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Optimization of Ulexite Mineral in Ammonium Hydrogen Sulfate Solution and the Potential of Its Filter Cake as a Fertilizer Ingredient

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ABSTRACT

The study investigates the dissolution process of ulexite mineral in ammonium hydrogen sulfate solution, focusing on temperature, solid/liquid ratio, and acid concentration. A Box–Behnken model was used for experimental design, and the yield of boric acid ranged between 77.10% and 99.36%. A reduced quadratic model was proposed, showing significant effects of solid/liquid ratio and acid concentration on extraction. The optimum conditions were set as temperature: 348.28 K, solid/liquid ratio: 0.022 g/mL, and acid concentration: 2.95 M. The optimum extraction yield was calculated as 97.38%, with a desirability function value of 0.94 and a relative error of 0.97%. The characterization of solid products was confirmed using Fourier transform infrared spectroscopy, X-ray diffraction, and scanning electron microscopy. The byproduct (filter cake) was added to the growing medium of *Lepidium sativum* to create a fertilizer effect, resulting in increased and decreased radicle and plumule lengths in plants at higher concentrations.

1 | Introduction

Boron is one of the most valuable subterranean resources in Türkiye, and the most common compounds containing boron in nature are ulexite ($Na_2O.2CaO.5B_2O_3.16H_2O$), colemanite ($Ca_2B_6O_{11}.5H_2O$) and tincal ($Na_2B_4O_7{\cdot}10H_2O$) [1–3]. These substances are evaluated according to the amount of boron (III) oxide (B_2O_3) they contain. The production of boron (III) oxide from boric acid serves as a crucial raw material for the glass [4], fertilizer [5, 6], and pharmaceutical [7] sectors as well as for fire retarding systems [8] and surface coating [9] The second-largest producer in the world, ETI Mine Works General Directorate, uses sulfuric acid to make boric acid, which is one of the boron compounds from colemanite in Türkiye [10].

Studies about producing boric acid from the mineral ulexite as an alternative to colemanite mineral have been drawing attention recently in order to make boric acid production sustainable. One of the countries having the greatest ulexite sources is Türkiye, with the majority of those being in Bigadiç/Balıkesir [11]. In order to extract boric acid from the ulexite mineral, papers have been published regarding the dissolution kinetics of ulexite in various solvents such as citric acid [12], ammonium acetate [13], oxalic acid [14], and potassium hydrogen sulfate [15]. On the other hand, the Taguchi experimental design model was used to determine optimal conditions for dissolving ulexite in solvents including SO_2-saturated water [16], ammonium chloride [17], and phosphoric acid [18]. In the research conducted by Ipeksever et al. [19], the Box–Behnken experimental design approach was utilized to find ideal process conditions for the dissolution of ulexite mineral in a supercritical carbon dioxide medium. Sulfuric acid, which is used in current technology to produce boric acid, and other solvents available in previous research articles have drawbacks such as separation of the main product from the byproduct and being uneconomical. As a result, the solvent type

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is critical to product purity, and the search for alternative solvents still continues. In this study, a solution of ammonium hydrogen sulfate, which has not previously been investigated in this context, was selected with the aim of minimizing impurities.

Many parameters including temperature, concentration, solid/liquid ratio, and grain size are effective in dissolving boron compounds. The number of experiments in traditional experimental methods is very high, making it difficult to study the effect of parameters on each other. Advanced statistical methods are useful for revealing the interaction of parameters more clearly [20]. Response surface methodology (RSM), which combines statistical and mathematical techniques, is one of these procedures and displays all parameter curves, parameter interactions, and nonlinear values. This approach starts with identifying the independent variables, followed by regression modeling and an optimization step [21], and the regression coefficients allow us to specify what the major effect of the element is. The most common designs in RSM are Box–Behnken and central composite. The Box–Behnken design is a RSM-based experimental design used to evaluate the relationships between independent variables and the response, while requiring a relatively small number of experimental runs. It is a three-level design developed by Box and Behnken that allows the estimation of second-order (quadratic) models and analysis of interaction effects between variables [22]. In their trials, Farahani et al. [23] analyzed data by both Taguchi and Box–Behnken design models and discovered that the Box–Behnken experimental design produces closer results to real conditions.

In this work, the best conditions for producing boric acid, by reacting ulexite mineral and ammonium hydrogen sulfate solution, were determined. Temperature, concentration, and solid/liquid ratio were regarded as variables with the aid of the Box–Behnken model. Furthermore, the potential of filter cake, a waste material of this process, was evaluated in the cultivation of a plant, relying on its great mineral availability. It is anticipated that the experimental findings will be helpful in resolving issues that arise during boric acid manufacturing using existing technology.

TABLE 1 | Chemical composition of ulexite.

Compound	CaO	B_2O_3	Na_2O	H_2O	Other
%	18.85	36.37	6.60	35.47	2.71

2 | Experimental Section

2.1 | Materials

The ulexite mineral was obtained from the ETI Mine Works General Directorate in Bigadiç/Balıkesir. After the mineral was broken and ground by a mill (GRT-10BL), it was sieved, and the particle size was 75 μm. Chemical analysis of related minerals was conducted according to Ekmekyapar et al. [24], and the results are given in Table 1. Ammonium hydrogen sulfate (NH_4HSO_4, 99% w/w) was supplied by Merck (Darmstadt, Germany).

