deslimed before flotation, acceptable  $B_2O_3\%$  grade and recovery values can be obtained according to optimal collector dosage usage. As seen from previous studies mentioned in Table 2, AP825, a sodium alkyl sulphonate sometimes commonly called R825, a petroleum sulphonate is an effective anionic reagent as almost 95% of the material was floated when it was used at an acceptable dosage of 2,000 g/Mg. Also, the addition of kerosene to AP825 was found not to be useful since it did not have a significant effect on grade or recovery values. The other collectors seen in Table 6, CA927 (a sulphosuccinamate), OMC111 (a modified alkyl sulphosuccinate), OMC5050P (a petroleum sulphonate), and OMC5020 (a partial sulphonated mixture of fatty material) were also tested in order to determine optimal dosages on original and deslimed colemanite samples. The optimal or acceptable dosages were found to be 1500 g/Mg for CA927, 1500

g/Mg for OMC111, 2000 g/Mg for OMC5050P, and 500 g/Mg for OMC5020. The reagent OMC5020 was found to be ineffective for colemanite flotation, but the rest of the collectors tried were determined to be almost as efficient as the other collectors.

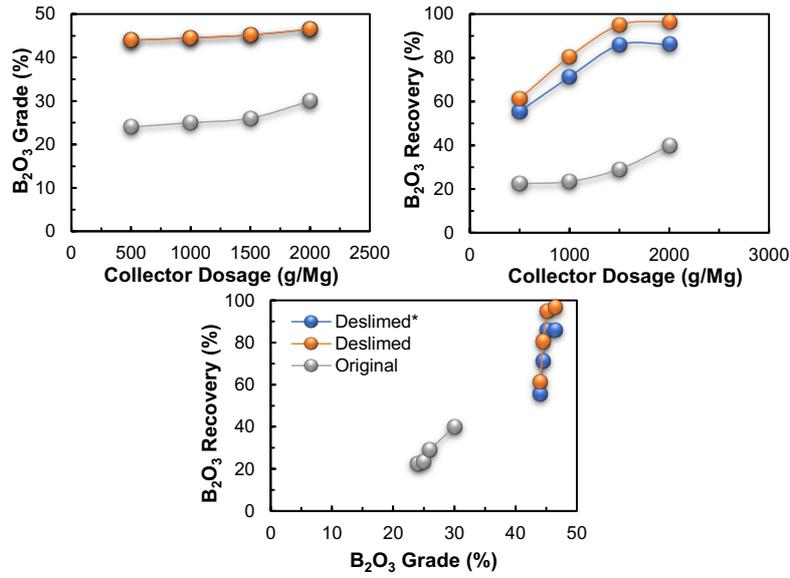
Table 6. Batch flotation test results of the previous work (Modified from Özkan, 1994)

