A Comparison of Ulexite Mineral and Borax Decahydrate to Synthesize Zinc Borate

M.B. Piskin[1] and N. Baran Acaralı[2]

Abstract—**The purpose of this study is to compare the zinc borate results by using ulexite mineral and borax decahydrate. Zinc borate production was carried out by using boric acid synthesized in first stage, zinc oxide and commercial zinc borate as seed. In second stage, zinc borate samples produced were filtered, dried at 105°C for 20 h. The effect of reaction time, H_3BO_3/ZnO ratio, seed on yield have been investigated. X-ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) analysis showed that zinc borate was synthesized successfully.**

Index Terms—**Borax decahydrate, ulexite, zinc borate**

I. Introduction

Ulexite which is a sodium–calcium borate is a structurally complex mineral. Borax decahydrate is one of the most important commercial boron compounds produced in large tonnage which is sodium [1]-[3]. Zinc borates [4] are widely used in plastic, rubber, wood applications, cement etc. due to its properties [5]. Commercial zinc borate widely used is white, nonhygroscopic, viscose, powder product. The most important properties of zinc borate are low solubility in water and high dehydration temperature. Zinc borates which have commonly usage dehydrates above 290°C and borate which has been known as anhydrous zinc borate has thermal resistance about 400°C and hydrolizes with strong acid and base. Zinc borate is widely used in plastic, rubber, ceramics, paint, etc. [4], [6], [7].

In this study, zinc borate production was carried out by using ulexite mineral and borax decahydrate, comparatively. Dry, fine powdered zinc borate particles were produced with increasing yield. X-ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) analysis showed that the zinc borate was synthesized successfully.

II. Experimental

Ulexite, borax decahydrate and commercial zinc borate as seed were supplied by Eti Mine Works and the zinc oxide (97%) from Colakoglu Chemicals Ltd.

Boric acid was synthesized by using ulexite mineral or borax decahydrate (see Fig. 1) with the reaction of sulphuric acid and water at 95°C. After filtration, boric acid crystals which was formed and cooled were dried at 50°C for 24 h. Sodium sulphate which was formed by the reaction was separated from boric acid crystals by washing hot distilled water. The XRD analysis showed that boric acid was synthesized successfully.

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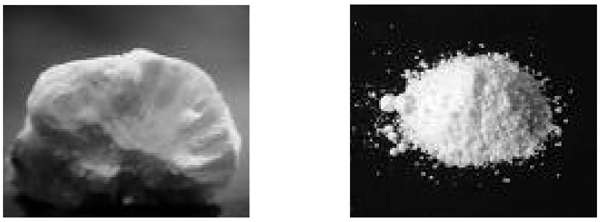

(a) (b)

Fig. 1 (a) Ulexite mineral, (b) Borax decahydrate

Zinc borate production was carried out in aqueous media using boric acid which was synthesized in first stage, zinc oxide and commercial zinc borate as seed. Experiments have been carried out in an isolated glass beaker. Temperature is controlled using a digital temperature sensor and magnetic stirrer provides constantly stirring during the reaction.

Zinc borate samples were filtered and dried at 105°C for 20 h. Dry, fine powdered zinc borate particles were produced under optimised conditions (see Fig. 2).

Fig. 2 Zinc borate synthesized

III. Results and Discussion

A. *The Effect of Process Parameters on Boric Acid Yield*

The effect of reaction time on boric acid yield was seen as in Fig. 3 by using ulexite mineral. The yield increased up to 4 hours of reaction time when reaction was under same conditions (95^{o}C, 62.5 ml of water, 500 rpm with reaction times: 2-5 hours). After 4 hours, the yield did not change any more. Therefore, 4 hours of reaction time was determined as an optimum reaction time.

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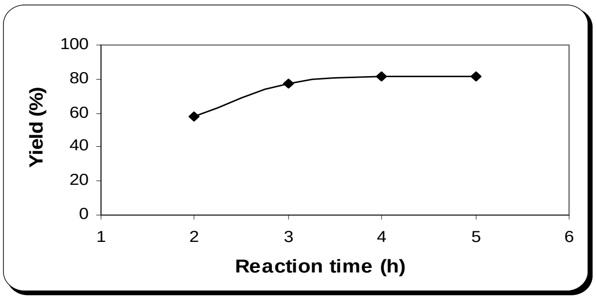

Fig. 3 The effect of reaction time on boric acid yield by using ulexite mineral (95^oC, 500 rpm)

The effect of the amount of H_2SO_4 on boric acid yield was seen as in Fig. 4 by using borax decahydrate. The yield increased up to 3 ml of sulphuric acid when reaction was under same conditions (95^oC, 500 rpm). After 3 ml of sulphuric acid, the yield did not increase any more. Therefore, 3 ml of sulphuric acid was determined as an optimum amount of H_2SO_4.