2.2 | Dissolution Reactions and Characterization

The experiments on the solubility of ulexite were carried out in a jacketed glass reactor with a volume of 500 mL. A water bath reactor (Dragon Lab OS20-5) with a circulator was used to keep the temperature constant during the dissolution processes, and the reactor contents were mixed with a mechanical mixer (Dragon Lab, OS20-S). After placing 200 mL of ammonium hydrogen sulfate solution (10%, w/v) with known concentration into the glass reactor, it was heated to the desired temperature. Then, a specific amount of ulexite mineral was added to the reactor, and the contents were blended for the duration of the test. The expected dissolution reactions in medium can be written as follows [25]:

$$6NH_4HSO_{4(aq)} \longrightarrow 6NH^{+}_{4(aq)} + 6HSO^{-}_{4(aq)}, \quad (1)$$

$$6HSO^{-}_{4(aq)} + 6H_2O_{(aq)} \longleftrightarrow 6H_3O^{+}_{(aq)} + 6SO^{-2}_{4(aq)}, \quad (2)$$

$$Na_2O.2CaO.5B_2O_3.16H_2O_{(s)} + 6H_3O^{+}_{(aq)} \longrightarrow 2Ca^{+2}_{(aq)} + 2Na^{+} + 10H_3BO_{3(aq)} + 2H_2O_{(l)}, \quad (3)$$

$$2Ca^{+2}_{(aq)} + 2SO^{-2}_{4(aq)} + 4H_2O_{(s)} \longrightarrow 2(CaSO_4.2H_2O)_{(s)}, \quad (4)$$

$$2Na^{+2}_{(aq)} + SO^{-2}_{4(aq)} \longrightarrow Na_2SO_{4(aq)}, \quad (5)$$

$$6NH^{+}_{4(aq)} + 3SO^{-2}_{4(aq)} \longrightarrow 3(NH_4)_2SO_{4(aq)}, \quad (6)$$

$$Na_2O.2CaO.5B_2O_3.16H_2O_{(s)} + 6NH_4HSO_{4(aq)} \longrightarrow 2(CaSO_4.2H_2O)_{(s)} + 10H_3BO_{3(aq)} + 3(NH_4)_2SO_{4(aq)} + Na_2SO_{4(aq)}. \quad (7)$$

As a result of the experiment, a vacuum pump (Isolab, GM-0) and a filtration device were used to separate the solid/liquid phases in the reactor. The amount of B_2O_3 dissolved from the ulexite mineral and transferred to the solution medium was determined by the titrimetric method, and the yield of extraction was calculated by Equation (8). The filter cakes were dried in an oven (Gemo DT104) at 105°C, until they reached a constant weight [19]. Triplicate experiments were performed for each run.

$$\text{Yield}(\%) = \frac{\text{concentration of boric acid in the solution medium}}{\text{concentration of boric acid in ulexite}} \times 100. \quad (8)$$

Solid products (ulexite, crytallized particles, and filter cake) were examined by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning electron microscope (SEM). FTIR spectra were recorded by a spectrometer (PerkinElmer Spectrum 65, USA) with a range of 4000–600 cm^{-1} wavenumber, in order to detect functional groups. The X-ray diffractometer (PANalytical, Empyrean) was run at 45 kV and 40 mA, while exposed to CuK-α radiation for the 2θ range of 5°–80°. Solids were photographed using appropriate magnifications through a SEM equipment (FEI, Quanta 650). Following the application of an Au-Pd coating, SEM micrographs were taken at an accelerating voltage of 10.0 kV and a pressure of 70 Pa.

2.3 | Experimental Design and Modeling

Box–Behnken design has been considered, because of being less expensive than central composite designs, and it has three levels for each factor [26, 27]. Preliminary experiments revealed that three parameters were chosen for optimizing boric acid extraction from ulexite mineral using ammonium hydrogen sulfate: temperature (X_1), solid/liquid ratio (X_2), and concentration (X_3) (Table 2). These parameters have been selected in accordance with the literature [20, 24].

The number of experiments is determined by the following equation:

TABLE 2 | Factors and their levels of dissolution tests.

Factor	Term	Actual level		
		−1	0	1
Temperature (K)	X_1	338	343	348
Solid/liquid ratio (g/mL)	X_2	0.02	0.06	0.10
Acid concentration (M)	X_3	2.0	2.5	3.0

$$N = 2k(k-1) + \text{nc}, \quad (9)$$

where k and nc indicate the number of levels and central trials, respectively [27]. Recommended number of tests to be performed at the center was 5, and therefore, the number of required experiments was equal to $2 * 3(3-1) + 5 = 17$ (Table 3).

An analysis of variance (ANOVA) at 95% confidence level was applied to analyze the results, which were carried out using Design-Expert software (trial Version 13.0, Stat-Ease, Minneapolis, MN, USA). A second-order polynomial equation was beneficial for representing the mathematical relationship between independent variables and the extraction yield of boric acid. The corresponding mass values were also calculated, but in the modeling step, only yield values were accepted as response. "y" designated the estimated yield of boric acid in consequence of extraction (response), while "ε" stood for the noise or error of response. A three-dimensional surface and contour graph were also utilized to demonstrate the relevance between factors and boric acid yield.

$$y_n = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_1^2 + \beta_5 X_2^2 + \beta_6 X_3^2 + \beta_7 X_1 X_2 + \beta_8 X_1 X_3 + \beta_9 X_2 X_3 + \varepsilon. \quad (10)$$

In Equation (3), y_n was the dependent variable and a function of the independent variables, which were encoded as X_1, X_2 and X_3. β_0, and $\beta_1 - \beta_3$, $\beta_4 - \beta_6$, and $\beta_7 - \beta_9$ were the coefficients of regression, linearity, quadratic, and interaction, respectively [28].

