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Synopsis

Application of magnetic separation was investigated for the enrichment of colemanite ores by removing the magnetic gangue fraction. The detailed XRD analysis of the ore has indicated that the gangue fraction is composed predominantly of the iron-bearing silicates such as smectite and, to a lesser extent, illite. Magnetic susceptibility measurements of the colemanite and gangue samples have proved that colemanite is diamagnetic in character whereas gangue fraction is weakly paramagnetic to allow the removal of this fraction by magnetic separation. Magnetic separation of the crushed and sized ore samples (-5 mm +75 µm) was performed using a high intensity permanent magnetic separator. Under the test conditions a colemanite concentrate (41.29 wt.% B2O3) was found to be produced from the crushed ore (30.76 wt.% B₂O₃) at a recovery of 96.76%. These findings suggest that magnetic separation has great potential as an alternative process for the production of a concentrate of commercially sufficient grade.

Introduction

In recent years, the industrial use and importance of boron compounds has further increased with the potential application of boron as hydrogen carrier in the form of BH₄ in the exploitation of hydrogen as clean energy. Boron compounds can be produced from a variety of boron minerals and boronrich brines. Colemanite (Ca₂B₆O₁₁.5H₂O), ulexite (NaCaB₅O₆(OH)₆.5H₂O) and borax $(Na_2B_4O_5(OH)_4.8H_2O)$ are the most important boron minerals of commercial interest. Colemanite is extensively used in the production of boric acid and preferentially in the textile and glass industries where sodium content is often undesirable¹. Turkey is the largest producer of colemanite concentrates and boron compounds.

In Kestelek plant (Turkey), the colemanite ore produced from open pits is processed to produce saleable colemanite concentrates of 90.000 ton per annually. Processing of the ore (25–35 wt.% B_2O_3) involves size reduction, screening, washing, scrubbing and handsorting stages to remove clay minerals and hence to upgrade the B_2O_3 content of the ore to 38–45 wt.%. The treatment process is

relatively inefficient since a large amount of tailings is produced with significant boron losses (5-15 wt.% B₂O₃) and waste management problems associated with the boron release to the environment. Development of more cost-effective, environmentally acceptable and simple processes for the upgrading of the ore is, therefore, required to reduce the boron losses to the tailings. Several alternative processes including attrition-scrubbing/classification^{2,3}, electrostatic separation4, ultrasonic pretreatment5,6, thermal decompositiondecrepitation7-13, flotation14-17 and leaching18-22 were extensively studied to recover colemanite from the ore and the tailings. None of these processes apart from attritionscrubbing followed by classification has been further developed due to their inherent shortcomings. To illustrate, slime coating was identified to be the most severe problem, adversely affecting flotation recoveries whereas the thermal treatment methods such as decrepitating are energy intensive. Although aqueous processes using inorganic and organic acids and gas-saturated solutions are attractive, they suffer from high reagent consumption and expensive solid/liquid separation.

Magnetic separation is one of the most extensively used separation methods in the mineral processing applications²³⁻²⁵. Weakly magnetic minerals with low susceptibilities ($<5 \times 10^{-6}$ m³ kg-1)²⁶ could be readily recovered by high intensity magnetic separators, which can generate high magnetic fields (>0.8 Tesla) and field gradients in the orders of 50–500 T m-1. Flores *et al.*²⁷ reported that, except for ulexite, which had weakly magnetic properties, other boron

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minerals including colemanite, hydroboracite and borax showed diamagnetic character. Clay minerals including nontronite, biotite, illite and chlorite are often present as gangue phase in most boron ores²⁸. The iron content of these clay minerals is of practical importance since it largely affects their magnetic properties, i.e. the magnetic susceptibility of clay minerals tend to increase with their iron content²⁹. In this respect, if the paramagnetic susceptibilities of clays are sufficiently high, i.e. they can respond to high magnetic fields³⁰, they can then be separated from the diamagnetic boron minerals such as colemanite³¹⁻³².

