Formation of Barium Carbonate by the Carbonation Method

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In the electron-tube the oxide-coated cathode is used to increase the thermionic emission, and for this purpose Ca-Sr-Ba-triple carbonate is coated on the tungsten filament. Previously, one of the authors studied on the formation of ultrafine Ca- and Mg-carbonates by the carbonation method (this Bull. 18, 115, 117 (1940); 26, 80 (1951)). In this experiment the kinetics of the carbonation of Ba(OH)$_2$ by CO$_2$ gas was studied and the shape and size of BaCO$_3$ were observed by electron microscope.

(A) Kinetics of the Reaction. CO$_2$ gas was introduced to Ba(OH)$_2$ solution at constant velocity, and pH of the reactant was measured every 30 seconds. The change of the concentration of Ba(OH)$_2$ was measured by the titration with 0.1 N HCl. In Table 1 the conditions of the formation of BaCO$_3$ are shown collectively.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Temperature (°C)</th>
<th>Concentration of Ba(OH)$_2$ solution (M)</th>
<th>Concentration of CO$_2$ gas (%)</th>
<th>Size of BaCO$_3$ precipitate (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10</td>
<td>0.100</td>
<td>100</td>
<td>0.1—1.0</td>
</tr>
<tr>
<td>2</td>
<td>20</td>
<td>0.100</td>
<td>100</td>
<td>0.2—2.0</td>
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<tr>
<td>3</td>
<td>30</td>
<td>0.100</td>
<td>100</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>10</td>
<td>0.100</td>
<td>25</td>
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</tr>
<tr>
<td>5</td>
<td>10</td>
<td>0.224</td>
<td>100</td>
<td></td>
</tr>
<tr>
<td>6</td>
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<td>0.100</td>
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<tr>
<td>8</td>
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<td>0.265</td>
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<td>0.265</td>
<td>25</td>
<td>0.5—2.0</td>
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<tr>
<td>10</td>
<td>30</td>
<td>0.352</td>
<td>100</td>
<td>2.0—9.0</td>
</tr>
</tbody>
</table>

* The flowing velocity was kept constant, 7.3 cc/sec.
** The width of all the crystals is 0.1 µ. Only the length is indicated here.

At an early stage of the reaction pH increased somewhat and the quantity of Ba (OH)$_2$ decreased with the reaction, which was probably the first order, then pH decreased about 1 until the concentration of Ba(OH)$_2$ decreased to 1/10 of the initial concentration, and pH decreased sharply during the reaction of residual Ba(OH)$_2$ to 6.5 as shown in Figs. 1 and 2.

As shown in Fig. 2 the higher the temperature, the slower the reaction velocity is. CO$_2$ used for the reaction was 100 and 25 %, which was diluted with air. With increase of the concentration of CO$_2$ the reaction velocity became faster and the reaction
time decreased. The higher the concentration of Ba(OH)$_2$ the slower the reaction velocity was.

It seemed that the solubility of CO$_2$ gas is the most dominant to the velocity of the reaction.

(B) Morphology of Barium Carbonate Precipitates. The precipitated BaCO$_3$

Photo 1. BaCO$_3$ (No.8) ×10,000
was observed and photographed by electron microscope. Various shapes and sizes of CaCO₃ were formed by changing the precipitating conditions, but the shape of BaCO₃ were almost unchanged by the reaction conditions. The shape of BaCO₃ was rod and the width was constant, 0.1 μ, and the length were 0.1 ~ 2.0 μ. One of them is shown in Photo 1. The size was scarcely changed by the reaction condition, but it was somewhat larger, when the temperature and the concentration of CO₂ were higher and the concentration of Ba(OH)₂ was lower as shown in Table 1. No aging effect was observed for 3 months. The precipitate turned to coagulated form by filtering and drying.

8. A Method of Sampling for Obtaining the Daily Average of Batch Composition

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Introducing the statistical treatment recently developed for the analysis of coal etc. to that of glass batch the method of sampling and dividing for finding out the statistically acceptable daily average of the batch composition as well as the method of routine analysis suitable for getting the results within the practical time restriction

Steps

A(76.8kg)..................................................................B(76.8kg)
2 §A₁ (38.4kg)
3 §(19.2kg)
3 §(9.6kg)
5 §(4.8kg)
6 §(2.4kg)
7 §(1.2kg)
8 §(0.6kg) A₁(Drying and Milling one hour)
9 §A₁₁
10 §A₁₁: (300gr)
11 §(150g)
12 §(75g)
13 §(38g)
13 §(19g)

(374)