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Optimization and Investigation of the Design Parameters for Boric Acid Production from Colemanite Using the Ultrasound-Assisted Extraction Process

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Abstract
In this study, boric acid was extracted from colemanite minerals employing a new extraction method, ultrasound-assisted extraction (UAE), which has recently drawn the attention. Since the extraction process depends on the experimental conditions, such as solvent/solid ratio, pH, extraction time and temperature, the response surface methodology (RSM) was used to investigate and optimize the aforementioned experimental parameters in order to obtain maximum boric acid extraction yield. A central composite design (CCD), which is one of the RSMs, was employed and the respective parameters were investigated for their individual and mutualistic effects on the boric acid extraction yield, which was called as the response in this study. At the end, a second order quadratic model was obtained, and optimum conditions were attained as 25.90 mL (1 g colemanite)-1, 10.76, 4.6 h and 75.65°C, respectively. The boric acid extraction yield was obtained as 99.73% under the optimum conditions.

Keywords: *boron, colemanite, optimization, ultrasound-assisted extraction, central composite design.*

1. INTRODUCTION

Borates are known as salts of boric acid, and various numbers of minerals are being used commercially to produce boric acid as those minerals have been used for many applications. In the production process, there are limited numbers of countries in the world, and Turkey is amongst these countries which has enormous amount of boron reserves. For boric acid production, naturally existed boron minerals, such as colemanite, tincal and ulexite, are utilized. Between those boron minerals, colemanite ($Ca2B6O11•5H2O$), which theoretically has 50.8% boric acid, is specially preferred by some industries and chemical plants due to its calcium content (i.e. production of boric acid, fiberglass, heat-resistant glass, washing products, etc.). In addition, colemanite has the lowest cost for boric acid production, and Turkey is its major supplier. In the world, Bigadic-Balıkesir location (Turkey) has the largest colemanite deposits (Helvaci and Alonso, 2000; Kistler and Helvaci, 1994; Ucbeyiay and Ozkan, 2014).

In previous studies, boric acid was conventionally produced by concentration, precipitation, extraction, crystallization, washing, filtration, and drying processes and the final product consisted of nearly 10% boric acid (Celik *et al.*, 2002; Kistler and Helvaci, 1994; Kuskay and Bulutcu, 2011). Some of these techniques result in loss of product and efficieny (Levent *et al.*, 2016). During boric acid production, hydrochloric acid, phosphoric acid, sulfuric acid, propionic acid, acetic acid, and nitric acid have been employed (Bulutcu *et al.*, 2001; Gür, 2007; Kuskay and Bulutcu, 2011; Temur *et al.*, 2000; Yesilyurt, 2004; Yesilyurt *et al.*, 2005). Amongst them, sulfuric acid has the most industrial usage; however, this production has some drawbacks (Levent *et al.*, 2016; Yesilyurt *et al.*, 2005). Since colemanite contains clays, carbonate minerals and arsenic containing compounds as gangue minerals, its recovery and production of boric acid tends to have low efficiencies (Kocan and Savas, 2004; Yesilyurt *et al.*, 2005). This problem can be significantly improved by different solvents (i. e. hydrochloric acid) coupled with novel ultrasound-assisted extraction (UAE) process, which is an alternative method to conventional techniques. UAE is used to extract many compounds from different types of samples, and is inexpensive, does not require many steps and instruments and does not need high extraction temperatures (Luque-Garcia and de Castro, 2002; Ramić *et al.*, 2015). In addition, UAE increases the mass transfer and therefore, allows faster and more efficient extraction (Roldán-Gutiérrez *et al.*, 2008).

In extraction processes, the process parameters are known to have significant impact on the product yield. However, previous studies show very limited information about process parameters and their results on the product yield, because researchers mostly studied the effects of temperature, speed of agitation, colemanite size and acid concentration on the reaction kinetics (Kuskay and Bulutcu, 2011). Furthermore, there has been no study investigating an appropriate production process and the optiumum design parameters to augment the boric acid extraction yield employing a statistical design method, such as response surface methodology (RSM). Therefore, it is highly desirable to find simple and economic methods for the boric acid production with high quality and yields.

