Microwave Technology: An Effective Approach to Rapid Material Synthesis

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Abstract

Microwave technology is useful in materials science due to its ability to focus energy to achieve consistent reaction conditions, which are difficult to obtain under normal laboratory settings. During these reactions, water can be heated above its boiling point while the temperature-pressure relationship of this superheated liquid is monitored. It has been demonstrated through water tests that the instrumental temperature-pressure data agrees with theoretical results to confirm the accuracy of this method. One project currently being undertaken with this technology is the synthesis of MgFe₂O₄, which takes advantage of the microwaves’ capabilities for finely tuning reaction parameters to yield desirable results. XRD data has confirmed the success of this synthesis and can be used to study products from future manipulations of reaction conditions.

Background

- **Figure 1:** The Discover SP Microwave Synthesis System
- **Figure 2:** Advantages and disadvantages of microwave synthesis.
- **Figure 3:** Conventional heating vs. microwave heating

<table>
<thead>
<tr>
<th>Conventional</th>
<th>Microwave</th>
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<tbody>
<tr>
<td>inconsistent heating → temperature variation in solution</td>
<td>Microwaves absorbed throughout solution → mostly homogeneous heating</td>
</tr>
<tr>
<td>performed under atmospheric conditions → limits temperature and pressure conditions</td>
<td>performed in sealed vessel under extreme conditions → temperature and pressure can reach superheated conditions</td>
</tr>
<tr>
<td>Microwave radiation does not have enough energy to break bonds</td>
<td>Microwaves induce rotational motion in molecules</td>
</tr>
<tr>
<td>Mechanism of heating is that molecules orient their dipole moment along oscillating electric field. Collisions between the molecules are much faster than rotations</td>
<td>Collisions and friction between water molecules produces thermal energy</td>
</tr>
<tr>
<td>Some inorganic materials susceptible to thermal runaway: The dielectric constant, which is dependent on temperature, determines rate of heating. This can cause localized points of superheating because higher temperature areas are able to heat faster</td>
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Water tests

- In order to take advantage of the precision microwave instruments provide, the temperature and pressure monitors within the instrument must be precise.
- Cross referencing of temperature and pressure data for a pure solvent with known thermodynamic data (e.g. water) provides a method for calibration and investigation into the accuracy of the temperature and pressure probes.

**Table:**

<table>
<thead>
<tr>
<th>Observed Temperature</th>
<th>Pressure</th>
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<tbody>
<tr>
<td>160°C</td>
<td>Observed: 84 PSI</td>
</tr>
<tr>
<td></td>
<td>Expected value: 90 PSI</td>
</tr>
</tbody>
</table>

**Figure 4:** Sample water test showing temperature/pressure relationship for water.

There is agreement between experimental and literature patterns. However, the signal to noise ratio in both patterns is not ideal, suggesting small crystallite size.

Results

- Microwave instruments are a novel piece of technology that provide many practical advantages for inorganic synthesis. The nature of this instrument provides advantages over conventional heating, especially the ability to reach superheated conditions. Thus it is important to precisely determine the temperature and pressure relationship within the reaction vessel. Although the temperature and pressure probes show some inconsistencies compared to theoretical values, this is most likely due to the method used to measure these values.
- Preliminary studies of MgFe₂O₄ show promising results. Future studies will center on further variation of reaction conditions using the microwave to change crystal properties of the material. After the synthesis is optimized, this material will be tested electrochemically for applications as a cathode in batteries.

Conclusions

Experimental

**Mg²⁺ + 2 Fe³⁺ + 4 H₂O → MgFe₂O₄ + 8 H⁺**

Fe(NO₃)₃ and Mg(NO₃)₂ are combined in a 2:1 ratio and dropped into excess KOH. The reaction is carried out in a Discover SP microwave for varying amounts of time and at different temperatures (typically around 150°C for 30 minutes).

The product is washed, ground and packed for analysis using power X-Ray Diffraction.

Acknowledgements

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References