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Ultrasonic treatment and its applicability for the selective treatment of borax clayey waste sludge

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Abstract: Sludge treatment is one of the most difficult challenges for many industrial plants. In recent years, the use of ultrasound power has received considerable attention as one of the increasing tool for managing of this problem. Power ultrasound enhances chemical and physical changes in a liquid medium through the generation and subsequent destruction of cavitations bubbles. Therefore, the increased friction and stress in solution resultant with cleaning of the solid surfaces and dispersion of bonded/aggregated solid particles. In this study, the basic fundamentals of ultrasonic treatment and process variables were briefly overviewed. The study was also extended with preliminary analyses performed to determine its ability on the extraction of major and trace elements from Kırka Borax Concentrator tailings mainly boron as boric acid and Li as Li reach pregnant solution (PLS). Prior and after the sonification tests, physical, chemical and morphological analyses were carried out to determine the treatment performance. It was found that sonication leads the liberation of boron minerals from sludge and with the help of temperature they are dissolved and re-crystallized on clay substrate during cooling stage. The extraction test performed with these crystals revealed that Li-rich solution (that can be further use as source of lithium carbonate or lithium hydroxide production), and boric acid could be produced with easy and low cost beneficiation method.

Keywords: ultrasonic treatment, boron waste water, separation, sludge treatment

1. Introduction

Minerals are the basic raw materials which contribute to the growth of both industrialized and industrializing countries. However, one of the extremely major problems and key challenge facing the modern world is the shortage of resources and environmental pollution resultant from industrial activities. Especially in mining industry, the increased production depending on the high demand to raw materials causes the depletion of the non-renewable natural resources. This over use also leads to production of more and more solid, liquid or mud-form wastes which induces the destruction on eco-environment (Sanchez et al., 2019).

The most common disposal route for these wastes is land application disposal in landfills or tailing dams. But all of them are suffering with serious drawbacks including possible contamination of farmland by micro-pollutants present in the tailing sludge, high construction and operating cost and loss of beneficiable materials within them (Lua and Caib, 2012). Therefore, there is no doubt that decreasing of the waste amounts by comprehensive utilization methods will have enormous economic and social benefits on the managing of resources shortage and environmental protection. In practice, although several pre-treatment or treatment methods such as precipitation, flotation, filtration are employed before disposal, in some applications, final slurries still suffer from the higher amount of solid part that consist of fine particles. For example, the gravity sedimentation process supported with flocculation is a widely employed method for the separation of solid particles prior the disposal, however, the mixture of fine particles with chemical flocculants becomes industrial waste, and the particles originally targeted for the solid–liquid separation are hard to use as resource (Nakamura et al.,

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2011). Therefore, as an alternative to these methods, in recent years, an ultrasonic treatment method that includes acoustic cavitation and bubble dynamics has gained a wide attention as an effective pretreatment method at lab, pilot and full scale applications (Bekker et al., 1997; Liu et al., 2009).

Sonication basically refers to the application of sound energy at frequencies largely higher than 20 kHz. Ultrasound disruption is more energy efficient and can achieve a higher degree of powder fragmentation, at constant specific energy, than other conventional dispersion techniques. It is a convenient, relatively inexpensive method that is simple to operate and maintain. The excellent performance, good technical (compact) and operational stability make it preferable tool for extensive applications where it is used mainly to break down powders or re-disperse stock suspensions to their nanoscale constituents. Furthermore, different applications of ultrasound technology in water/sludge treatment process such as filtration, turbidity and total suspended solid reduction by gravity settling, algae removal, disinfection process, water softening process and other pollutants removal such as halomethanes and dichloro diphenyl trichloroethane (DDT) have also showed that this technique could be used to improve the water quality without any hazardous emission or chemicals (Doosti et al., 2012).

Boron is a relatively rare element and does not appear on Earth in elemental form but is found as a mineral deposit in the crystalline group known as "Borates" (oxides of boron). They are distinguished by a structural framework formed of polymerized boron-oxygen-hydroxyl tetrahedral or triangles, which are linked to each other by means of cations. The usage of boron or its combined form of borax varies from made of insulation or textile fiberglass, heat resistant glass, detergent, soaps, personal care products, ceramics, agriculture micronutrients to aircraft fuel and hydrogen storage due to their own advantages (Kurama, 2011). The world production of natural and refined borates remains highly concentrated in Turkey and the US. The two countries provide about 80% of the total production. The actual world production of boron was reported as 4.3 Mt (2.1 Mt B_2O_3). Turkey has keep its leading role (55%), and Nord America (US-25%) (Eti Maden, 2018). Sodium borate pentahydrate ($Na_2B_4O_7 \cdot 5H_2O$) is the most commercially important product for the borate industry. Large amounts of this compound are used in the manufacture of fiberglass insulation and sodium perborate bleach. It crushes freely and dissolves readily in water; its solubility and rate of solution increases with water temperature. The Kırka-Eskişehir region is one of the principal borate mineral deposit in Turkey. In this region, mainly tincal (presents with lesser amounts of colemanite and ulexite) ore are mined. The borate layers contain minor amounts of realgar, orpiment, gypsum, celestite, calcite, and dolomite, and the clay partings contain some tuff layers, quartz, biotite, and feldspar. The clay is made up of smectite group minerals and, less frequently, illite and chlorite minerals (Helvacı, 2015; Karapınar, 2017).