Trials with Various Collectors at Optimal Dosages without Desliming Stage									
Products and Conditions	Concentrate			Tailing			Feed		
	W (%)	G (%)	R (%)	W (%)	G (%)	R (%)	W (%)	G (%)	R (%)
AP825 2000 g/Mg	48.07	31.07	39.44	51.93	44.17	60.56	100.0	37.87	100.0
AP825 1500 g/Mg + Kerosene 500 g/Mg	36.72	24.51	23.56	63.28	46.15	76.44	100.0	38.20	100.0
CA927 1500 g/Mg	42.26	26.03	29.09	57.74	46.43	70.91	100.0	37.81	100.0
OMC111 1500 g/Mg	46.74	28.12	34.98	53.26	45.88	65.02	100.0	37.58	100.0
OMC5050P 2000 g/Mg	44.74	29.19	33.01	55.26	47.95	66.99	100.0	39.56	100.0
OMC5020 500 g/Mg	26.01	24.54	16.31	73.99	44.27	83.69	100.0	39.14	100.0
Trials with Various Collectors at Optimal Dosages with Desliming Stage									
Products and Conditions	Concentrate			Tailing			Feed		
	W (%)	G (%)	R (%)	W (%)	G (%)	R (%)	W (%)	G (%)	R (%)
AP825 2000 g/Mg	95.94	48.92	98.35	4.06	19.39	1.85	100.0	47.72	100.0
AP825 1500 g/Mg + Kerosene 500 g/Mg	94.65	47.89	97.53	5.35	21.43	2.47	100.0	46.47	100.0
CA927 1500 g/Mg	92.82	45.18	95.00	7.18	30.76	5.00	100.0	44.14	100.0
OMC111 1500 g/Mg	71.17	47.61	72.58	28.83	44.40	27.42	100.0	46.68	100.0
OMC5050P 2000 g/Mg	94.32	47.04	96.87	5.68	25.22	3.13	100.0	45.80	100.0
OMC5020 500 g/Mg	41.39	45.40	40.98	58.61	46.18	59.02	100.0	45.86	100.0
SLIME FRACTION (-0.020 mm)	15.72	25.12	8.94						
Trials with Various Collectors at Optimal Dosages with Slimes taking into account									
Products and Conditions	Concentrate			Tailing			Feed		
	W (%)	G (%)	R (%)	W (%)	G (%)	R (%)	W (%)	G (%)	R (%)
AP825 2000 g/Mg	80.86	48.92	89.56	3.42	19.39	1.50	100.0	44.17	100.0
AP825 1500 g/Mg + Kerosene 500 g/Mg	79.77	47.89	88.60	4.51	21.43	2.24	100.0	43.12	100.0
CA927 1500 g/Mg	78.23	45.18	85.88	6.05	30.76	4.52	100.0	41.15	100.0
OMC111 1500 g/Mg	59.98	47.61	65.96	24.30	44.40	24.92	100.0	43.29	100.0
OMC5050P 2000 g/Mg	79.49	47.04	87.88	4.79	25.22	2.84	100.0	42.55	100.0
OMC5020 500 g/Mg	34.88	45.40	37.18	49.40	46.18	53.55	100.0	42.60	100.0

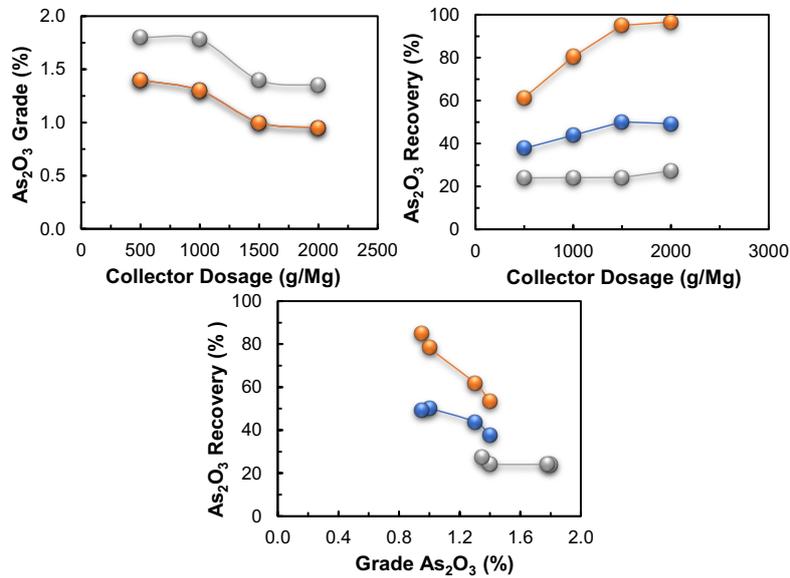
W: Weight; G: Grade  $B_2O_3$ ; R:  $B_2O_3$  Recovery

In this assessment, although AP825, a sodium alkyl sulphonate has been known to be a very effective collector for colemanite for many years by several researchers as shown in Table 2, an unconventional flotation reagent, CA927, sulphosuccinamate (anionic type) was chosen as an alternative in order to see its floating effectiveness after desliming for comparison. Flotation feed samples were coded as original, deslimed, and deslimed\* for comparison. Here, the original means all feed at  $-210 \mu\text{m}$ , deslimed means at  $-210+20 \mu\text{m}$  after sieving the original feed with a  $20 \mu\text{m}$  aperture sieve before flotation, and finally deslimed\* means as same as deslimed feed, but  $-20 \mu\text{m}$  fraction was taken into account in total mass and target&gangue assays before evaluation of the final flotation recovery data. Fig. 6 shows a detailed comparison of batch flotation results according to different feeding and desliming strategies and collector dosages against grade and recovery data including  $B_2O_3$ ,  $As_2O_3$ ,  $Fe_2O_3$ ,  $Al_2O_3$ , and  $SiO_2$  percentages altogether.