In this study, the potential application of a high gradient dry rare-earth permanent magnetic roll separator for the recovery of colemanite from the ore was studied. Effect of particle size on the separation of colemanite from the gangue minerals was shown. The mineralogical properties of the ore with particular reference to clay minerals present were examined in some detail. The magnetic susceptibilities of colemanite and gangue present in the ore were also determined prior to magnetic separation tests.

Materials and methods

Ore sample

The colemanite ore sample used in this study was obtained from Kestelek plant in Turkey. The sub-samples were prepared from the bulk sample for the chemical and mineralogical analyses. The mineralogical analysis of the ore using a Rigaku DMAXIIC X-ray difractometer revealed that the ore was composed predominantly of colemanite as the only boron phase detected (Figure 1). The main gangue minerals were identified to be calcite (CaCO₃), dolomite (CaMg(CO₃)₂) and clay minerals including smectite and illite (K_x (Al,Mg)₂(Si,Al)₄O₁₀(OH)₂.nH₂O). Clay fraction of the ore was separated using the procedure proposed by Gündoğdu and Yılmaz³³ for the detailed characterization of clay

minerals. The XRD analyses of the clay fraction obtained (Figure 1) showed that smectite minerals occur as trioctehedral smectite of nontronite

 $(Na_{0.33}Fe_2(Si,Al)_4O10(OH)_2$. $nH_2O)$ and dioctehedral smectite of montmorillonite

((Na,Ca) $_{0.33}$ (Al,Mg) $_2$ Si $_4$ O $_{10}$ (OH) $_2$.nH2O) $^{34-36}$. It should be noted that the XRD analyses were performed in duplicate to ensure the identification of the clay phases present. The chemical analysis of the ore sample using a Jeol X-ray fluorescence (XRF) indicated that the ore sample contained 30.65 wt.% B $_2$ O $_3$ and 2.1 wt.% Fe $_2$ O $_3$ (Table I).

A number of size fractions from the ore sample were prepared by crushing the ore down to 5 mm followed by wetsieving. Each fraction was dried, weighed and analysed for B_2O_3 content, as shown in Table II. The volumetric titration method using mannitol and NaOH was used to determine B_2O_3 contents of samples³⁷. The chemical analysis of the finest fraction (-75 μ m) revealed that the clay minerals and other fine grained phases were concentrated in this fraction, which has the lowest B_2O_3 content of only ~3% amounting to

Table I								
Chemical composition (wt%) of the colemanite ore								
Components	%	Components	%					
B ₂ O ₃	30.65	P ₂ O ₅	0.04					
SiO ₂	18.65	MnO	0.04					
Al ₂ O ₃	3.91	Cr ₂ O ₃	0.02					
Fe ₂ O ₃	2.10	Ва	0.03					
MgO	3.81	Sr	0.34					
CaO	21.15	LOI	14.51					
Na ₂ O	0.35	Tot C	2.71					
K ₂ O	0.98	Tot S	0.53					
TiO ₂	0.15	Total	99.97					

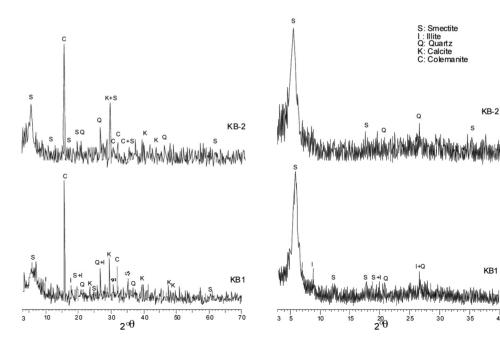


Figure 1—XRD profile of the ore (left) and the clay fraction isolated from the ore sample (right) in replicate (top and bottom)

 $\sim 1.4\%$ of B₂O₃ present in the ore. This was exploited in that the removal of the -0.075 mm fraction would allow the recovery of 98.62 wt.% B₂O₃ contained in the ore corresponding to a 14.15% reduction in mass. Therefore, only the size fractions coarser than 0.075 mm were used in the magnetic separation tests.