In Kırka boron plant, the ore having an average grade of 25% B_2O_3, is processed to produce borax penta and decahydrate powders. The tailings slurry, resultant from the dissolving and crystallization stages that consist of mainly clay minerals, dolomite and considerable amount of borax, is directly sent to the tailing ponds by gravity for natural settling without any treatment. Although, process water is further collected and re-used in the beneficiation process, a large amount of water and large areas of land are tied up by impounding these clayey tailings for a long period of time. This situation causes serious technical and environmental problems in terms of the need for additional ponds and the challenges for the closure and rehabilitation of the tailings ponds. Therefore, an effective tailings disposal method is necessary for the borax clayey tailings regarding to both environmental and technical point of view.

As noted above, ultrasonic cavitation gives rise to extreme local pressure and temperature gradients, as well as shock waves and jet streams that can effectively break down powder agglomerates. However, under certain conditions, the applied acoustic energy can conversely induce particle agglomeration or even lead to the formation of aggregates. Coagulation in ultrasonic fields can occur from enhanced particle-particle interactions due to the increased collision frequency as well as the favorable reduction in free energy that accompanies the resulting reduction in the liquid-solid interface.

Consequently, it can be proposed that ultrasonic disruption is an effective tool for sludge treatment. However, it also be noted that sonication is also complex method depending on the solution characteristics and process variables. Therefore, the aim of the present study is to perform preliminary sonication tests to determine its applicability as pre-treatment method. The study was also extended with preliminary analyses performed to determine its ability on the extraction of major and trace elements from Kırka Borax Concentrator tailings mainly boron as boric acid and Li as Li reach pregnant solution (PLS).

1.1. Ultrasound

Ultrasound is a longitudinal cyclic (sound) pressure wave with a minimum frequency of 20 kHz. This frequency is above the sonic range (20 Hz to 20 kHz) at which humans can hear and below the mega-sonic region (>600 kHz). Based on their frequency, ultrasonic waves can be classified into two main categories, that is, (a) low to medium frequency (20–1000 kHz) waves, which are applied at high power and generally used in industry such as nanotechnology, ultrasonic therapy and sonochemistry; on the other hand, (b) high frequency (2–10 MHz) waves, which are being used at low power intensities for diagnostic applications in medical imaging and non-destructive testing (Tyagi et al., 2014; Poli et al., 2008; Suslick et al., 1986).

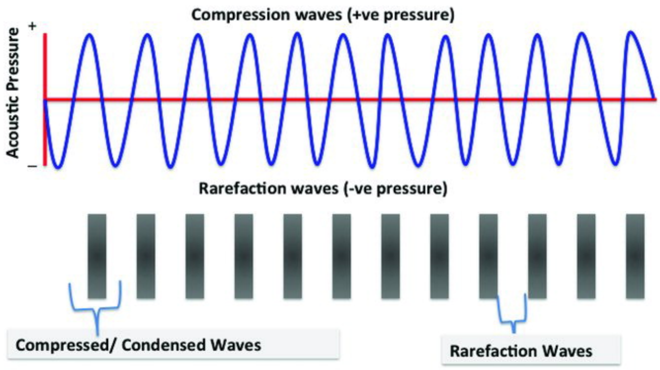

Fig. 1. Compression and rarefaction (expansion) waves of ultra-sonication (Tyagi et al.,2014)

During the ultrasonic process, the sound wave is transmitted as a series of compression and rarefaction cycles through the liquid medium and hence, energy is transmitted by the vibration of the molecules in the environment (Fig. 1). When the negative pressure of the rarefaction cycle exceeds the attractive forces between the molecules of the liquid, a void is formed. This void or cavity in the structure takes in a small amount of vapor from the solution so that it does not totally collapse in the compression cycle, but instead continues to grow in successive cycles to form. This process known as cavitation. The bubbles then collapse during the high pressure cycle (compression) producing a localized shock wave that releases tremendous mechanical and thermal energy. The occurred shock wave generally referred as a common mechanism that contributing to the powder breakage. As described above, during ultrasonic disruption, extremely high local pressures are generated, and high-pressure shockwaves emanate from the location of the bubble. These are capable of causing mechanical damage to the surrounding material. In cases where the bubble is adjacent to a solid surface, a high-velocity liquid jet may shoot through the bubble, impacting and damaging the neighboring cells. When ultrasound energy is applied to powder clusters in suspension, fragmentation can occur either via erosion or fracture. Erosion or "chipping" refers to the detachment of particles from the surface of the parent agglomerates, while fracture or "splitting" occurs when agglomerates partition into smaller agglomerates or aggregates due to the propagation of cracks initiated at surface defects (Fig. 2). These fragmentation effects may occur simultaneously or in isolation, depending on the powder, its environment and the energy levels involved (Taurozzi et al., 2014). The impact of the jets on the solid surface is very strong. Hereby, this can cause production of highly reactive surfaces. Distortions of a collapsing bubble impact on a surface area several times greater than the resonant dimensions of the bubble. Therefore, unlike the agglomeration or de-agglomeration applications, the ultrasound assisted leaching has also been the subject of many research studies in the literature due to the positive effects on dissolution rates, metal recoveries and reduced reagent consumption (Swamy et al., 1995; Luque-Garcia and Luque de Castro, 2003; Chemat et al., 2017).