2.4 | Usage of Solid Byproduct as a Fertilizer

2.4.1 | Seed Germination Assay

Lepidium sativum seeds used in studies were purchased from a local market in Osmaniye Province. Before placing the seeds in the petri dishes (with double-layer Whatman No.1 paper inside), they were sterilized with sodium hypochlorite (% 10 v/v) for 30 min [29]. The seeds sterilized ($n = 30$) were placed in petri dishes, and fertilizer (filter cake accepted as fertilizer, and no soil was used in cultivation) diluted with appropriate concentrations (10, 20, 40, and 80 ppm) was added to the medium. Double distilled water was used as the control group. The petri dishes containing the cress seeds were covered thoroughly with parafilm and kept at room temperature. At the end of the 5th day, the radicle and plumule lengths of the germinated seedlings were measured with the help of a ruler.

2.4.2 | Total Chlorophyll and Carotenoid Contents

To determine the total chlorophyll content, the leaves (0.1 g) of germinated seedlings were crushed in 80% acetone (10 mL) and kept at 4°C for two days in the refrigerator [29]. The extracts taken from the refrigerator were centrifuged at 5000 rpm for 5 min, and the supernatant was measured at 480, 645, and 663 nm in a UV–Vis spectrophotometer (Model: V-730, JASCO Inc., USA). Chlorophyll (a and b) and carotenoid contents were calculated according to Arnon [30] and Duxbury and Yentsch [31], respectively.

2.4.3 | Statistical Analysis

All data obtained were evaluated with the one-way ANOVA test by using the SPSS program (Version 18.0, SPSS Inc., Chicago, IL, USA). Furthermore, all assays were carried out in triplicate.

3 | Results and Discussion

3.1 | Model Fitting

As depicted in Table 4, the proposed model for the extraction yield of boric acid was significant ($p = 0.0037 < 0.05$), with a determination coefficient (R^2) level of 0.8538 and unimportant lack of fit ($p = 0.9853 > 0.05$). If a R^2 is higher than 0.8, it could be said that the related model serves an adequate correlation between predicted and observed values [32, 33]. The difference between predicted and adjusted R^2 was lower than 0.2, which proved a reasonable agreement. Furthermore, the inability of the model to represent data in the domain at points that are not included in the regression is highlighted by the lack of fit [19],

TABLE 3 | Experimental matrix with responses.

Run	Factor 1 X_1: temperature K	Factor 2 X_2: solid/liquid ratio g/mL	Factor 3 X_3: concentration M	Response 1* Yield %	Response 2 Mass g
1	343	0.10	3.0	82.80	30.11
2	348	0.10	2.5	79.14	28.78
3	348	0.02	2.5	94.80	34.47
4	343	0.02	2.0	89.85	32.69
5	343	0.06	2.5	79.15	28.79
6	338	0.02	2.5	90.10	32.77
7	348	0.06	3.0	93.80	34.11
8	338	0.06	3.0	92.05	33.48
9	348	0.06	2.0	78.60	28.59
10	343	0.06	2.5	81.40	29.61
11	338	0.06	2.0	77.15	28.06
12	343	0.06	2.5	81.16	29.52
13	343	0.06	2.5	81.35	29.59
14	338	0.10	2.5	78.01	28.37
15	343	0.10	2.0	77.10	28.04
16	343	0.02	3.0	99.36	36.14
17	343	0.06	2.5	92.37	33.59

*Mean of duplicate extraction.

TABLE 4 | ANOVA table in modified process mode.

Source	Sum of squares	df	Mean square	F-value	*p* value
Model	735.09	7	105.01	7.51	0.0037
X_2: solid/liquid ratio	192.52	1	192.52	13.77	0.0048
X_3: concentration	226.50	1	226.50	16.20	0.0030
X_2X_3	3.63	1	3.63	0.2596	0.6227
X_2^2	19.82	1	19.82	1.42	0.2643
X_3^2	17.81	1	17.81	1.27	0.2882
$X_2^2\ X_3$	27.71	1	27.71	1.98	0.1928
$X_2X_3^2$	0.3042	1	0.3042	0.0218	0.8860
Residual	125.83	9	13.98		
Lack of fit	14.58	5	2.92	0.1049	0.9853
Pure error	111.25	4	27.81		
Cor total	860.93	16			