Determination of magnetic susceptibility of the samples

A number of the ore samples, which are rich in colemanite and gangue minerals, were collected from the bulk sample. These samples were then analysed for their purity by determining their boron and iron contents prior to the measurement of their magnetic susceptibilities. It was aimed to evaluate the dependence of magnetic susceptibility on the content of B₂O₃ and Fe of the samples and, correspondingly, to characterize the relative magnetic properties of colemanite and gangue (clay fraction in particular). These measurements formed the basis for the magnetic separation tests.

Magnetic susceptibilities of the air-dried granular samples were determined using the low intensity alternating magnetic field (80 A m⁻¹) MS2B dual frequency sensor (operating at 0.465 kHz) attached to a Bartington[©] MS2 system. The resolution of the system is 0.1×10^{-5} [SI] units allowing the measurement of diamagnetic (negative) values38.

Magnetic separation tests

A laboratory type Permanent Magnetic Roll Separator (PERMROLL®) was used in the magnetic separation tests. The magnetic roll (Φ72 mm), manufactured from Sm-Co magnetic discs can generate high magnetic fields (up to 1.6 Tesla) and field gradients (up to 300 T.m-1) on the roll surface. Prior to the tests, the strength of magnetic field on the roll surface of the separator was measured to be 1 100 mT using a Digital Gauss/Tesla Meter (Unilab), which uses a small Hall probe mounted on a rotatable ball and socket head. Furthermore, preliminary test runs were performed to determine the operating conditions by adjusting feed rate

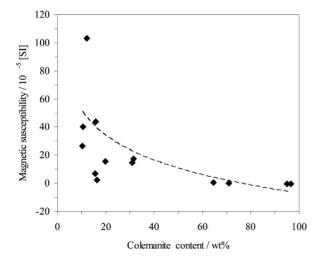


Figure 2-Dependence of magnetic susceptibility on colemanite content of the samples

(50–150 gram per minute), roll speed (50–180 rotate per minute) and splitter angle (5–20°). Magnetic separation tests were carried out under the fixed operating conditions of 5° for splitter angle, 50 gram per minute for feed rate and 60 rotate per minute for roller speed.

A number of narrow size fractions were prepared from the ore sample (Table II). In view of the fact that the drymagnetic separation is not effective for the particles finer than 75 um²⁶ and more than 98.6 wt.% of B₂O₃ was distributed in the fractions coarser than 75 µm, only the coarse size fractions (+75 µm) were subjected to magnetic separation tests. The tests for each fraction were performed in three stages. In the first stage, the sample was fed to the separator and a colemanite concentrate was produced as the non-magnetic fraction. The concentrates produced were then cleaned further in the following two stages of the magnetic separation tests. The magnetic fractions from each stage of the operation were combined, to form the final tailings for each size fraction. On the completion of the magnetic separation tests, the concentrate and the tailings products were weighted and analysed for their B₂O₃ contents.

Results and discussion

Magnetic susceptibility of the samples

Figure 2 and 3 illustrate magnetic susceptibilities of a number of samples as a function of their contents of colemanite and Fe₂O₃. It should be noted that the measured values are the magnetic susceptibility of the bulk sample used in the measurement, which is intimately determined by the quantity and the magnetic properties of the individual mineral phases present. In this regard, the Fe₂O₃ content of the sample is the indicative of the purity of colemanite and the quantity of iron-bearing phases such as clay minerals. The magnetic susceptibility of the samples appeared to be closely related with their iron content in such a way that the magnetic susceptibility tended to increase with increasing Fe_2O_3 content (Figure 3), i.e. the increase in the mass fraction of paramagnetic iron-bearing silicate minerals, e.g.

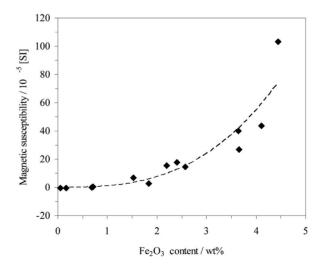


Figure 3-Dependence of magnetic susceptibility on Fe₂O₃ content (as an indicative of quantity of the clay phases) of the samples

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smectite³⁹. The studies on the paramagnetic properties of the silicate minerals including smectite group minerals demonstrated that the paramagnetic characteristics of these minerals were closely associated with their iron content and, the oxidation state of iron and the distribution of iron in the structure⁴⁰⁻⁴¹. It was also reported that additional magnetic phases naturally present in the clay fraction can also increase the magnetic susceptibility of layered silicates⁴².