Furthermore, the high temperatures and pressures produced by acoustic cavitation provide a unique interaction of energy and matter. This resultant with the formation of free radicals and other compounds; in particular, sonication of pure water causes thermal decomposition to H atoms and OH radicals. Both generation of free radicals and later also ability of the production of hydrogen peroxide, has been found that an effective tool for many surface cleaning and flotation applications of particles in literature (Farmer et al., 2000). This topic is well explored in a recent study performed by Hassanzadeh et al., 2021. As a result of the comparative evaluation of wettability measurements, in the presence of constant low-power (60 W) acoustic pretreatment conditions, the authors reported that all three minerals became relatively hydrophilic. In addition, except for quartz, the increase in sonication intensity increases the hydrophilicity. This situation mainly attributed to the formation of hydroxyl radicals.

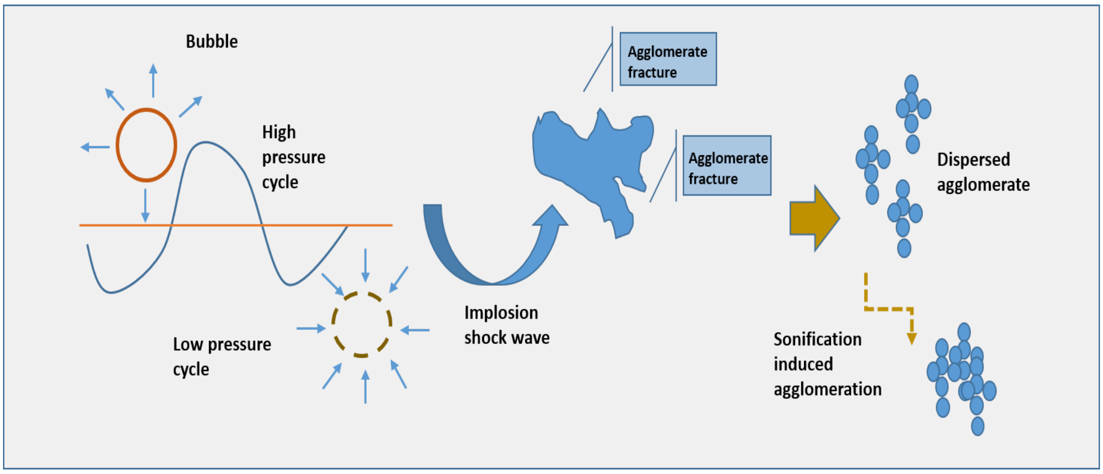

Fig. 2. A schematic illustration of the typical effects of sonication on particle size as a function of delivered sonication energy (Taurozzi et al., 2014)

1.2. Effecting factors

The power density, frequency, specific energy input, irradiation time, the solids concentration and the type of sludge have been proven to be crucial factors that affect the ultrasound processing of sludge. So, it is needed to obtain the optimum power density, frequency and irradiation time to reach an effective cost. Furthermore, cavitation can be initiated by either setting up a tension in the liquid or by depositing energy into it and is accompanied by a number of effects originating from the dynamic behavior of the generated bubbles (Clark and Nujjoo, 2000).

2. Materials and methods

2.1. Tailings used

The tailing slurry used in sonication tests was supplied from the Etibank Kırka Boron works concentration plant in Turkey. The particle size distribution of the sample (performed by Malvern Mastersizer 2000 laser particle size analyzer) was given in Fig. 3. It was found that d_{90} value of sample was 16.215 µm.

The mineralogical phase compositions of the solid part of samples performed with an Panalytical Empyrean brand X-ray diffractometer with nickel filtered Cu Kα radiation are given in Fig. 4. It was found that tailings solid mainly consists of boron minerals (tincalconite), dolomite and minor amount of quartz.

2.2. Sonication tests

Sonication tests were performed within 250 cm³ glass beaker using the Bandelin Sonopuls HD 2200 ultrasonic homogeniser. In experiments, a 200 cm³ of waste solution as received or pre-heated (90 ^{0}C) forms (in order to evaluate temperature effect) was filled into the beaker and continuously sonicated at high and lover-power disruption rates with help of ultrasonic homogeniser. After 20 minutes, the sonication was stopped and then the upper part of solution (group D) was transferred into a secondary beaker via a tube (Fig. 5). The slurries in the both beakers were then allowed to settle of solid particles, decanted, and samples taken from the solid/liquid parts were sent to the analyses. The definitions of the tests performed according to the process variables for the slurries in the first (B) and the second decanted beaker (D) are given in Table 1.