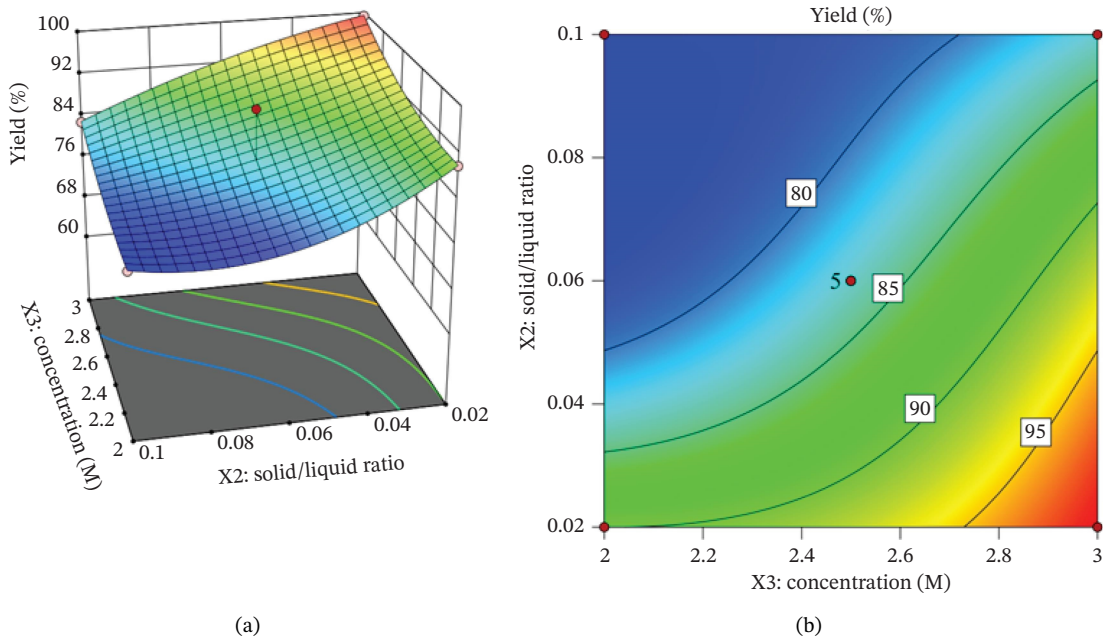
Abbreviation: df, degrees of freedom.

hence it must be insignificant. The standard deviation of two replicates was 3.74, and the coefficient of variance (CV) was 4.39% (data not shown). CV is an indicator of the reproducibility of the mathematical model, and it should not exceed 10% [34, 35].

Prediction error sum of squares (PRESS) values are ought to be minimum in order to get a well-accorded model [36]. The cubic model was aliased, and the low PRESS of 221.35 showed that the reduced quadratic model responded better than the others. In model summary statistics, PRESS levels were computed as 263.13, 391.77 and 635.95 for linear, 2FI, and quadratic models, respectively (data not shown). A second-order polynomial model has been widely used in RSM to optimize boric acid extraction processes from boron minerals such as ulexite [37].

3.2 | Influence of Process Variables on Extraction Yield

The F-value is the ratio of the variance explained by a model term to the variance of the error. It indicates the significance of each factor in the leaching process, and higher F-values correspond to lower *p* values, meaning greater statistical significance [38, 39].



(a) (b)

FIGURE 1 | 3D surface plot (a) and contour plot (b) of extraction yield.

ANOVA table showed that significant linear effects were observed in the extraction yield of boric acid (Table 4), and the most important one was concentration (F-value = 16.20). Both solid/liquid ratio and concentration influenced yield in a significant way; however, process temperature did not demonstrate a similar behavior. Besides that, the related unit operation possessed interaction (X_2X_3), quadratic (X_2^2, X_3^2), and interactive-quadratic ($X_2^2\ X_3$, $X_2X_3^2$) impacts of same factors. Paying attention to F-values, the importance of factors was as follows, except linear terms: $X_2^2\ X_3 > X_2^2 > X_3^2 > X_2X_3 > X_2X_3^2$. From the response surface plot and contour plot exhibited in Figure 1, the effect of solid/liquid ratio was more pronounced at higher concentration values. The extraction yield decreased with increasing solid/liquid ratio and acid concentration, although the interaction of these two factors had a tendency to enhance yield percentage. Surprisingly, temperature did not have a remarkable potential for recovering boric acid from the solution medium, and it may have arisen from the fact that the studied temperature gap was narrow. In fact, some studies on the extraction of boric acid available in the literature did not consider the impact of temperature [40–43]. The stages of extraction yield ranged between 77.10% and 99.36% (Table 3), and the maximum efficiency was found in these conditions: 343 K, 0.02 g/mL, and 3 M. Gezer et al. [38] extracted boric acid from tincal mineral by ultrasound assisted technology, and independent variables were pH (1–7), time (30–70 min), temperature (30°C–100°C), and solvent/solid ratio (15–35 g/mL). They found yield values in the range of 26.25% and 82.75%, which were comparably lower than the results of this study.

The mathematical expression generated by Design-Expert software in actual terms was

$$\text{Yield}(\%) = 161.57 - 1856.55X_2 - 51.62X_{3-} + 705.75X_2X_3 + 12986.84X_2^2 + 10.56X_3^2 - 39.00X_2X_3^2 - 4653.13X_3X_2^2. \quad (11)$$

3.3 | Confirmation of Optimum Conditions

A numerical optimization step was performed, and constraints were established as "in range" for all factors and "maximize" for the response. The software recommended 100 solutions with different desirability (D) values, and the verified conditions were temperature: 348.3 K, solid/liquid ratio: 0.022 g/mL, and concentration: 2.95 M. The experiment was run twice, and the average result was calculated. According to the experimental findings, under optimal circumstances, boron removal effectiveness was up to 97.38%. The relative error between the average and the predicted value was 0.97%, and the D function was equal to 0.94. D function has a value of $0 < D < 1$, and zero desirability signs an unacceptable limit of any factor, although 1 points out the best performance of independent variables [44]. Ekinci et al. [45] modeled boric acid extraction by using carbon dioxide and sulfur dioxide from colemanite, and under optimum conditions, they stated the yield as approximately 99.9%.