The magnetic susceptibilities of the samples with high colemanite contents (≥63 wt.%) were determined to be consistently low ($\leq 5.2 \times 10^{-6}$ [SI]) (Figure 2). The increasingly negative magnetic susceptibilities measured for the samples that contained high levels of colemanite, i.e. increasing the purity of colemanite resulted from the diamagnetic characteristic of colemanite in accordance with the previous reports²⁷. It can be inferred from these findings that the diamagnetic colemanite can be separated suitably from the paramagnetic silicate fraction present in the ore, which can be readily captured when a sufficiently high magnetic field is applied. Therefore, in the following section, the enrichment of the colemanite ore by high-intensity magnetic separation was examined.

Enrichment of colemanite from the ore using highintensity magnetic separation

The response of a particle to an applied field is driven by the magnitude of magnetic force (F_m) (Equation [1]) acting against the competing forces: a vertically-downward gravitational force (F_g) (Equation [2]), a radially-outward centrifugal force (F_c) (Equation [3]) and friction force $(F_f)^{26}$. The net resulting force is responsible for the relative motion of the particle in the applied magnetic field where the magnetic force acting on the particle should be sufficiently high. The magnitude of magnetic force generated on the

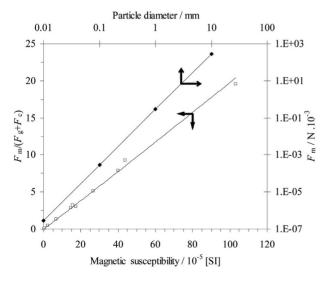


Figure 4-The hypothetical plots of mass magnetic susceptibility versus magnetic force per competing forces (gravitational and centrifugal forces) ((Fm/(Fg+Fc) for the particles, and particle size versus magnetic force at a magnetic susceptibility of 1.03x10-7 [SI] (the calculations were based on the operating conditions of the magnetic separator used, B: 1.1 Tesla, ΔB: 300 T/m, roll speed: 100 rpm, roll diameter: 72 mm)

particle is controlled by the properties of the particle, i.e. magnetic susceptibility and mass/size of the particle, and the applied magnetic field, i.e. field intensity and gradient. When two particles of the same size, but, different magnetic susceptibilities are exposed to the same magnetic field, magnetic forces generated on both particles will be different allowing the separation of these particles⁴³.

$$F_{\rm m} = (\chi_{\rm p} - \chi_{\rm f}) \ m_{\rm p} B \Delta B / \mu_{\rm o} \qquad (\chi_{\rm f} << \chi_{\rm p})$$
 [1]

$$F_{g} = (\rho_{p} - \rho_{f}) V_{p} g$$
 [2]

$$F_{\rm c} = (\rho_{\rm p} - \rho_{\rm f}) \ \omega^2 V_{\rm p} r$$
 [3]

where χ_f is the magnetic susceptibility of fluid (zero for air); χ_p is the specific mass magnetic susceptibility; μ_0 is the permeability of vacuum: B is the magnetic flux density: ΔB is the magnetic field gradient; m_D is the particle mass; ρ_f and ρ_D are the density of the medium and particle, respectively; g is the acceleration by gravity; *r* is the radial position of the particle and ω is the angular velocity.