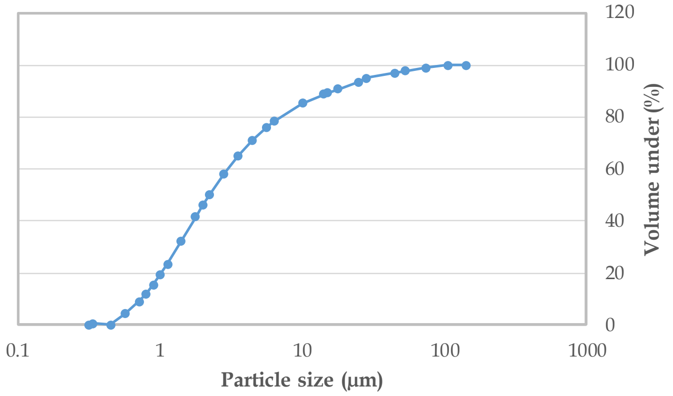

Fig. 3. Particle size distribution of tailing

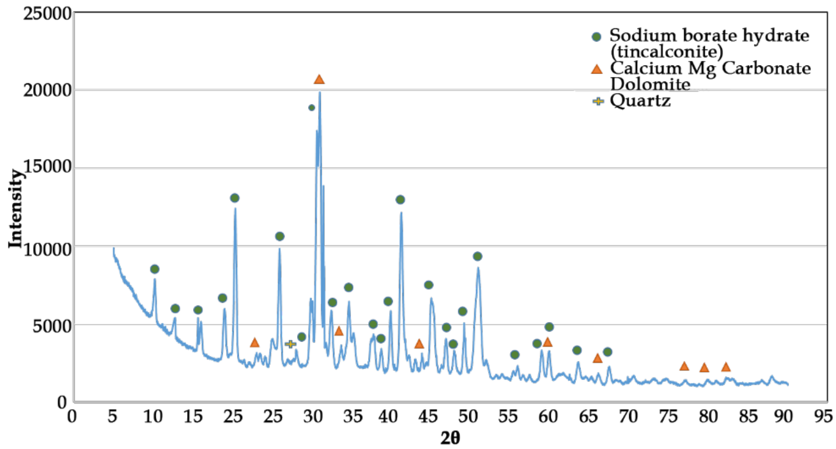

Fig. 4. The phase composition of the solid part of tailing

Table 1. Sonication experiment variables

Run	Specifications
1B	45% power, cold, 25 ^{0}C, pH of 9.90
1D	45% power, cold, 25 ^{0}C, pH of 9.90
2B	90% power, cold, 25 ^{0}C, pH of 9.90
2D	90% power, cold, 25 ^{0}C, pH of 9.90
3B	45% power, hot, 85 ^{0}C, pH of 9.40
3D	45% power, hot, 85 ^{0}C, pH of 9.40
4B	90% power, hot, 85 ^{0}C, pH of 9.40
4D	90% power, hot, 85 ^{0}C, pH of 9.40

*The temperatures given at table indicate the initial solution values

In order to evaluate the effect of sonication, the boron concentration of samples was analyzed by Thermo iCAP RQ ICP-MS Inductively Coupled Plasma Mass Spectroscopy. The morphological analyses of the samples were performed by Hitachi Regulus 8230 Field Emission Scanning Electron Microscope.

3. Results and discussion

3.1. Effect of process variables on boron concentration

The boron concentrations of each run are given at Table 2. Although, the concentration of each run are

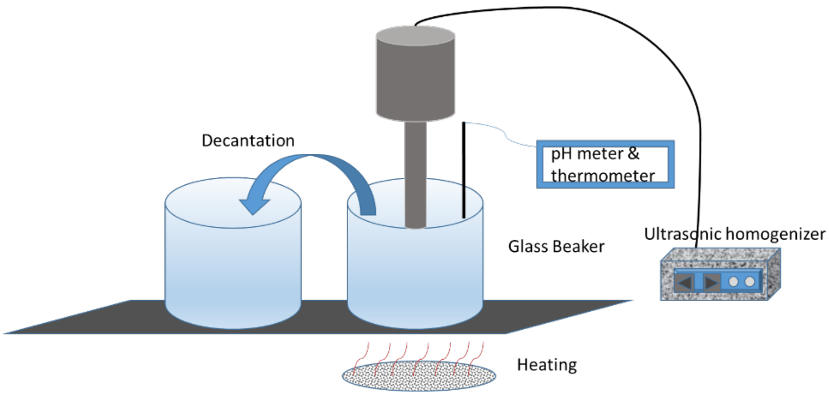

Fig. 5. Sonication experimental set-up

nearly the same, it was found that relatively higher boron concentrations can be obtained at lower power levels (45%) and increased solution temperatures. The polar nature of clay minerals, especially smectites (montmorillonite), allows them to form van der Waals bonded networks of grain clumps, that develop immediately after dispersing stops, and hence leading to the thixotropic behavior of aqueous clay slurries. Therefore, the nearly same boron concentrations for B and D parts can be attributed to movement of dissolved boron ions together with suspended fine and light clay grains or micro flocks from primary beaker to secondary one and still existing of dissolved boron ions in the first beaker. In addition, relatively higher solid content of the waste solution (14% solid) may also be given as reason why clay flocks still available at the first beaker even applied ultrasound power. As reported by Pilli et al (2011), the higher total solid (TS) content (6%) affects interruption of the formation of cavitation bubbles which resultant to attenuation in sonication intensity in the aqueous medium.