RSM is beneficial for developing, improving and optimizing various processes by minimizing the number of experiments required. In the RSM, independent variables (i.e. solvent/solid ratio, pH, extraction time and temperature) are employed and a response (i.e. product yield) is taken into consideration to investigate the synergistic and antagonistic effects between those variables. Although there are many types of designs in the RSM, central composite design (CCD) is one of the most important experimental design tools to optimize the process parameters and has been employed in a wide range of studies (Okyay and Rodrigues, 2014).

The aim of this study was to optimize the boric acid production from colemanite mineral using ultrasound-assisted extraction method to get a maximum yield by screening the process parameters (independent variables) namely solvent/solid ratio, pH, extraction time, and extraction temperature. To do that, central composite design was used for the first time and the boric acid extraction yield was defined as a function of these independent variables. ANOVA analyses were performed, and the model equation was obtained.

2. EXPERIMENTAL SECTION

2.1. Preparation of colemanite mineral

In this research, concentrated colemanite obtained from the General Directorate of Bigadiç mining operations (Balikesir, Turkey) was used. First, colemanite minerals were crushed and ground. Next, particles were pulverised in a ball mill, and separated according to their size. 0.075 mm (200 mesh) colemanite powder was used in this study.

2.2. Chemicals

In the present study, analytical grade HCl (37% pure) and NaOH (98%, pellets) (Merck) were utilized. Before each assay, 0.15 M NaOH, 2.5 M NaOH and 2.5 M HCl solutions were freshly made.

2.3. Design of experiments

To get a maximum yield for boric acid extraction from colemanite particles, a central composite design (CCD) was employed. For this aim, the Design Expert software (version 8.0.7.1) was utilized. In the CCD, which contained 30 experiments, solvent/solid ratio (X1), pH (X2), extraction time (X3) and extraction temperature (X4) were tested at 5 different levels (Table 1) to get optimum conditions for boric acid extraction. The response of the CCD was the boric acid extraction yield (%) (Table 2).

Table 1. Coded levels of independent variables used in the central composite design (CCD).

Independent variables	Symbol	Levels Lowest -2	Low -1	Center 0	High +1	Highest +2
Solvent/solid ratio (mL (1 g colemanite)$^{-1}$)	X_1	15	20	25	30	35
pH	X_2	9	10	11	12	13
Extraction time (h)	X_3	1	2	3	4	5
Extraction temperature (°C)	X_4	40	50	60	70	80

Table 2. Central composite design (CCD) used in this study.

Run	Solvent/solid ratio	pH	Extraction time	Extraction temperature	Yield (%)
1	25	11	3	60	49.517
2	20	10	4	70	76.791
3	30	10	2	70	59.612
4	30	10	4	70	82.926
5	20	10	4	50	58.37
6	20	10	2	70	59.864
7	25	11	3	60	49.606
8	30	12	4	50	77.124
9	30	12	2	50	46.286
10	30	10	4	50	63.123
11	20	12	4	50	58.765
12	20	12	2	50	49.637
13	30	10	2	50	37.985
14	25	11	3	60	49.498
15	20	12	4	70	70.845
16	20	10	2	50	49.762
17	25	11	3	60	49.503
18	30	12	4	70	96.81
19	30	12	2	70	70.764
20	20	12	2	70	66.442
21	25	11	3	80	80.634
22	25	11	3	60	49.528
23	25	11	1	60	50.854
24	25	11	5	60	79.487
25	25	13	3	60	69.52
26	25	9	3	60	50.845
27	25	11	3	40	53.372
28	35	11	3	60	59.612
29	25	11	3	60	49.502
30	15	11	3	60	43.479

2.4. Ultrasound-assisted extraction method (UAE)

For each experiment, Erlenmayer flasks containing 1 g of colemanite powder was used. UAE was done in a thermostatically controlled ultrasonic bath (Bandelin Sonorex) at 50 kHz using different HCl/solid ratio (mL (1 g colemanite)-1). These ratios were indicated in the experimental design (Table 2). Similarly, in UAE, different pH values specified in Table 2 were used, and therefore each solution's pH was adjusted with a pH-meter (Mettler Toledo).

