A study of silica separation in the production of activated carbon from rice husk in Viet Nam

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Abstract

Environmental pollution due to mismanagement of the waste from rice production is a serious problem in agricultural countries where rice cultivation occupies the largest proportion of the crops produced, including in Viet Nam. At the same time the use of activated carbon as an adsorption agent for water or gas purification is rapidly increasing due to the development of industry and technology in the developed countries. In this paper we consider the process of separating silica from charcoal, which is an important step in the production of activated carbon from rice husks in Viet Nam. The efficiency of the process rises with increasing temperature up to 133°C, the ratio of alkali/charcoal up to 0.6 and sodium hydroxide concentration up to 6 M as well. A regression equation has been obtained, which allows describing the influence of the parameters on the degree of silica separation from the carbon. Under optimal values of process parameters, the efficiency reaches up to 95.6%

Keywords: rice husks; activated carbon; silica oxide; absolute method of burning

1. Introduction

Rice is one of the most popular foods in the world. It has the highest gross harvest, and it occupies the second largest area of cultivated crops, after wheat. In 2013, according to the general statistics office of Viet Nam, the productivity of rice in Viet Nam was 45 million tons and will continue to increase in the near future. During the

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processing of rice for export and domestic consumption, large quantities of husks are produced. Husk, which
occupies about 20% by weight of rice, is considered to be a waste product of production. Therefore, the search for
effective ways to convert the waste of rice production into activated carbon is actually a task of great importance in
protecting the environment and improving incomes of the people of rice producing states.

Currently, there are several ways of utilizing and processing rice husks. For example, the creation of special
dumps for rice waste, the addition of rice husks in construction materials as additives, burning rice husks, or the use
of rice husks in the manufacture of fuel briquettes, in the tire and cement industries. Processing of rice husk into the
activated charcoal was reported in a few researches. In the studies 1,2 activated carbon is obtained by activation of
rice husk ash with various reagents. In these methods, silica was not separated before activation and the purity of
activated carbon obtained in these processes was not mentioned. On the other hand, the presence of silica in the
carbon does not allow for the formation of activated carbon with a high purity. In other studies 3,4,5 silica dioxide was
obtained by oxidation of rice husk ash at optimum temperatures. With these methods it is possible to obtain high-
purity silica, but under these methods a large amount of carbonaceous material in the husks is not utilized. We
propose a different way of processing rice husks, using which it is possible to obtain activated carbon, and silica in a
single production. The aim of this work is also to improve the efficiency of the separation of silica from charcoal.

The method consists in the following stages: the burning of raw materials at optimum temperatures, the
separation of the silica from the ashes, the activation of the produced carbon and the formation of silica. The
schematic diagram of the process is illustrated in Figure 1.

The object of the study is rice husk formed at the rice processing of area of the Red River Delta of Viet Nam.
Average representative samples were collected at the processing enterprises.

In our previous work 6, the process of thermal decomposition of the rice husk was studied in detail. In this work
the focus has been on the process of separating silica, because after incineration, the ash contains approximately
45% SiO₂, which does not allow the production of activated carbon with a high purity.

![Fig. 1. Schematic diagram of the production of activated carbon and silica for rice husk](image)

2. Experimental

Two methods of separation were used: a physical method and a chemical method. The physical method is based
on the difference in the densities of silica and carbon, which are 2.65 and 1.8÷2.1 g/cm³, respectively. After grinding
to the required size, the ashes are loaded into a separating system, which consists of a vertical tube, the upper part of
which is connected to the air suction pump. Silica and carbon particles with the same diameter have different
resistance forces. Under the airflow (i.e. at different flow rates) carbon particles, due to their lower density, will
move up and collect in a chamber for the product. This product is called enriched carbon (M₁). Silica particles have
a higher density and will collect at the bottom of the tube and are directed to another chamber for product. This
product is called silica-rich (M₂). Comparison of carbon content before and after the separation in the products M₁
and M₂ allows assessing the efficiency of the separation process. The experimental results are shown in Tables 2 and 3.