Table 2. Sonication test results

Run	Boron Concentration, ppm	Run	Boron Concentration, ppm
1B	7580	3B	9550
1D	7315	3D	8500
2B	6096	4B	8232
2D	5950	4D	7679

3.2. Phase analyses

According to mineralogical phase analyses results given in Fig. 6, it can be proposed that a partly separation of dolomite and the clay product can be obtained by sonication.

The determined high dolomite intensities for B parts and inversely, the presence of increasing smectite phases for the D parts, indicated that the lower part of the solution in the first beaker is consists of mainly dolomite, while the upper part consists of smaller aggregates or single clay grains as dust clusters. As described by Neis (2000), at low ultrasonic frequency (20 kHz), the cavitation bubbles act as mechanical shredders by the formation of water jets in the solution. Hence, by the first impact, the sludge flocks are separated and numbers of single cells are released. While sonication continues, these cells act as nuclei for the formation of new bubbles.

Same conclusion can also be drawn for sonication performed at higher temperatures. However, it was also noted that the phase of boron mineral become more visible due to positive temperature effect on the dissolution and re-crystallization of boron minerals.

3.3. Morphological analyses

The SEM images of the samples given in Fig. 7 supported the previous XRD analyses. The existence of visible boron crystals on the clay surface especially for third and four runs clearly indicated the positive effect of sonication on sludge treatment. As discussed at previous part the sonication caused the disintegration of clay flocks which leads the separation of boron minerals from them and with help of temperature these are dissolved and re-crystallize on clay substrate during cooling stage (Fig. 8).

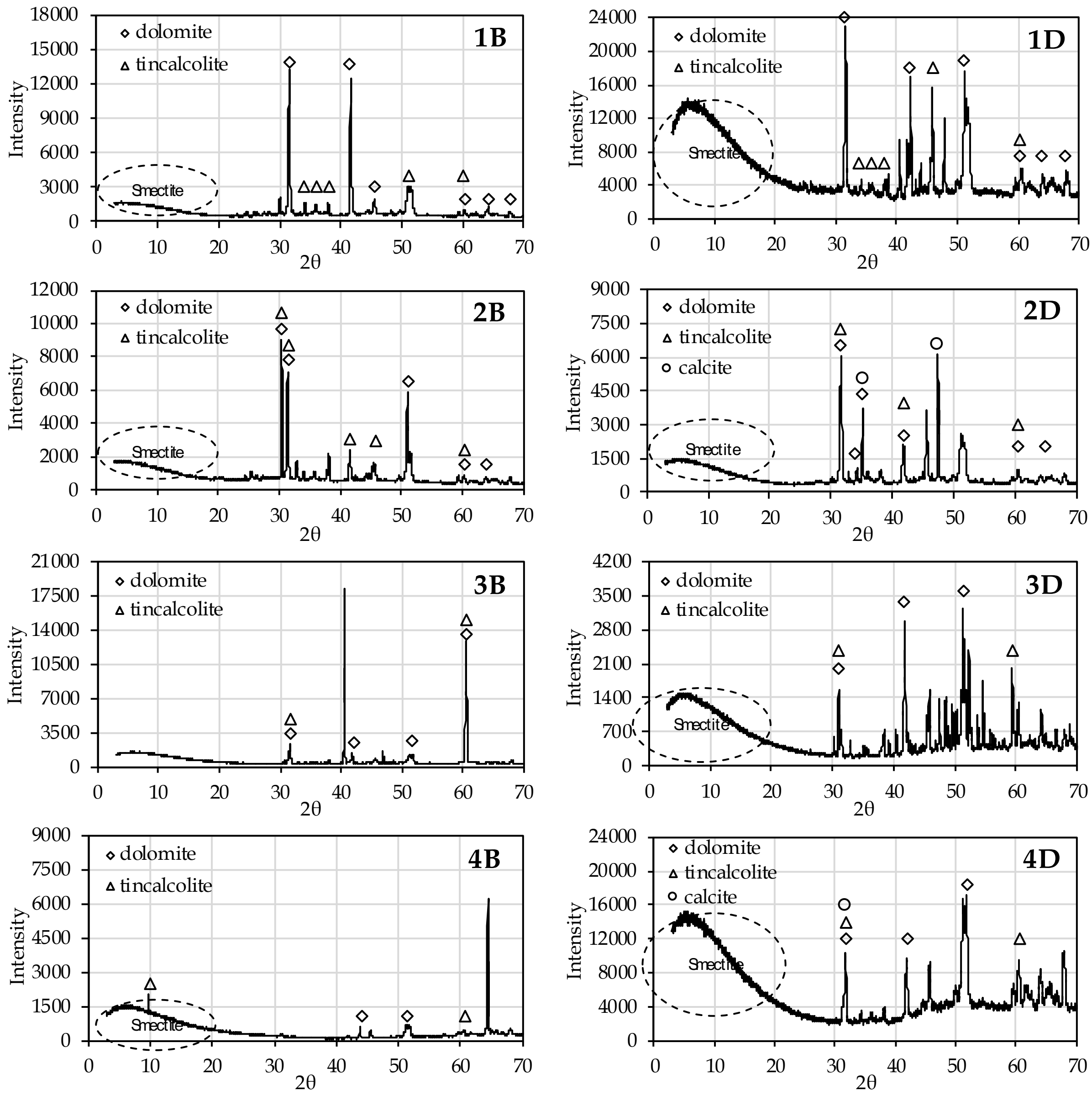

Fig. 6. The X-Ray diffraction pattern of solid samples

The phase comparison of the XRD analyses result of these easily separable crystals (Fig. 9) with the typical phase composition of glassy tincalconite that collected from same area (Fig. 10) is revealed that the phase composition of these crystals are different, mainly consist of boron hydrogen lithium.