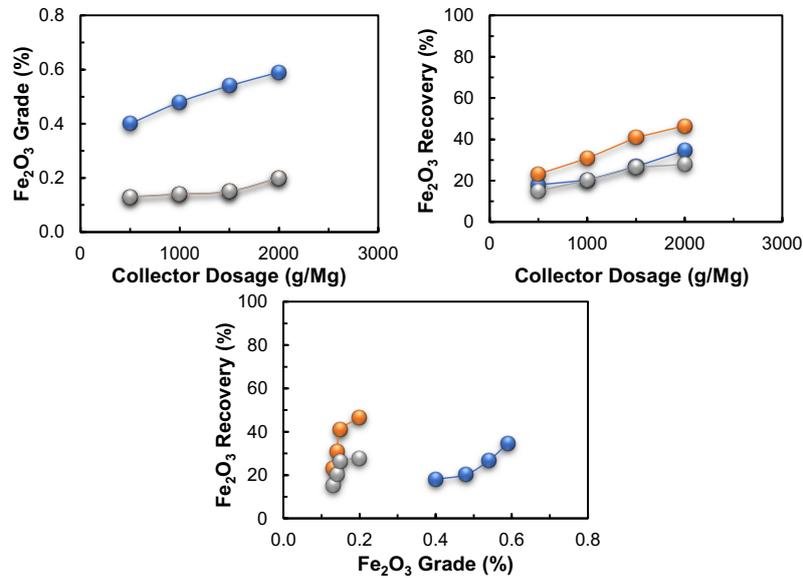
Additionally, the selectivity of  $B_2O_3\%$  as target grade against the gangue contents such as  $As_2O_3$ ,  $Fe_2O_3$ ,  $Al_2O_3$ , and  $SiO_2\%$  assays was investigated as given in Fig. 7 as separate plots.