Erlenmayer flasks were placed into the ultrasonic bath, and the extraction was done at different temperatures and extraction times, which were specified for each flask in Table 2.

After each experiment was completed, each reaction mixture was filtered through a Whatman filter (0.45 μm). Afterward, each filtrate was used todetermine the boric acid content by a conductometric method (Sert *et al.*, 2012). The boric acid extraction yield (%) was calculated according to the following equation:

$$\text{Boric acid yield } (\%) = C/C\_0 \times 100 \qquad (1)$$

where c is the amount of extracted boric acid after the time (specified in Table 2) passed, and c0 is the amount of boric acid in the tincal ore.

2.5. Data analysis

In the present study, Design Expert software was also used for the statistical analysis, optimization and graphics. The quadratic model predicting the optimum values of the independent variables used here was shown below:

$$\begin{aligned} Y = {} & \beta 0 + \beta 1X1 + \beta 2X2 + \beta 3X3 + \beta 4X4 + \beta 11X12 + \beta 22X22 + \beta 33X32 + \beta 44X42 \\ & + \beta 12X1X2 + \beta 13X1X3 + \beta 14X1X4 + \beta 23X2X3 + \beta 24X2X4 + \beta 34X3X4 \qquad (2) \end{aligned}$$

In Eq. (2), X1, X2, X3, and X4 are the independent variables indicate the response Y. β0 is the offset term, whereas β1, β2, β3, and β4 are linear coefficients. In addition, β11, β22, β33, and β44 are the quadratic coefficients while β12, β13, and β14 indicate interaction coefficients. p=0.05 was taken into consideration as a level of significance in the variance analysis (ANOVA). The quality of the model was presented by the coefficient of determination (R2).

3. RESULTS AND DISCUSSION

3.1. Central composite design (CCD) results

In our study, we investigated how solvent/solid ratio (15-35 mL (1 g colemanite)-1), pH (9-13), extraction time (1-5 h), and extraction temperature (40-80°C) affected the yield of boric acid extraction by CCD. These parameters and their levels used in this study were shown in Table 1. 30 experiments were run according to the design table, and the boric acid extraction yields were calculated (Table 2). The results of the regression analysis done after the CCD experiments were completed were presented in Table 3. According to this, quadratic model was suggested by the Design Expert software with a R2 of 0.9767. This finding indicates that the statistical model obtained in this study was able to explain 97.67% of the variability in the response. In the data analysis, the adjusted R2 (0.9533) and the predicted R2 (0.8454) were also observed, and their results were shown in Table-3. For a significant model, it was reported that R2 should be at least 0.80 (Joglekar and May, 1987). Even though the adjusted R2 and predicted R2 were found slightly smaller than the R2, this situation is accepted if there are many test variables in a study (which is the case here) (Okyay and Rodrigues, 2014). These higher R2 values indicate the significance of this study.

Table 3. The results of the regression analysis of the CCD.