3.4. Extraction of lithium from borate crystals

Lithium in lithium-ion batteries is an essential part of electric vehicles which make them quickly becoming better and more economical than internal combustion vehicles. Spodumene, brine from lakes and also secondary material extracted from recycling the battery wastes are the main source for Li that can use lithium-ion batteries. Within these source, spodumene has attracted much interest as a source of lithium because brine sources alone cannot satisfy the increasing demand for lithium. Therefore, in recent years, the number of studies on methods of extraction and production of high purity lithium salts are rapidly increasing. According to recent review published by Salakjani et al., 2021, the beneficiation processes can be categorized into two groups, namely pyrometallurgical and hydrometallurgical processing. For the pyrometallurgical processes, roasting spodumene with a chemical reagent is the main extraction step and generally involves subsequent leaching to separate the lithium from the insoluble material. Among all the reported methods, acid roasting with sulfuric acid is a proven and

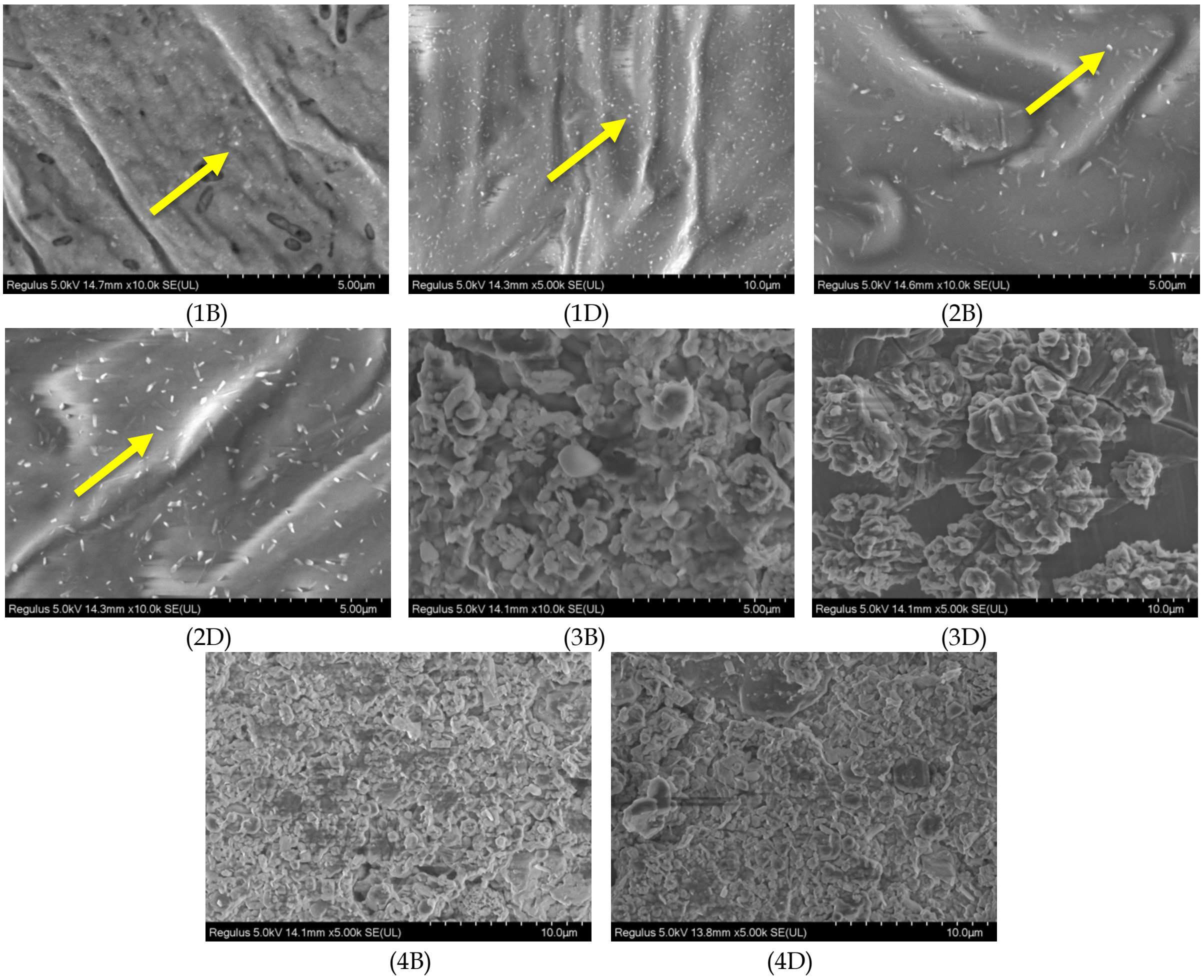

Fig. 7. The SEM images of the samples

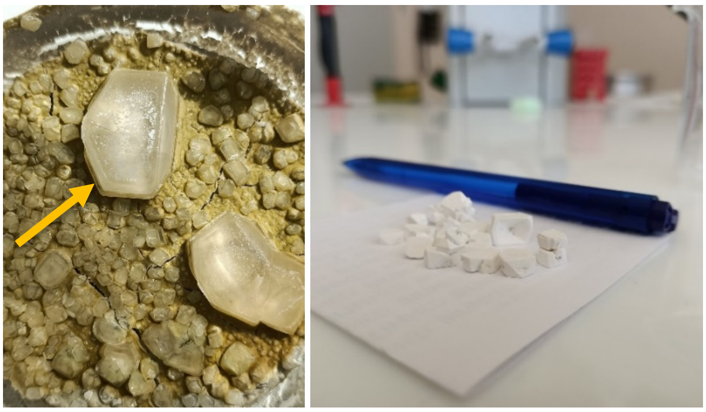

Fig. 8. Picture of boron crystals formed in the beaker after sonication (a) and the boron crystals separated from clays (b)

commercialized process since 1950. Leaching with HF, Si leaching process, leaching with a mixture of HF and H_2SO_4, leaching with HCl, NaCl, Na_2CO_3 and Na_2SO_4 can be given most studied methods for hydrometallurgical processing.

On the other hand, boric acid is an integral part of a wide variety of industrial products, such as high performance glass and ceramics. For these reasons, nowadays, both lithium and boron resources and effectively use of them have become critical importance, as they are generally not substitutes. A recent project, called Rhyolite Ridge (RRP) developed by Iooner Company can be given as an attractive example for the economic production of lithium carbonate, lithium hydroxide and boric acid (Rhyolite