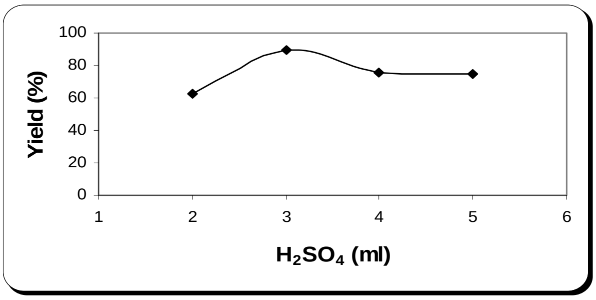

Fig. 4 The effect of the amount of H_2SO_4 on boric acid yield by using borax decahydrate (95^oC, 500 rpm)

B. The Effect of Process Parameters on Zinc Borate Yield by Usage of Ulexite Mineral

The optimum yield was 95.75% in range of 2-6 h at different reaction times. The purpose of this study was to produce zinc borate with high efficiency. As a result, the highest yield was achieved at determined stoichiometric ratio for 3 hours of reaction time. The zinc borate product was obtained like white powder as expected (see Fig. 5).

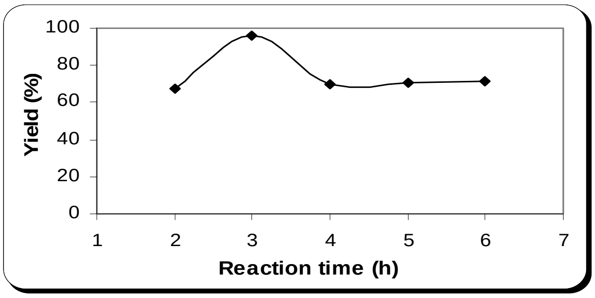

Fig. 5 The effect of reaction time on zinc borate yield by using ulexite mineral (H_3BO_3:ZnO=3.5:1, 25 ml of water, 1% of seed)

The effect of H_3BO_3/ZnO on zinc borate yield was shown in Fig. 6. The yield decreased due to increase of H_3BO_3/ZnO ratio and did not change after 5:1 point. The excess of boric acid was removed by washing and filtration.

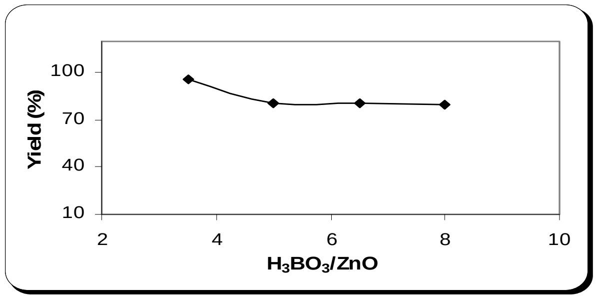

Fig. 6 The effect of H_3BO_3/ZnO ratio on zinc borate yield by using ulexite mineral (3 h, 25 ml of water, 1% of seed)

The commercial zinc borate added to the reaction as seed is dissolved and provides a saturation concentration for zinc borate, so that the zinc borate formed from the reaction can precipitate on the zinc oxide surface.

The formation of zinc borate on the surface of zinc oxide particles may be considered as nucleation followed by the growth of zinc borate crystals. At this point, the importance of seed appears. Optimum amount of seed was 1% and then the yield did not change (see Fig. 7).