Source	Std. dev.	R^2	Adjusted R^2	Predicted R^2	PRESS	
Linear	8.55	0.6879	0.6359	0.5381	2597.96	
2FI	8.48	0.7698	0.6419	0.5066	2775.27	
Quadratic	3.06	0.9767	0.9533	0.8454	869.80	Suggested
Cubic	1.66	0.9971	0.9863	0.1455	6442.43	

The coefficients of response were provided in Table 4, and the model F-value (41.84) and its p-value (<0.0001) shows that the model obtained was highly significant. The p-values less than 0.05 and p-values less than 0.01 indicate the significant and highly significant model terms, respectively, and hence, X1, X2, X3, X4, X1X2, X1X3, X1X4, X22, X32, X42 were found as the significant terms for the response. According to the results, all the four test parameters (X1, X2, X3, and X4) were found to be important individually for the boric acid extraction, which have very small p values (Table 4). Amongst the coefficients, X2X3, X2X4, X3X4 and X12 showed no significant effects on the response, as their p>0.05. This indicates that only X1X2, X1X3 and X1X4 have mutual effects on the yield.

Table 4. Second order response model constants and regression equation coefficients for the response.

Variable	Sum of Square	df	Mean Square	F Value	P-value	
Model	5493.06	14	392.36	41.84	< 0.0001	*Highly significant*
X_1	243.33	1	243.33	25.95	0.0002	
X_2	305.24	1	305.24	32.55	< 0.0001	
X_3	1694.58	1	1694.58	180.70	< 0.0001	
X_4	1625.69	1	1625.69	173.35	< 0.0001	
X_1X_2	134.77	1	134.77	14.37	0.0020	
X_1X_3	274.48	1	274.48	29.27	< 0.0001	
X_1X_4	49.65	1	49.65	5.29	0.0373	
X_2X_3	0.80	1	0.80	0.085	0.7749	
X_2X_4	0.60	1	0.60	0.064	0.8041	
X_3X_4	0.57	1	0.57	0.061	0.8087	
X_1^2	28.57	1	28.57	3.05	0.1028	
X_2^2	277.34	1	277.34	29.57	< 0.0001	
X_3^2	537.51	1	537.51	57.32	< 0.0001	
X_4^2	654.52	1	654.52	69.79	< 0.0001	
Residual	131.29	14	9.38			
Lack of Fit	131.29	10	13.13	6538.13	< 0.0001	*significant*
Pure error	8.032E-003	4	2.008E-003			
Cor. Total	5665.30	29				

X_1=Solvent/solid ratio, X_2=pH, X_3=extraction time, X_4=extraction temperature, df=degrees of freedom
$p<0.01$ highly significant, $0.01 \leq p<0.05$ significant, $p \geq 0.05$ not significant.
Cor. totals of all information corrected for the mean.

From Table 2 and Table 4, it can be seen that the three highest yields were observed in the experiments 4, 18 and 21, and it was found that solvent/solid ratio of 25-30 mL (1 g colemanite)-1; 10≤pH≤12; extraction time of 3-4 h; and extraction temperature of 70-80oC had significant effects on the yield.

After multiple regression analyses were done, a second order model was generated by the software as shown in Eq. (3) below:

$$Y = 822.95209 - 12.38836X1 - 81.39258X2 - 35.27854X3 - 6.07593X4 + 0.58045X1X2 + 0.82838X1X3 + 0.035232X1X4 - 0.22325X2X3 + 0.019350X2X4 - 0.018887X3X4 + 0.040823X12 + 3.17983X22 + 4.42683X32 + 0.048850X42 \quad (3)$$

where Y is the boric acid extraction yield; X1 is solvent/solid ratio; X2 is pH; X3 is extraction time, and X4 is extraction temperature. After the insignificant regression coefficients were removed from the Eq. (3), a new model representing the response better was shown in Eq. (4):

$$Y = 822.95209 - 12.38836X1 - 81.39258X2 - 35.27854X3 - 6.07593X4 + 0.58045X1X2 + 0.82838X1X3 + 0.035232X1X4 + 3.17983X22 + 4.42683X32 + 0.048850X42 \quad (4)$$

The sum of this equation was depicted in Figure 1 which shows a powerful linear relationship between the observed and predicted values.