Figure 4 illustrates the response of the particles of different size to an applied magnetic field based on the theoretical calculations using the operating data of the magnetic separator used in the current study. It can be seen that the particles of different size (0.01–10 mm) would respond only to the applied magnetic field, i.e. $F_{\rm m}/(F_{\rm g}+F_{\rm c})$ is greater than unity, when they have a sufficiently high magnetic susceptibility of >5 \times 10-5 [SI] (Figure 4). It can be inferred from these calculations and the earlier findings (Figure 3) that, once liberated, the iron-bearing silicate phases present in the ore could be readily separated using the roll magnetic separator allowing the enrichment of colemanite. It is also pertinent to note that the effectiveness of magnetic separation would tend to be adversely influenced by decreasing the particle size. This can be attributed to the decrease in the intensity of the driving magnetic force acting on the finer particle (Figure 4). The competing forces, hydrodynamic drag force in particular, become increasingly more effective with decreasing the particle size below 50 um and, therefore, a particle size of 75 µm is often considered as the threshold limit in dry-magnetic separation processes where the sizing of the material into narrow size fractions is also required to improve the efficiency of magnetic separation26.

The results of three-stage magnetic separation tests on the various size fractions obtained from the ore sample are presented in Table II. Colemanite concentrates of 35.76-45.65 wt.% B₂O₃ in grade were produced at the recoveries of 77.15–99.63%. Despite a relatively high recovery of 99.3%, the the lowest grade of concentrate (35.76 wt.% B₂O₃) was obtained from the +4.750 mm fraction of the ore. On the other hand, the recovery of B₂O₃ from the finest fraction (-0.105 + 0.075 mm) of the ore tested was only 77.15 wt.% of B_2O_3 . The separation efficiency (Equation [4])²⁹ for each size fraction was also evaluated, as shown in Figure 5. It was found that separation efficiency tended to increase with decreasing particle size from +4.75 mm to -1.675 + 0.853mm fraction. This could result from the insufficient liberation of paramagnetic fraction of the ore at these coarse sizes (+0.853 mm).

$$SE = Rd - Rg$$
 [4]

Where SE is the separation efficiency; Rd and Rg are the recoveries of colemanite and gangue to the concentrate, respectively29.

Table II								
The results of magnetic separation tests on the different size fractions of the ore								
Size range	Product	Mass	Mass in	B ₂ O ₃ content	Colem. content	B ₂ O ₃ dist.	B ₂ O ₃ dist.	
mm		wt%	total wt%	wt%	wt%	wt%	in total wt%	
+4.750	Conc.	88.78	28.32	35.76	70.38	99.30	33.49	
	Tails	11.22	3.64	1.97	3.88	0.70	0.23	
	Head	100.00	32.96	31.97	62.92	100.00	33.72	
-4.750	Conc.	89.98	19.81	44.89	88.35	99.63	28.91	
+1.675	Tails	10.02	2.20	1.49	2.93	0.37	0.10	
	Head	100.00	22.02	40.57	79.85	100.00	29.01	
-1.675	Conc.	77.63	6.18	45.65	89.84	96.25	9.17	
+0.853	Tails	22.37	1.78	6.17	12.14	3.75	0.35	
	Head	100.00	7.96	36.81	72.45	100.00	9.52	
-0.853								
+0.500	Conc.	77.29	5.95	43.66	85.93	98.13	8.44	
	Tails	22.71	1.74	2.82	5.55	1.87	0.16	
	Head	100.00	7.69	34.38	67.66	100.00	8.60	
-0.500	Conc.	75.88	8.11	45.58	89.71	96.58	12.02	
+0.210	Tails	24.12	2.58	5.07	9.98	3.42	0.43	
	Head	100.00	10.69	35.80	70.46	100.00	12.45	
-0.210	Conc.	73.69	1.49	45.20	88.96	90.82	2.19	
+0.152	Tails	26.31	0.53	12.79	25.17	9.18	0.23	
	Head	100.00	2.03	36.67	72.17	100.00	2.42	
-0.152	Conc.	73.12	1.21	45.61	89.77	95.21	1.79	
+0.105	Tails	26.88	0.44	6.23	12.26	4.79	0.09	
	Head	100.00	1.65	35.02	68.92	100.00	1.88	
-0.105	Conc.	40.32	0.53	43.58	85.77	77.15	0.75	
+0.075	Tails	59.68	0.78	8.72	17.16	22.85	0.22	
	Head	100.00	1.31	22.77	44.81	100.00	0.97	
-0.075	Tails	100.00	14.15	3.02	5.94	100.00	1.38	
Total			100.00	30.76	60.54		100.00	