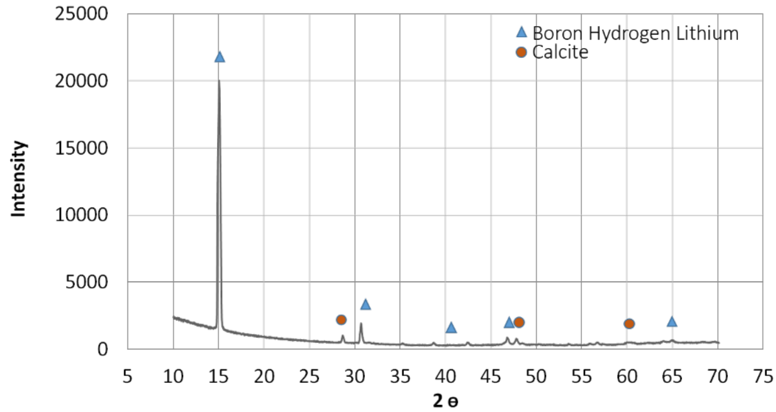

Fig. 9. The phase composition of the separated crystals

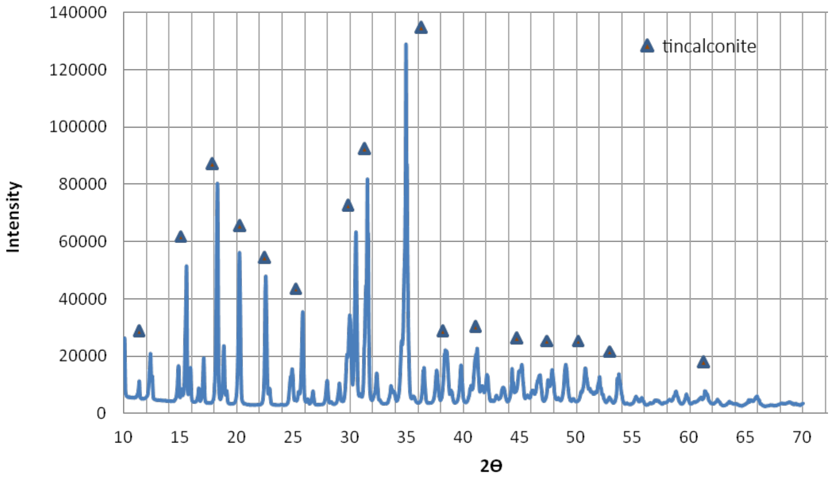

Fig. 10. The typical phase composition of glassy tincalconite

Ridge, 2021). Therefore, similar to the RRP project, the following part of the study was devoted to determine whether it is possible to extraction of lithium and boric acid from borate crystals. The dissolution of Li from borate crystals were performed in 100 cm^3 glass beaker placed on magnetic stirrer. In each test, 3 g of crystal were placed in 30 cm^3 distilled water. The solution was stirred and heated continuously until the solution temperature reached 98 ^{0}C. H_2SO_4 was used as a leaching reactive. During dissolution tests the temperature control of solution was carried out with thermocouple equipped to magnetic stirrer and the Li-ion concentration of pregnant solution (PLS) was analysed by ICP. After leaching, the solution was cooled, allowed the settle of solid precipitate and filtered with blue band filter. The leaching tests parameters and the effects of these parameters on dissolution are given in Table 3 and Fig. 11, respectively. The results revealed that the Li-ion concentration in solution (PLS) increased with increasing leaching time. The maximum Li concentration was obtained at 120 min leaching time and 0.15 mol/dm^3 H_2SO_4 concentration. Further increase on acid concentration adversely affects the solubility recovery. The relatively lower Li concentration relative to the initial sample (140 ppm) can be explained by the loss of solution during filtration. It is thought that this problem can be solved with a more controlled filtration or centrifuge application.