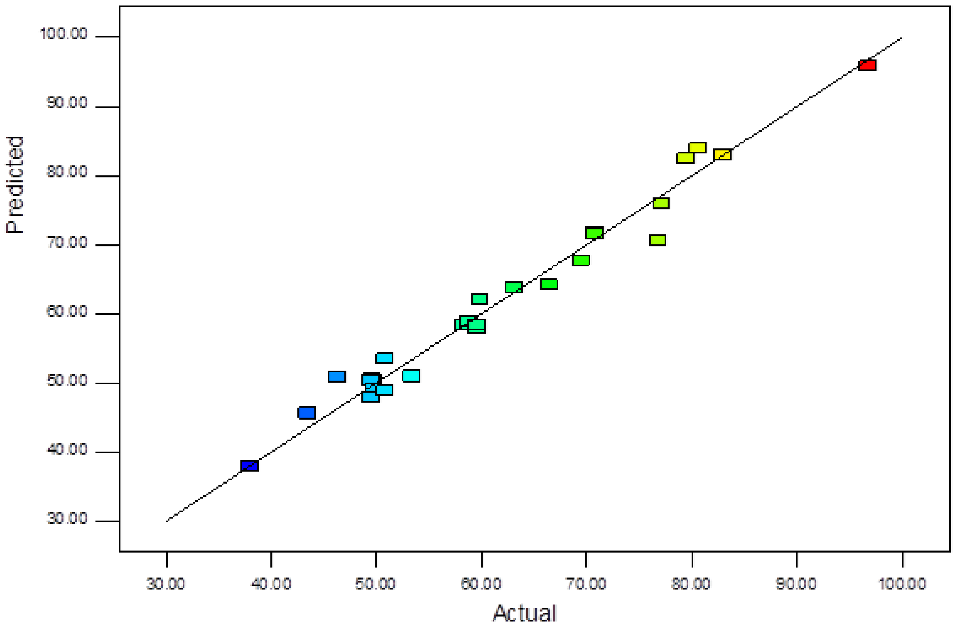

Figure 1. Parity plot of CCD showing actual and predicted values for the response.

(Left blank for a better view of Figure 2.)