3.4 | Structural Characterization by XRD

The X-ray diffractograms of powder from filtrate and product precipitated from mother liquor are depicted in Figures 2(a) and 2(b), respectively. In Figure 2(a), the characteristic peaks were observed at 2θ values of 18.78°, 25.42°, 29.39° and 31.42°, which corresponded to boric acid (H_3BO_3) (JCPDS no. 30–0199). Also, Bingol and Copur [46] enlightened the structure of boric acid obtained from colemanite mineral using carbon dioxide, and Levent et al. [47] leached boric acid from tincal mineral under supercritical and subcritical ethanol conditions and specified it

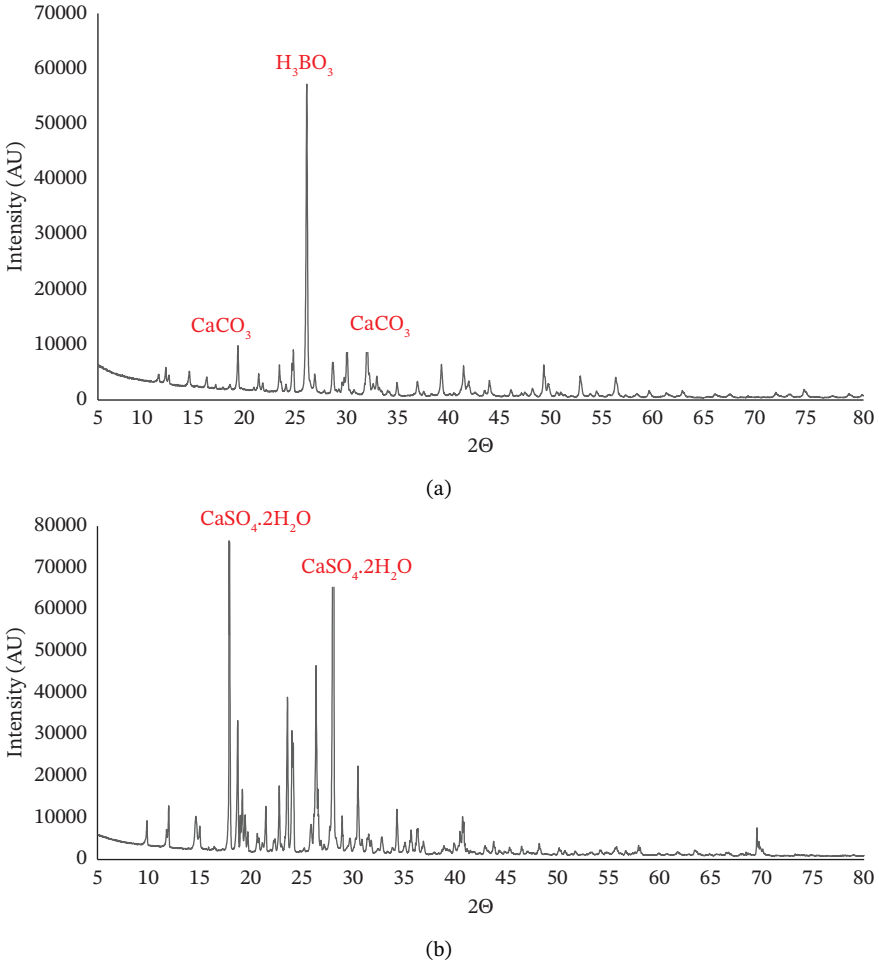


FIGURE 2 | XRD graphs of (a) boric acid and (b) filter cake.

by XRD. Moreover, the XRD patterns of filter cake indicated that the product was calcium sulfate ($CaSO_4$) [48]. The peaks on Figure 2(b) at 9.84°, 11.99°, 17.86°, 18.68°, 21.45°, 22.76°, 23.60°, 26.39°, 28.11°, 30.51°, 34.33°, 40.80° and 69.51° belonged to different forms of $CaSO_4$ such as bassanite, anhydrite, and dihydrate [49].

3.5 | Chemical Characterization by FTIR

FTIR analysis was performed in the 4000–800 cm^{-1} region to investigate the functional groups and chemical structure of the samples (Figure 3). A broad band observed in the 3600–3200 cm^{-1} region is attributed to O–H stretching vibrations associated with structural water molecules in hydrated mineral phases. The band around 1650 cm^{-1} corresponds to the H-O-H bending vibration of water molecules, indicating the presence of adsorbed or crystallization water in hydrated phases [50]. Ulexite contains both hydroxyl groups and structural water molecules, which contribute to the O-H stretching bands observed in the FTIR spectra. The strong absorption bands in the 1100–1000 cm^{-1} range are due to the asymmetric stretching vibrations of sulfate (SO_4^{2-}) groups. These bands are typical of sulfate-containing mineral phases. Sulfate compounds usually have extra deformation bands in the lower wavenumber range, which is around 660–600 cm^{-1} [50].

The bands observed in the 1450–1380 cm^{-1} range arise from the asymmetric stretching vibrations of carbonate (CO_3^{2-}) groups. Meanwhile, the band located around 864–876 cm^{-1} corresponds to the out-of-plane bending vibration of carbonate ions [50]. These bands indicate the presence of carbonate species within the sample.

When comparing the FTIR spectra of samples ulexite (Figure 3(a)) and filter cake (Figure 3(b)), noticeable differences in band intensity can be observed in certain regions. In Spectrum A, the carbonate-related bands around 1450–1380 and ~864 cm^{-1} are stronger. This means that there are still carbonate species such as $CaCO_3$ present. In the 1100–1000 cm^{-1} range, Spectrum B, on the other hand, has relatively stronger sulfate-related bands. This suggests that sulfate-containing phases play a bigger role. These findings correspond with the XRD results, which reveal the presence of carbonate phases in sample ulexite and sulfate phases in sample filter cake.