The results for the different size fractions were combined to produce the grade and recovery data for the +0.075 mm ore, as shown in Table III. Figure 6 also shows the plot of cumulative grade/recovery versus oversize fractions. The combined results revealed that the magnetic separation process could be readily exploited to beneficiate the ore with increasing the B₂O₃ content from 30.76 wt.% to 41.29 wt.% corresponding to a recovery of 96.76% and a reduction of 29.4% in mass. Of commercial importance is that the colemanite concentrates are required to contain ≥40 wt.%

In the Kestelek plant, washing followed by hand-sorting of the ore is currently practised as the process route at the

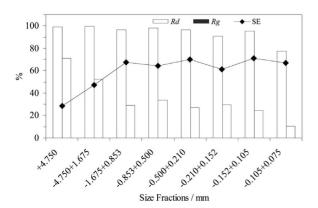


Figure 5—Recoveries of colemanite (R_d) and gangue (R_g) into the concentrates produced as non-magnetic fraction, and separation efficiency (SE) for each size fraction

expense of significant losses to the tailings. More recently, the attrition scrubbing/washing method to reduce losses (-3 mm) into the tailings was developed and incorporated into the beneficiation process in the Kestelek plant. However, the dry processes are most desirable for the treatment of boron ores due to the environmental concerns associated with the release of boron and, hence, the requirement for the treatment of process waters prior to the discharge. The current findings suggest that dry magnetic separation can be exploited as a simple, effective and environmentally acceptable alternative process for the beneficiation of the colemanite ores. Magnetic separation can be also considered as a more practical option than flotation and decrepitation

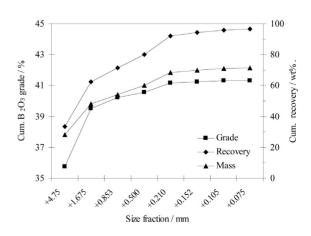


Figure 6-The plots of cumulative recovery/grade/mass vs oversize material for the combined results of magnetic separation tests

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Table III								
Combined results of the magnetic separation tests on the ore								
Product	Mass wt%	B ₂ O ₃ content wt% Colemanite content		B ₂ O ₃ distr. %				
Concentrate	71.6	41.29	81.26	96.76				
Tails	28.4	3.53	6.95	3.24				
Head	100.00	30.76	60.54	100.00				

methods since the application of the former is limited to fine size fractions with the potential problems caused by slime-coating¹⁵⁻¹⁷ and of the latter is hampered by its high cost of thermal treatment and by the undesired decomposition of crystal structure of colemanite⁷⁻¹⁰.

Conclusions

The iron-bearing silicates, i.e. clays, are often present in the colemanite ores. Magnetic susceptibilities of the samples with different contents of colemanite and iron (indicative of the abundance of iron-bearing silicates present) were examined to characterize the magnetic properties of colemanite and gangue fraction. The gangue fraction of the ore appeared to be paramagnetic in character apparently due to the ironbearing clays such as smectite and, to a less extent, illite present in the ore. These findings suggested that the paramagnetic gangue fraction could be readily captured and separated from the diamagnetic colemanite in an applied magnetic field of sufficient intensity. Based on these findings and theoretical calculations, the three-stage magnetic separation tests on the different size fractions obtained from the ore were designed using a Permanent Magnetic Roll Separator. The results of these tests have revealed that the magnetic separation process can be readily applied for the beneficiation of colemanite ores to remove magnetic fraction and, hence, to produce concentrates with a commercially acceptable grade (~41.29 wt.% B₂O₃) an average at high overall recoveries (~96.76%).

Magnetic separation process examined in this study has great potential as a viable and effective alternative for the treatment of colemanite ores to reduce the losses into tailings and to improve the overall process performance. Furthermore, the dry mode of operation of magnetic separation offers further environmental benefits for the control of boron release compared with the other potential wet methods.

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