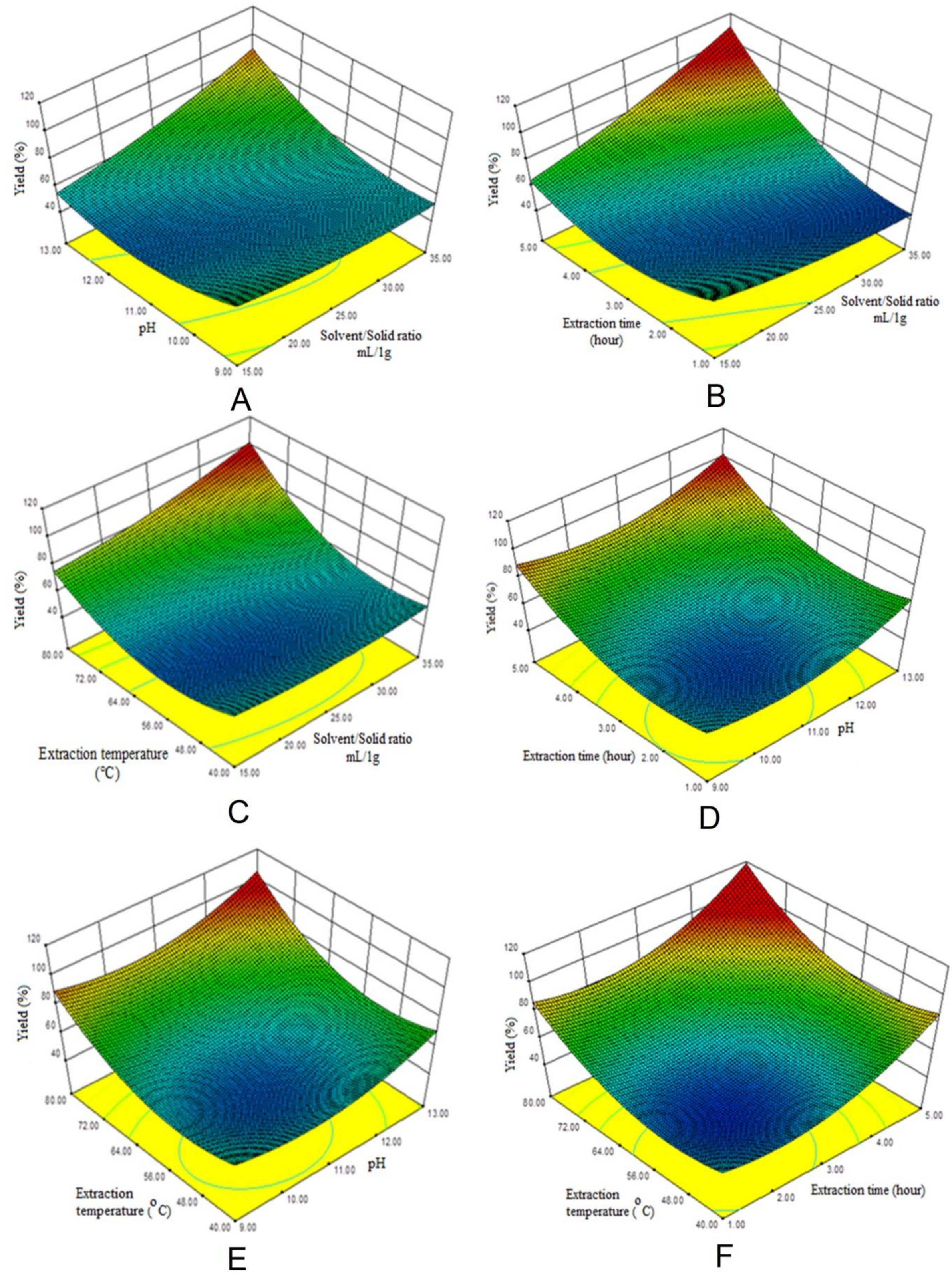

Figure 2. Response surface plots showing the individual and synergistic effects of A) pH and solvent/solid ratio (mL (1 g colemanite)$^{-1}$); B) extraction time (h) and solvent/solid ratio; C) extraction temperature (°C) and solvent/solid ratio; D) extraction time and pH; E) extraction temperature and pH; F) extraction temperature and extraction time on the boric acid extraction yield (%).

The response surface plot in the Figure 2 - A showed that more alkaline pH with higher solvent/solid ratio (which means higher volumes of HCl) increased the yield. This can be seen as red/orange color zone on the surface plot. In Figure 2 - B, when the HCl volume changes between 25 to 35 mL and extraction time kept at least 4 h, boric acid extraction yield reached to the red zone (the optimum zone). This can also be seen in Table 2, where the yield results were shown.

In case of extraction temperature and solvent/solid ratio (Figure 2 - C), a proportional relationship was observed; to obtain higher yields, higher temperature and higher HCl volume are required. In Table 4, this mutual relationship (X1X4) showed a p-value of 0.03, which was indicated as not highly-significant but significant. However, the surface plot displayed that both parameters individually had a highly-significant effects on the yield.

In Figure 2 - D, it was presented that optimum yield zone was reached if the extraction of boric acid was carried out for longer than 4 h even the pH values changes between 9 and 13. However, it can be seen that higher yields can be obtained when pH went up to 13, which was shown as a red zone having a larger area.

Moreover, the interaction between the extraction temperature and pH (Figure 2 - E) was found to be similar to the results of Figure 2 - D. This emphasized that all the pH values tested in this CCD (between 9 and 13) worked when the temperature was kept higher enough (here at least 72oC). To sum up, both figures (2 - D and 2 - E) represented that a wide range of pH may work to get a maximum yield, if temperature and/or extraction time is changed. However, these two mutual relationships were found to be insignificant (Table 4), as they both had p-values>0.7.