In general, the FTIR spectra show that the samples contain hydroxyl, sulfate, and carbonate groups. The presence of both sulfate and carbonate bands at the same time suggests that the material has sulfate minerals and leftover carbonate parts, which fits with what the XRD analysis showed about the material having multiple phases [50, 51].

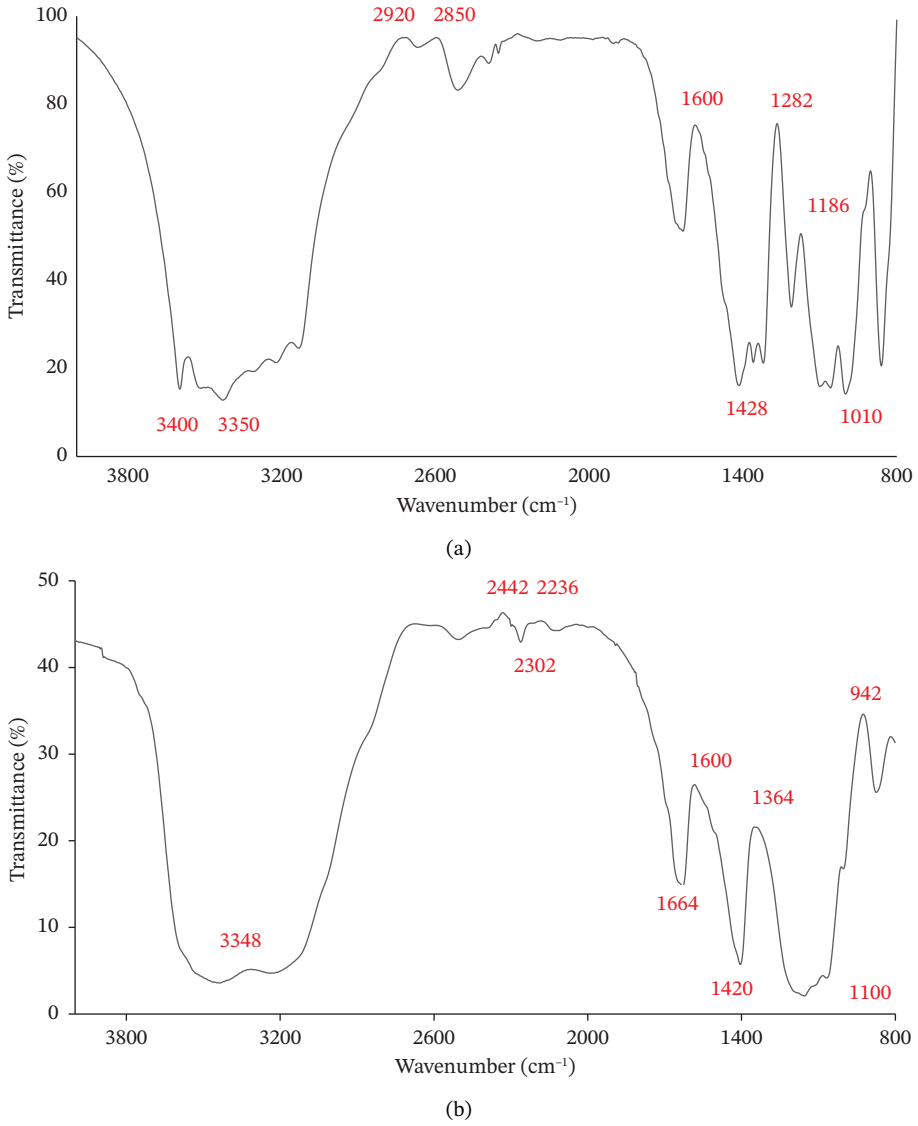


FIGURE 3 | FTIR spectra of (a) ulexite and (b) filter cake.

3.6 | Morphological Characterization by SEM

The microstructure of ulexite mineral, a powder product from filtrate and filter cake, is visualized in Figure 4. The dimensions of ulexite particles did not seem equal to each other, which may have probably originated from grinding in terms of Figures 4(A) and 4(B), but their surfaces were mostly smooth and vitreous [52]. A very similar image of this mineral was captured by Kuslu et al. [53]. When a deep investigation was performed for other photographs, it can be inferred that the surface roughness increased. Relatively high process temperature might cause this outcome. Also, some openings existed, especially in filter cake due to the leaving of leaching agent [13], and boric acid particles had a spherical morphology (Figure 4(D)). The size of calcium sulfate granules was smaller than that of boric acid ones (Figure 4(F)).

3.7 | Fertilizer Potential of Filter Cake

In vitro effects on *L. sativum* as a fertilizer ingredient are investigated at different concentrations (0, 10, 20, 40, and 80 ppm) (Figure 5). The concentrations used were determined in accordance with studies in the literature [54]. It is determined that the radicle and plumule lengths of *L. sativum* with the application of filter cake at the lowest concentration (10 ppm) increased and decreased at higher concentrations depending on dose increase, respectively, when compared to the control. At the same time, the chlorophyll and carotenoid contents of the tested plant increased with fertilization.