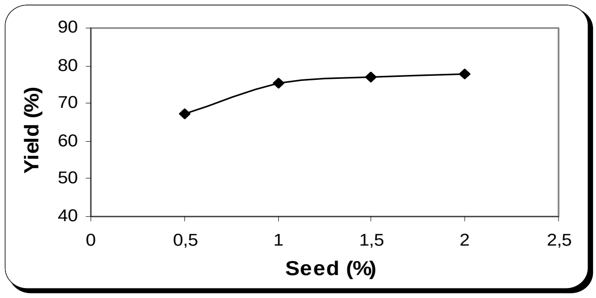

Fig. 7 The effect of seed on zinc borate yield by using ulexite mineral (H_3BO_3:ZnO=3.5:1, 3 h, 25 ml of water)

C. The Effect of Process Parameters on Zinc Borate Yield by Usage of Borax Decahydrate

The highest yield in range of 2-5 h at different reaction times was obtained at 3 hours of reaction time. The zinc borate product was obtained like white powder as usage of ulexite mineral (see Fig. 8).

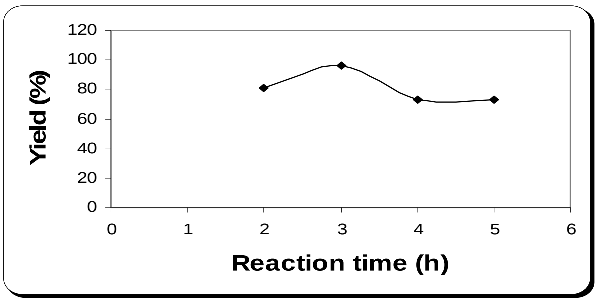

Fig. 8 The effect of reaction time on zinc borate yield by using borax decahydrate (H_3BO_3:ZnO=3.5:1, 25 ml of water, 1% of seed)

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The effect of H_3BO_3/ZnO on zinc borate yield by using borax decahydrate was shown in Fig. 9. The yield increased up to 3.5:1 point. Then it decreased and did not change after 5:1 point. Therefore, 3.5:1 point was obtained as optimum H_3BO_3/ZnO ratio.

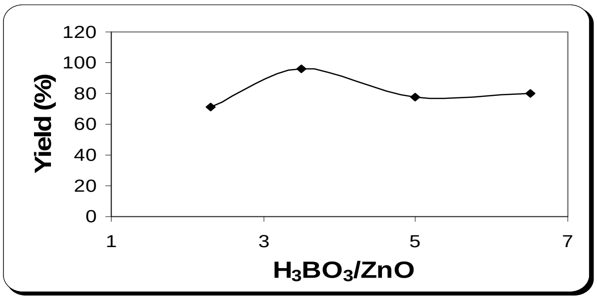

Fig. 9 The effect of H_3BO_3/ZnO ratio on zinc borate yield by using borax decahydrate (3 h, 25 ml of water, 0.5% of seed)

Commercial ZB was used as seed crystals to reduce reaction time and improve quality of the product. Because, seed indicates catalyst effect and reduces reaction time. The yield was increased due to usage of seed crystals up to 0.5% (w/w). The amount of seed above 0.5% did not affect the yield much, so optimum amount of seed was determined as 0.5% in terms of boric acid as shown in Fig. 10.

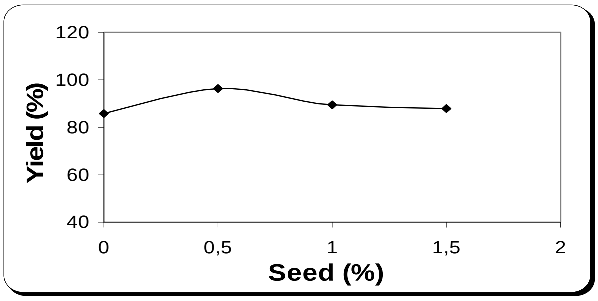

Fig. 10 The effect of seed on zinc borate yield by using borax decahydrate ($H_3BO_3:ZnO$=3.5:1, 3 h, 25 ml of water)

D. Characterization

Zinc borate peaks were similar to commercial zinc borate with peaks when we produced zinc borate by using ulexite mineral. The characteristic peaks of zinc borate were observed in range of 15-70°2θ from XRD analysis as expected (see Fig. 11, 12).