In case of Figure 2 - F, higher yields can be obtained when the boric acid extraction is done at temperatures>64oC for at least 3 h. This mutual interaction also had a p-value of 0.8, which is also an insignificant value. Nonetheless, unlike Figure 2 - D and Figure 2 - E, this effect was not obvious on the surface plot. This may indicate the lower importance of pH on the yield than the importances of extraction time and temperature.

On the other hand, it was previously reported by Tekin and Okur (2011) that decreasing particle size had a great effect on the colemanite dissolution rate when producing boric acid [19]. In our case, 0.075 mm (200 mesh) colemanite powder was used to extract boric acid using HCl in an ultrasonic bath. The result of using such a small size can be seen in our yield results in Table 2: even the optimization is not done in the first part of this study, one of the CCD experiments already showed a great yield value, 96.81% (in the 18th run of the CCD). This finding may be a result of UAE utilization, since it was previously claimed that ultrasound has a positive impact on the dissolution of colemanite mineral (Taylan *et al.*, 2007).

3.2. Optimization

In the present study, Design Expert software was utilized for the optimization aim. Optimum levels of four test parameters, solvent/solid ratio, pH, extraction time and temperature, were evaluated to get a maximum boric acid extraction yield from colemanite mineral particles. The optimum conditions were predicted by the software as 25.90 mL (1 g colemanite)-1, 10.76, 4.60 h, and 75.65°C, respectively. To detect whether the predicted conditions are able to augment the yield, a new set of experiments were run with six replicates, and the results were averaged.

Finally, it was found that boric acid extraction yield as 99.73%. Table 5 summarizes the optimum conditions of the four test variables and the yield.

Table 5. The conditions showed optimum boric acid extraction yield from colemanite.

Solvent/solid ratio (mL (1 g colemanite)$^{-1}$) X_1	pH X_2	Extraction time (h) X_3	Extraction temperature (°C) X_4	Yield (%) Y
25.90	10.76	4.60	75.65	99.73

In the literature, it can be seen that the conventional boric acid production from colemanite minerals is carried out at 85-90oC (Bayca, 2013) and the researchers revealed that increasing temperature results in higher yields (Künkül *et al.*, 2012). However, lower extraction temperature, 75.65oC, was found by the help of this study. In another study, researchers said that calcination step, which is typically carried out at 450-600oC, is necessary to decrease the impurities and increase the boric acid production yield (Künkül *et al.*, 2012). This kind of necessity may cause an unclean and costly production of boric acid by pollutant discharges and extra energy consumption (An and Xue, 2014). Nevertheless, our study indicated that almost 100% production yield can be achieved at the optimum conditions obtained in this study. Additionally, our study may also lead more economic and environmental friendly boric acid production.

4. CONCLUSIONS

In this study, ultrasound-assisted extraction process was employed to extract boric acid from colemanite minerals. Using a central composite design, solvent/solid ratio (15, 20, 25, 30, and 35 mL (1 g colemanite)-1), pH (9, 10, 11, 12, and 13), extraction time (1, 2, 3, 4, and 5 h), and extraction temperature (40°C, 50°C, 60°C, 70°C, and 80°C) were evaluated and the extraction yield was investigated. After a total of 30 CCD runs were completed, the results demonstrated that all the test variables were very important for the boric acid production yield. The optimum conditions were 25.90 mL (1 g colemanite)-1 for solvent/solid ratio, 10.76 for pH, 4.6 h for extraction time and 75.65°C for extraction temperature. Under these conditions, the yield of boric acid was obtained as 99.73%. This study also showed that RSM and CCD are powerful methods for boric acid production through UAE method. Compared to previous studies, this study involves clean and economic boric acid production.

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