The application increased the elongation of both the radicle and the plumule of the specimen by 14.92% and 22.14%, respectively, at 10 ppm concentration (Figure 6). The amounts of Chlorophyll-a, Chlorophyll-b, and carotenoid increased at the rates of 64.50%, 108.98%, and 72.09% for 20 and 10 ppm concentrations, respectively (Figure 7). In previous studies, the effects of ammonium sulfate and ammonium nitrate fertilizers on the growth and yield of buckwheat (*Fagopyrum esculentum Moench*) plants were investigated, and an increase in grain yield, biological yield, grain weight, agronomic efficiency, and protein content was observed [55]. Another study examined the

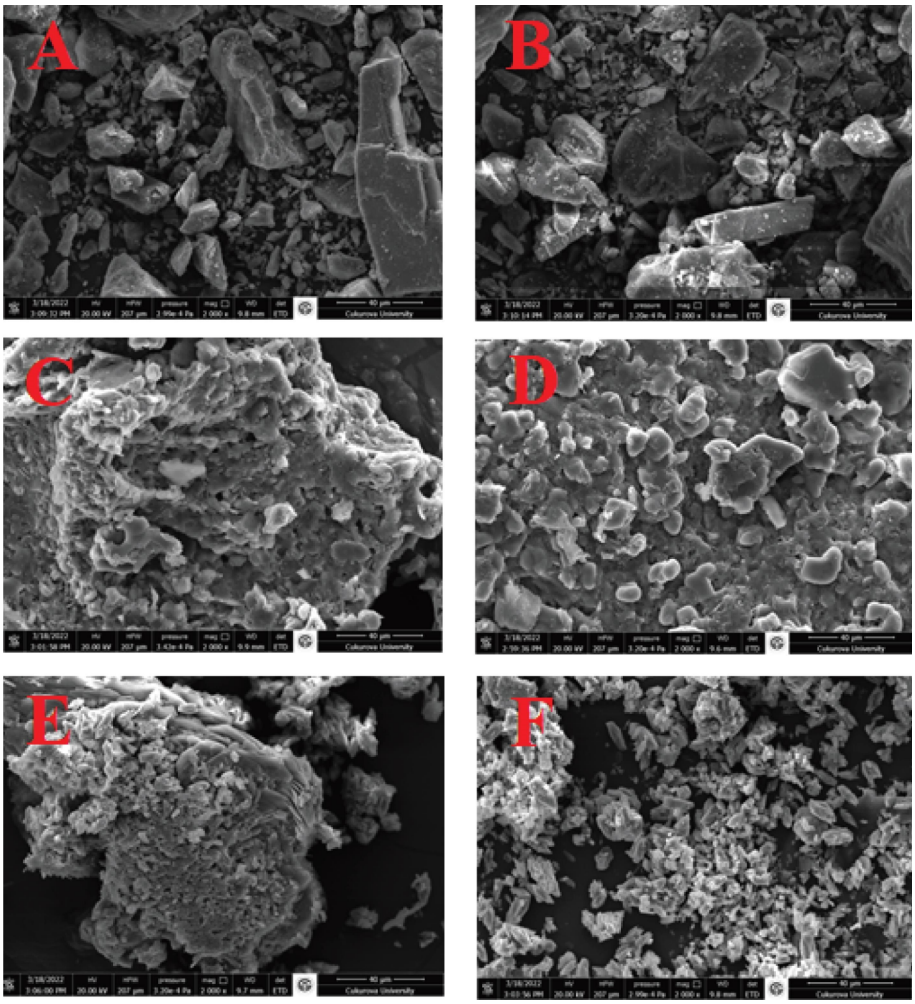


FIGURE 4 | SEM microphotographs of (A–B) ulexite, (C–D) filter cake, and (E–F) remaining solid.

use of ammonium sulfate fertilizer on broccoli plants, and it was found that the application of this fertilizer resulted in increased leaf and root length [56].

Based on the abovementioned results, it was concluded that the applied fertilizers affected the radicle and plumule growth of the tested plant and increased the total chlorophyll and carotenoid

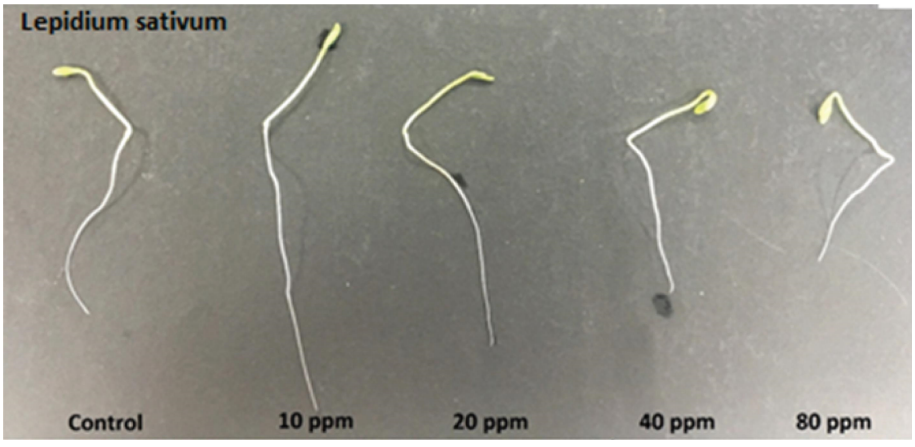


FIGURE 5 | Dosage responses of the *L. sativum* plant.