(a) In terms of grade B<sub>2</sub>O<sub>3</sub>% and % recovery data



(b) In terms of grade As<sub>2</sub>O<sub>3</sub>% and % recovery data



(c) In terms of grade Fe<sub>2</sub>O<sub>3</sub>% and % recovery data

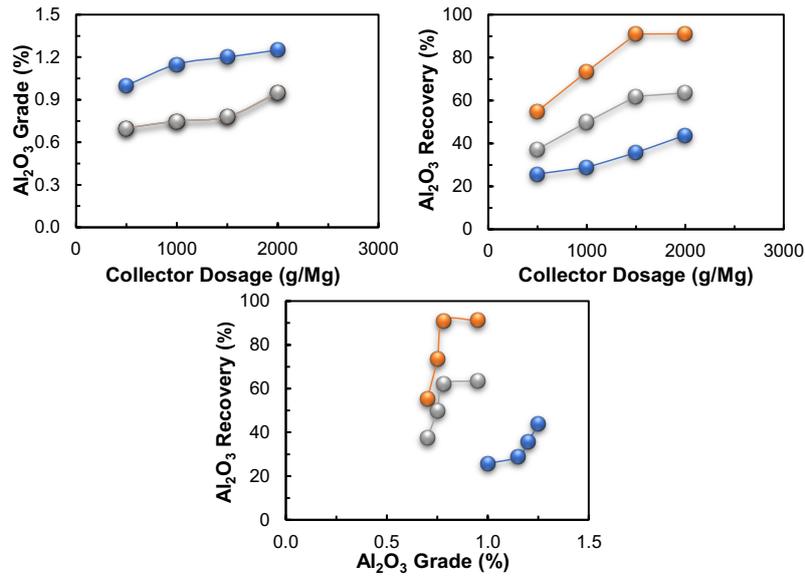
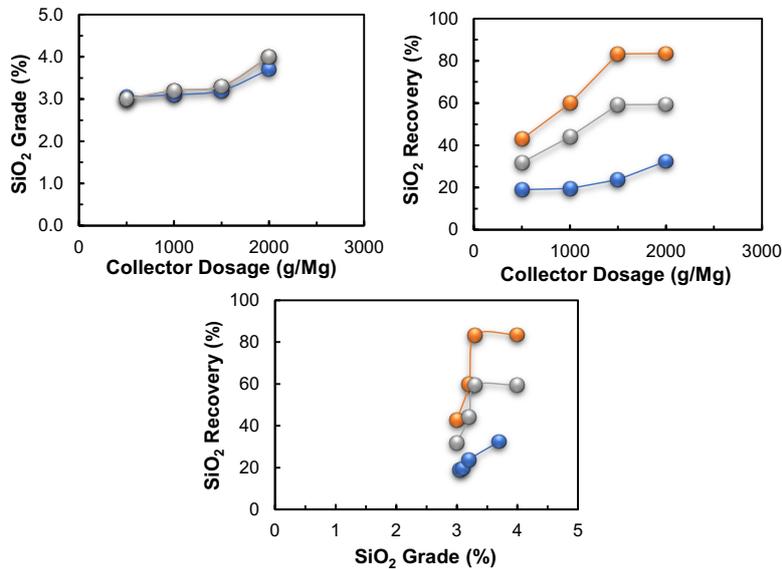
(d) In terms of grade Al<sub>2</sub>O<sub>3</sub>% and % recovery data(e) In terms of grade SiO<sub>2</sub>% and % recovery data

Fig. 6. The assessment of batch flotation results according to grade and recovery data

This study shows the necessity of controlling optimum grinding time for targeted liberation particle size, the importance of the desliming process in order to avoid losses of valuable components in fines, and the consideration of gangue components in all products at all particle size fractions during colemanite flotation when evaluating the overall recovery. The following outcomes can be summarized from the current assessment and comparison of the previous and present material characterization and batch flotation data for representative colemanite samples from the Hisarcik region at the Emet borate deposits of Türkiye:

- Visual observation, microscopic investigations, and chemical analysis results of the feed and flotation products from previous and present work showed B<sub>2</sub>O<sub>3</sub>, As<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, and SiO<sub>2</sub> phases, and the current X-ray Diffraction and Mineral Liberation Analysis results proved the existence of those components except arsenic minerals. There is also a very small portion of unknown phases inside the feed sample from those analyses.
- Although the use of controlled dry grinding of the samples with a rod mill for 15 min period was determined as sufficient for obtaining optimal liberation at -210 μm, newly constructed particle