The Chemical separation method is based on the interaction between silica and sodium hydroxide, described by the following reaction:

\[
\text{SiO}_2 + 2\text{NaOH} \rightarrow \text{Na}_2\text{SiO}_3 + \text{H}_2\text{O}
\]  

(1)

Ash, after incineration, is subjected to grinding in a ball mill. The particle-classification according to the diameter is presented in Table 1. The separation process is carried out in an autoclave to provide the necessary pressure.

<table>
<thead>
<tr>
<th>Particle diameter (mm)</th>
<th>1</th>
<th>0.63</th>
<th>0.43</th>
<th>0.32</th>
<th>0.25</th>
<th>0.16</th>
<th>0.1</th>
<th>&lt;0.1</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass percentage (%)</td>
<td>0.11</td>
<td>0.91</td>
<td>0.29</td>
<td>2.44</td>
<td>0.04</td>
<td>6.36</td>
<td>5.87</td>
<td>83.98</td>
<td>100</td>
</tr>
</tbody>
</table>

In the course of experiments the effect of alkali concentration, the ratio of alkali/ash and pressure on the efficiency of the separation process was examined. In order to reduce the number of experiments a factorial experiment was made based on the preliminary results, using the following values of the variables: concentration of alkali (3–6 M), the ratio of alkali/ash (0.3–0.6), pressure (1 to 3 at), which corresponds to the temperature range (100–133°C). After grinding, ashes are mixed with sodium hydroxide in predetermined ratios. The resultant mixture is loaded into an autoclave and water is added in an amount calculated from the predetermined concentration of sodium hydroxide. Experiments were carried out in an autoclave under a predetermined pressure for 1 hour. After the separation the resulting mixture was filtered and washed. The resulting carbon was dried at a temperature of 90°C for 24 hours, and the filtrate was neutralized with acid followed by filtration and washing, and then subjected to heat treatment so as to produce silicon dioxide.

Comparison of carbon content before and after the separation allows us to evaluate the influence of parameters on the efficiency of the process. The experimental results are shown in Table 4.

3. Results and discussion

3.1 Physical method of separation

<table>
<thead>
<tr>
<th>Airflow rate (m³/hr.)</th>
<th>0.23</th>
<th>0.5</th>
<th>1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Input mass (g)</td>
<td>100</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mass M₁ (g)</td>
<td>18</td>
<td>21</td>
<td>23</td>
</tr>
<tr>
<td>Mass M₂ (g)</td>
<td>82</td>
<td>79</td>
<td>77</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Airflow rate (m³/hr.)</th>
<th>Initial carbon content (%)</th>
<th>M₁</th>
<th>M₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.23</td>
<td>55</td>
<td>62.5</td>
<td>44</td>
</tr>
<tr>
<td>0.5</td>
<td>55</td>
<td>57.72</td>
<td>51</td>
</tr>
<tr>
<td>1</td>
<td>55</td>
<td>59.7</td>
<td>49</td>
</tr>
</tbody>
</table>
From Tables 2 and 3 it is seen that the carbon content of the product M₁ increased in all modes in comparison with the original content, and carbon content decreased in the product M₂. With increasing air flow M₁ yield increases but only slightly, and its carbon content decreases. The physical separation method proved to be ineffective as silica in charcoal not only exists as individual particles but also in the form of different compounds with carbon. In addition, the particles have different diameters, i.e. carbon particles with a large diameter can enter the product M₂.

2.2. Chemical method separation

Table 4. Results of experiments on the separation of the silica by chemical method