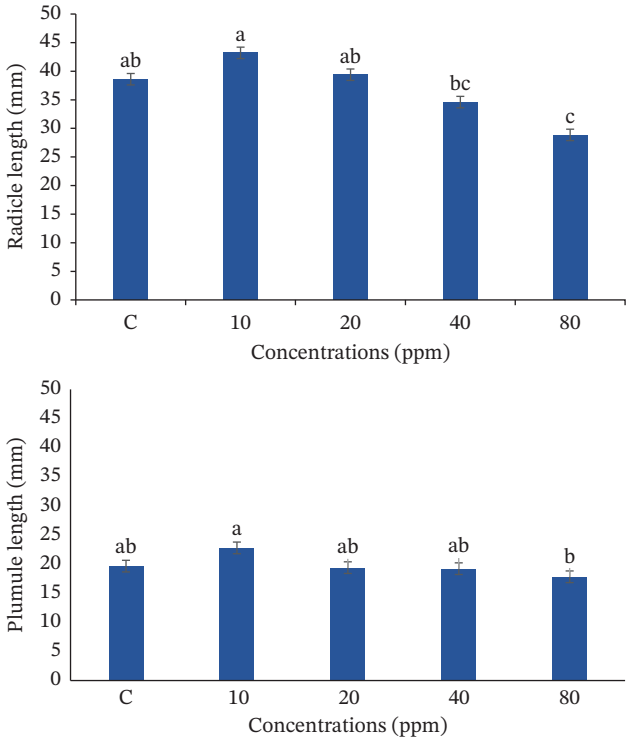


FIGURE 6 | In vitro effects of fertilizer on the radicle and plumule growth of *Lepidium sativum*. C: control. Means indicated with different letters are significant at $p > 0.05$.

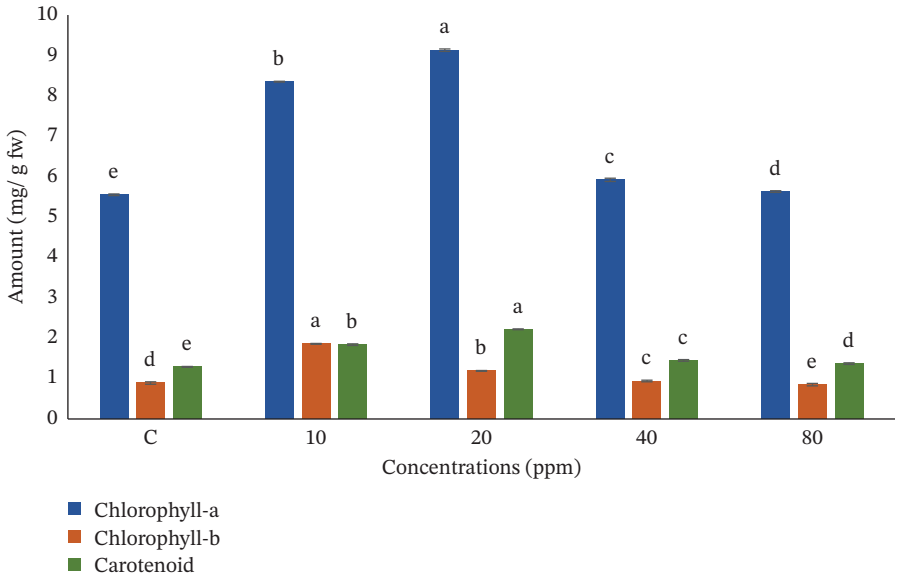


FIGURE 7 | Fertilizer on the total chlorophyll and carotenoid contents of *Lepidium sativum*. Means indicated with different letters are significant at $p > 0.05$.

contents. Future research should instead examine the effects and mechanisms of action of fertilizers made using various chemical or biological processes on growth and development traits by misting them over the leaves of various plants, both in vitro and in the field.2

4 | Conclusion

An optimization study about the extraction of boric acid from ulexite mineral using ammonium hydrogen sulfate as an agent was implemented by this research, and some concluding points became known within the following:

- To the best of our knowledge, this is the first successful optimization study focused on the removal of boric acid from ulexite by employing a less corrosive and a nonvolatile solvent, ammonium hydrogen sulfate, by using the RSM approach.
- Three factors considered for the extraction process were temperature, solid/liquid ratio, and acid concentration.

Even though temperature was not an important criterion in leaching, the effect of concentration was higher than the solid/liquid ratio.

- Boric acid was mixed with calcium sulfate dihydrate, ammonium sulfate, and sodium sulfate in the medium, after the reaction occurred.
- Regarding the 3D surface plot of extraction yield, a saddle point was observed, which may indicate the presence of interaction effects between variables in the experiments.
- A second-order polynomial regression model was developed by eliminating nonsignificant terms, yielding an acceptable coefficient of determination (R^2).
- Among 100 different solutions, the second one was verified, and the average boric acid extraction yield was recorded as 97.38%, which was a highly impressive and satisfying result with a low percentage of relative error.
- Desirability function value nearly reached 1, which proved the criteria determined within the limits of the study.
- RSM in boric acid production was an efficient tool, and further research studies should be conducted about using different chemical solvents.
- The application of filter cake as a fertilizer influenced the growth of the radicle and plumule in *L. sativum*, ultimately resulting in increased chlorophyll and carotenoid contents.

Nomenclature

β_0-β_9	Regression coefficients
CV	Coefficient of variance
D	Desirability value
df	Degrees of freedom
K	Reaction temperature
M	Solution concentration
p:	Significance value
PRESS	Prediction error sum of squares
R^2	Determination coefficient
v	Volume
w	Mass
X_1, X_2, X_3	Codes of temperature, solid/liquid ratio, and acid concentration, respectively
y	Estimated yield

Author Contributions

Havva Mumcu Şimşek: conceptualization, methodology, validation, formal analysis, resources, writing–original draft, writing–review and editing, visualization, data curation, project administration, investigation, and supervision.

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Ethics Statement

The author has nothing to report.

Conflicts of Interest

The author declares no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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