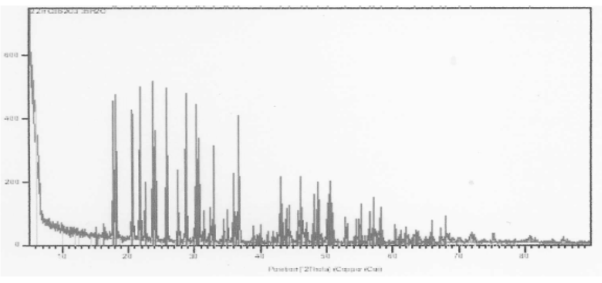

Fig. 11 XRD analysis of commercial zinc borate as seed

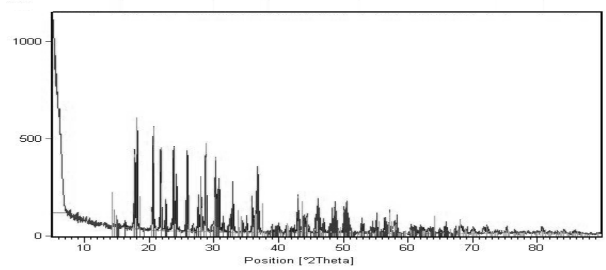

Fig. 12 XRD analysis of zinc borate by using ulexite mineral (3 h, $H_3BO_3:ZnO$=3.5:1, 25 ml of water, 1% of seed)

The increase of reaction time does not have a structural effect but it sharpens the peaks as shown in Fig. 13.

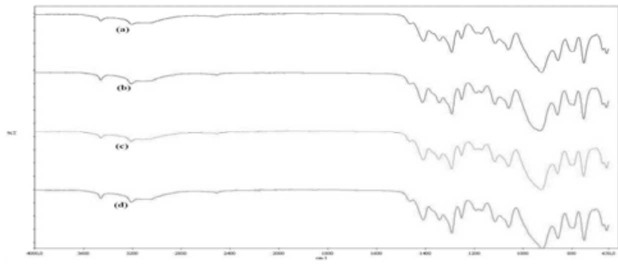

Fig. 13 FTIR analysis of zinc borate for various reaction times when ulexite mineral used in the reactions: (a) 2h, (b) 3h, (c) 4h, (d) 5h ($H_3BO_3:ZnO$=3.5:1, 25 ml of water, 1% of seed)

The characteristic peaks of zinc borate when borax decahydrate used in reactions were observed in range of 15-70°2θ from XRD analysis as zinc borate peaks when ulexite mineral used in reactions (see Fig. 14).

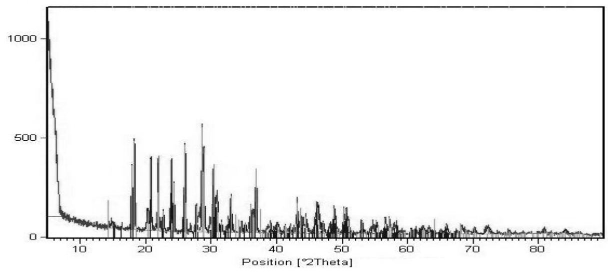

Fig. 14 XRD analysis of zinc borate by using borax decahydrate (3 h, $H_3BO_3:ZnO$=3.5:1, 25 ml of water, 0.5% of seed)

The increase of reaction time does not have a structural effect definitely as shown in Fig. 15.

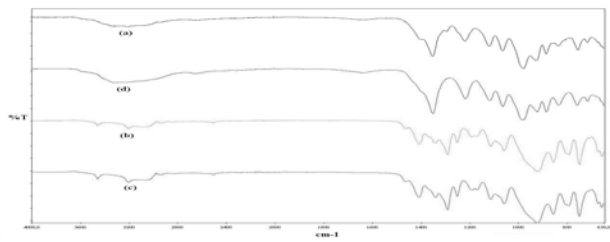

Fig. 15 FTIR spectums of zinc borate by using borax decahydrate for different reaction times: (a) 2h, (b)3h, (c) 4h, (d) 5h (H_3BO_3/ZnO=3.5:1, 0.5% of seed, 25 ml of water)

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IV. Conclusion

In conclusion, boric acid was synthesized by using ulexite mineral or borax decahydrate successfully.

Optimum reaction time for zinc borate production was 3 hours for usage of ulexite mineral or borax decahydrate.

Optimum H_3BO_3/ZnO ratio for usage of ulexite mineral was obtained 3.5:1 as the usage of borax decahydrate.

The amount of seed above 1% did not affect the yield much, so optimum amount of seed was determined as 1% (w/w) in terms of boric acid by using ulexite mineral. But, when borax decahydrate was used in reactions, the optimum amount of seed was 0.5% (w/w).

The difference of the process parameters did not effect the structure and it was observed from XRD analysis that the synthesis was carried out successfully.

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