<table>
<thead>
<tr>
<th>T,°С</th>
<th>C, M alkali/charcoal</th>
<th>Ratio of alkali/ash</th>
<th>Content of SiO₂ before separation % mass</th>
<th>Content of SiO₂ after separation, % mass.</th>
<th>Degree of separation , %</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>3</td>
<td>0.3</td>
<td>45</td>
<td>40</td>
<td>11</td>
</tr>
<tr>
<td>133</td>
<td>3</td>
<td>0.3</td>
<td>45</td>
<td>15</td>
<td>67</td>
</tr>
<tr>
<td>100</td>
<td>6</td>
<td>0.3</td>
<td>45</td>
<td>38</td>
<td>16</td>
</tr>
<tr>
<td>133</td>
<td>6</td>
<td>0.3</td>
<td>45</td>
<td>22</td>
<td>51</td>
</tr>
<tr>
<td>100</td>
<td>3</td>
<td>0.6</td>
<td>45</td>
<td>36</td>
<td>20</td>
</tr>
<tr>
<td>133</td>
<td>3</td>
<td>0.6</td>
<td>45</td>
<td>18</td>
<td>60</td>
</tr>
<tr>
<td>100</td>
<td>6</td>
<td>0.6</td>
<td>45</td>
<td>23</td>
<td>48.9</td>
</tr>
<tr>
<td>133</td>
<td>6</td>
<td>0.6</td>
<td>45</td>
<td>2</td>
<td>95.6</td>
</tr>
</tbody>
</table>

The extreme levels of temperature and concentration of sodium hydroxide is selected on the base of the results of preliminary experiments, thermodynamic constraints of the temperature rise, economic considerations and safety.

The regression equation describing the process is as follows:

\[ y = b_0 + b_1 X_1 + b_2 X_2 + b_3 X_3 + b_{12} X_{12} + b_{13} X_{13} + b_{23} X_{23} + b_{123} X_{123}, \]  (2)

where: y - degree of separation; X₁ - temperature; X₂ - alkali concentration; X₃ - ratio of alkali/ash.

After removal of insignificant coefficients, the following regression equation was obtained:

\[ y = 46.1 + 22.22 X_1 + 6.67 X_2 + 10 X_3 + 9.44 X_{23} - 3.333 X_{123}. \]  (3)

From the equation it is seen that temperature has the greatest impact on the degree of separation (coefficient b₁ has the highest value). Increasing the temperature increases the degree of separation. At a temperature of 100°C, the ratio of NaOH/ash=0.3 g/g and C₉NaOH=6M, the degree of separation reaches only 16%. By increasing the ratio of NaOH/ash to 0.6 g/g, the degree of separation has already reached 48.9%. Increasing the temperature to 133°C considerably increases the degree of separation to 51% and 95.6%, respectively. A further increase in temperature (pressure) is dangerous when performing the experiment. The significant influence of temperature on the degree of separation is due to the fact that reaction 1 is an endothermic reaction (\( \Delta H = 55.5 \text{ KJ/mol} \)), hence, increasing the temperature is favorable for product formation. In addition, the increase in temperature leads to the intensification of the process of separation.

Increasing the concentration of alkali increases the degree of separation. At a temperature of 133°C, the ratio of alkali/ash=0.6 g/g and by increasing the concentration of alkali from 3M to 6M increases the degree of separation from 60 to 95.6. A further increase in concentration causes a difficulty in filtering at the next stage of the processing.

The ratio of alkali/ash has a significant impact on the degree of separation. An increase in this ratio increases the degree of separation. From equation 1 it is seen that, the separation of 60g SiO₂ requires 80g NaOH. The separation of 45g of SiO₂ (100g ash) requires 60g of NaOH. That means the ratio of alkali/ash is 0.6 g/g. Therefore, when the ratio of alkali/ash increases from 0.3 to 0.6 g/g the degree of separation increases dramatically. The further increase
is disadvantageous, as experimenting with the ratio of alkali/ash =0.9 g/g showed that the degree of separation increases slightly, while a large amount of alkali is used thus reducing the economic efficiency of the process.

4. Conclusion

Production of activated carbon from Vietnam rice husk is an important task because this material can be widely used in wastewater treatment processes and adsorption of harmful impurities from waste gases of various plants, including rice processing. To obtain activated carbon with a high purity, silica must be separated from the rice husk ash before activation. This may be accomplished by a physical method, but the efficiency is very low. High efficiency and a significant degree of separation are accomplished in the chemical method. The optimum conditions for separation are: temperature 133°C; alkali concentration of 6M; ratio of alkali/ash of 0.6 g/g and separation time of 1 hour. The maximum degree of separation is up to 95.6%.

References