Synthesis of Zinc Borate: Effects of Different Modifying Agents and Isopropyl Alcohol on Yield and Hydrophobicity by Using Zinc Carbonate

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Abstract. In this study, the aim of this study was to synthesize zinc borate by using different modifying agents and isopropyl alcohol in the presence of zinc carbonate, reference boric acid and seed. The experiments were carried out by dissolving of boric acid in a glass beaker in distilled water. Temperature is controlled using a digital temperature sensor and magnetic stirrer provides constantly stirring during the reaction. The effects of modifying agents and isopropyl alcohol on hydrophobicity were investigated. As a result, the analysis results showed that the usage of modifying agents with isopropyl alcohol affected the hydrophobicity of zinc borate and zinc borate which was successfully synthesized has been observed with X-ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FT-IR) analysis that was carried out after experiments.

Keywords: zinc carbonate, hydrophobicity, modifying agent, zinc borate

1. Introduction

Zinc borates have been used as flame retardant, smoke suppressant, afterglow suppressant, and anti tracking agent in both halogen-containing and halogen-free polymers. Studies of the preparation of zinc borate with nanostructures and the hydrophobic properties have been relatively few. Surface modification of zinc borate with hydrophobic properties lead to a great expansion of its applications. Zinc borate particles are hardly dispersed in a polymer matrix. Therefore, hydrophobic zinc borate is used in different applications. Therefore, the studies about hydrophobic zinc borate production become very important subject due to its large applications [1-6].

In this study, zinc borate was synthesized under optimised conditions, and the effects of various modifying agents on the yield were investigated. Seed crystals were added to the reaction medium to reduce reaction time and to improve the quality of the product. The products were characterised by analytical techniques such as FT-IR, XRD which indicated that the synthesis of zinc borate was successful and changing modifying agents and usage of different amounts of isopropyl alcohol affected the yield and hydrophobicity of zinc borate.

2. Experimental

The synthesis of zinc borate was carried out by the reaction of zinc carbonate and boric acid by using Propylene Glycol (PG), Kerosene, Oleic Acid (OA) in Isopropyl Alcohol (IPA). Reference zinc borate was used as a seed crystal to reduce reaction time and to improve the quality of the product. The experiments were carried out by dissolving of boric acid in a glass beaker in distilled water. Temperature is controlled using a digital temperature sensor and magnetic stirrer provides constantly stirring during the reaction. Dry, fine powdered zinc borate particles were produced. The XRD, FT-IR analysis showed that zinc borate was
synthesized successfully. It was determined by contact angle tests that usage of different modifying agents and isopropyl alcohol affected the hydrophobicity of zinc borate.

3. Results and Discussion

Various ratios of modifying agents and isopropyl alcohol were investigated under otherwise identical conditions (H$_3$BO$_3$/ZnCO$_3$: [4:1], 2 hours, 0.5% of seed, 95°C, 500 rpm). Modifying agents were used to improve hydrophobicity. The results revealed that the yield changed slightly after 3% value when the modifying agent were PG and Kerosene. Thus, the optimal point for PG and Kerosene was selected as 3%. In addition, the optimal point for oleic acid was determined as 1%. The isopropyl alcohol was used to disperse the modifying agents. Isopropyl alcohol which was used for different amounts provided homogenous medium for each modifying agent. The optimal points for PG, Kerosene and OA were 2, 4, 2 ml, respectively (Fig.1-3).

![Fig. 1: Effect of propylene glycol and isopropyl alcohol on yield: (a) PG, (b) IPA](image1)

![Fig. 2: Effect of kerosene and isopropyl alcohol on yield: (a) Kerosene, (b) IPA](image2)
The contact angle (Optical Contact Angle / Surface Tension Meter, Cam 200) test results for optimal points were shown as Table 1. There is improvement from hydrophilic to hydrophobic. In conclusion, it was seen that changing modifying agents affected the hydrophobicity of zinc borate.

Table 1: Contact angle test results for optimal points

<table>
<thead>
<tr>
<th>Modifying agent</th>
<th>IPA (ml)</th>
<th>Contact Angle (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PG (3%)</td>
<td>2</td>
<td>15.81</td>
</tr>
<tr>
<td>Kerosene (3%)</td>
<td>4</td>
<td>12.00</td>
</tr>
<tr>
<td>OA (1%)</td>
<td>2</td>
<td>103.19</td>
</tr>
</tbody>
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XRD (X-Ray Diffraction) (Philips Panalytical, X’Pert Pro) analysis showed that zinc borate peaks were similar to reference zinc borate with peaks. The characteristic peaks of zinc borate were observed in range of 15-70°2θ from XRD analysis as expected (Fig. 4).

FT-IR (Fourier Transform Infrared Spectroscopy) analysis was carried out by using Perkin-Elmer, Spectrum One instrument. The band which indicates stretching vibrations of O-H is obvious at 3210 cm\(^{-1}\). Bending vibrations of H-O-H band which are due to crystal water included in compound is slightly seen at 1349 cm\(^{-1}\). The presence of the band at 1220 cm\(^{-1}\) assigned to asymmetric stretching vibrations of trihedral
(BO$_3$) borate groups. The peaks at around 1063 and 1116 cm$^{-1}$ are assigned to asymmetric and symmetric stretching vibrations of tetrahedral (BO$_4$) borate groups. The peak observed at 753 cm$^{-1}$ wavelength indicates in plane bending vibrations of trihedral (BO$_3$) groups. As a result, FT-IR spectrums of the product show the formation of zinc borate (Fig. 5).

![FT-IR spectrum](image)

Fig. 5: FT-IR analysis of produced zinc borate (PG: 3% - IPA: 2 ml)

4. Conclusion

The analysis results showed that the synthesis of zinc borate was achieved. Optimum points for each modifying agent were determined. The contact angle of reference zinc borate was determined as 0° which means the reference zinc borate had a hydrophilic structure. There is improvement from hydrophilic properties to hydrophobic properties. In conclusion, it was seen that changing modifying agents and usage of different amounts of isopropyl alcohol affected the yield and hydrophobicity of zinc borate.

5. References