Table 3. The leaching test parameters

Run	H_2SO_4 Cons. (mol/dm^3)	Time (min)
1	0.3	15
2	0.3	60
3	0.3	120
4	0.6	120
5	0.15	120

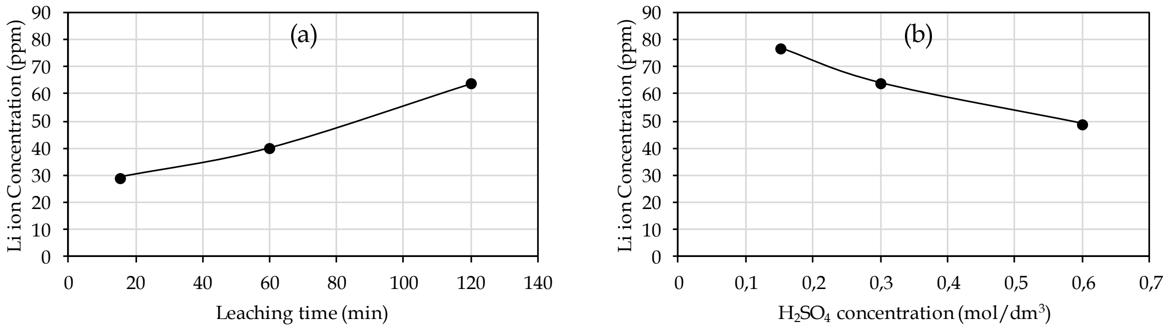

Fig. 11. a) A variation of Li-ion concentrations in PLS according to the leaching time, b) Effect of H_2SO_4 concentrations on Li-ion concentration

The content of PLS is close to saturation in boric acid, therefore, the crystalization of boric acid from PLS was achieved by cooling the leaching solution. The phase analyses of the filtered solid part after cooling stage is given at Fig. 12. It exhibits typical phase composition of pure boric acid.

According to the experimental results given above, Li-rich PLS (that can be further use as source of lithium carbonate or lithium hydroxide production), and boric acid could be produced with easy and low cost beneficiation method in accordance with the method previously introduced and implemented by the RRP. Also, the lower acid concentration and the purity of PLS over the same time period can be given highlighted advantages of the proposed method compared with previous studies focusing on lithium extraction from the same kind of materials (bauxitic clay) (Gu et al., 2020; Obut et al., 2020). This is very important for the production of lithium carbonate or lithium hydroxide in subsequent applications.

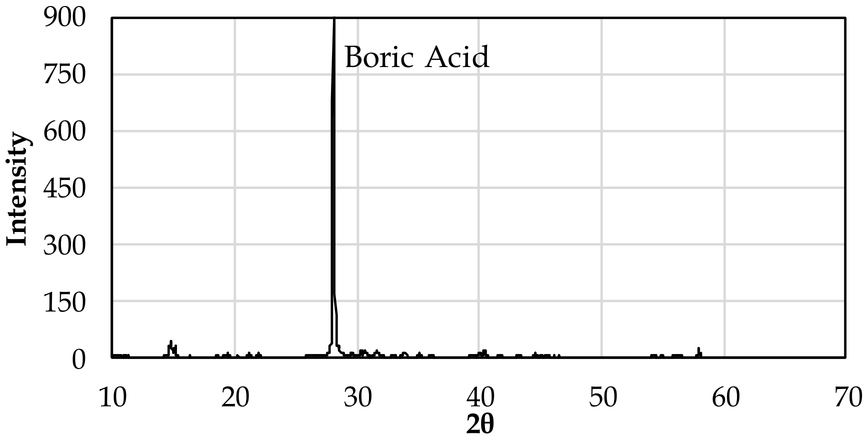

Fig. 12. The XRD analysis result of the precipitated solid part

4. Conclusions

During the past decades, ultrasonic technology has gained wide interest as an effective mechanical pretreatment method for sludge treatment. Its excellent performance, good technical and operational stability, compactness, and being an environmental friendly method make it distinguished.

In this paper, it was intended to provide a brief review on the fundamentals of ultrasonic treatment and to determine its applicability for sludge treatment by means of borax clayey waste waters. The results indicated that the use of ultrasonic treatment has a positive effect on the disintegration of clay flocks in the waste sludge. This leads the liberation of boron minerals from them and with the help of temperature they are dissolved and re-crystallized on clay substrate during cooling stage. In addition, it was also determined that the re-crystallized boron crystals can also be evaluated as Li source.

Acknowledgments

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