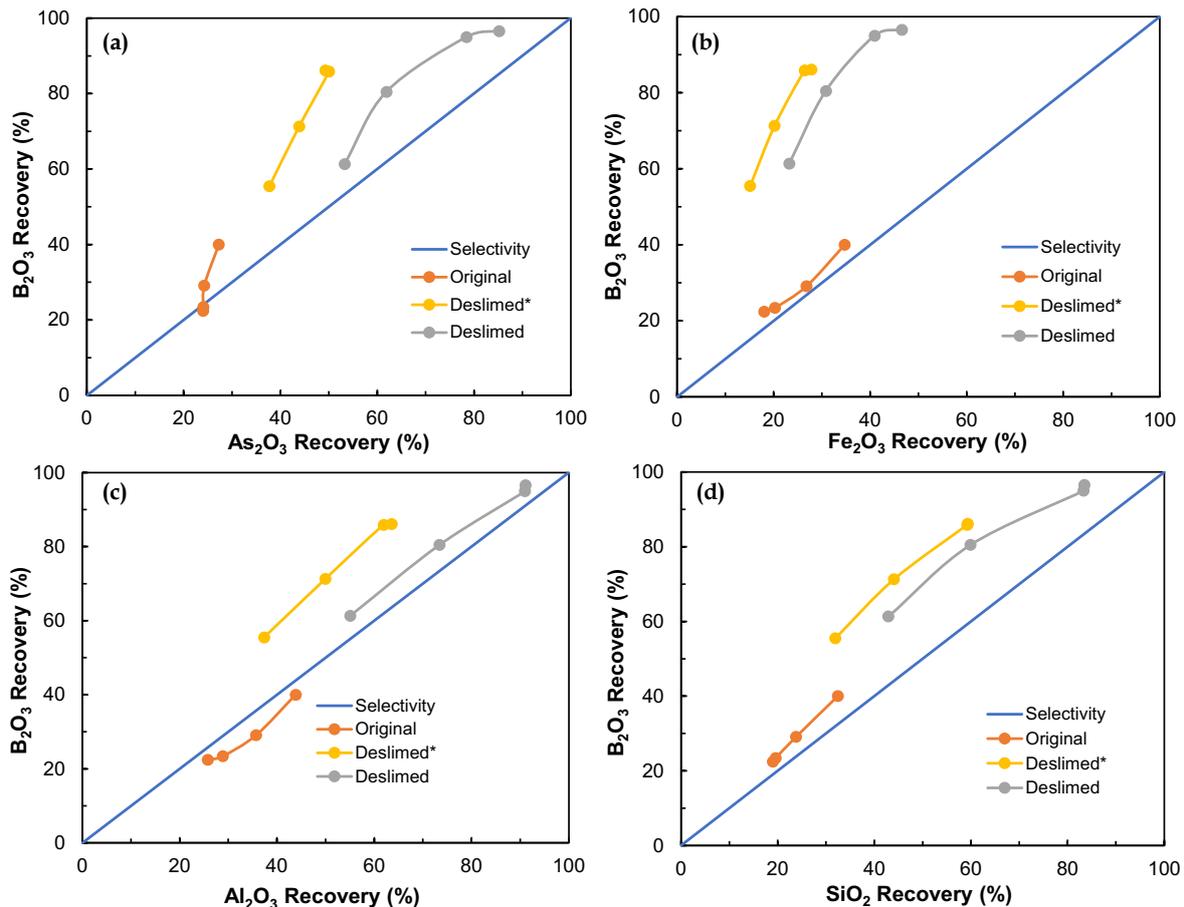


Fig. 7.  $B_2O_3$ % Selectivity against gangue assays during colemanite batch flotation (a)  $As_2O_3$  (b)  $Fe_2O_3$  (c)  $Al_2O_3$ , and (d)  $SiO_2$

size distribution graphs in this article show even 10 min of grinding would be more relevant, especially if the slimes would be ultimately problematic for further flotation tests.

- Re-constructed cumulative undersize (CUS) grade and distribution graphs showed that the desliming process is certainly required and relatively effective as much of the gangue mineral components could be removed easily at  $-20\ \mu m$  before flotation. However, desliming might also cause  $B_2O_3$  losses in slimes, and this time the final mass and assay data should be assessed for further grade-recovery calculations.
- Although the previous work was mainly dependent upon  $B_2O_3$  recovery and grade data of the products, the current evaluations were conducted according to not only  $B_2O_3$  but also  $As_2O_3$ ,  $Fe_2O_3$ ,  $Al_2O_3$ , and  $SiO_2$ % assays. This assessment showed that if the removed slimes is taken into account for the whole processes, the final data would be different from the previous evaluations, such as mass loss of 15% at a grade of 25%  $B_2O_3$  would cause the reduction in recovery up to 10% in final products.
- Although AP825, a sodium alkyl sulphonate has been known to be a very effective collector for colemanite for many years by several researchers, the current assessment was performed by using an unconventional flotation reagent, CA927, sulphosuccinamate (anionic type) as an alternative in order to see its floating effectiveness. Despite the fact that this collector seemed desirable after desliming for the targeted  $B_2O_3$ % content, selectivity against gangue constituents, such as  $As_2O_3$ ,  $Fe_2O_3$ ,  $Al_2O_3$  and  $SiO_2$  in the current conditions cannot support this idea.

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