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ScienceDirect

Procedia Chemistry 15 (2015) 308 – 312

Procedia Chemistry

16th International Scientific Conference "Chemistry and Chemical Engineering in XXI century" dedicated to Professor L.P. Kulyov, CCE 2015

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%

Peer-review under responsibility of Tomsk Polytechnic University

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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1876-6196
Peer-review under responsibility of Tomsk Polytechnic University
doi:10.1016/j.proche.2015.10.049

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% SiO_2, which does not allow the production of activated carbon with a high purity.

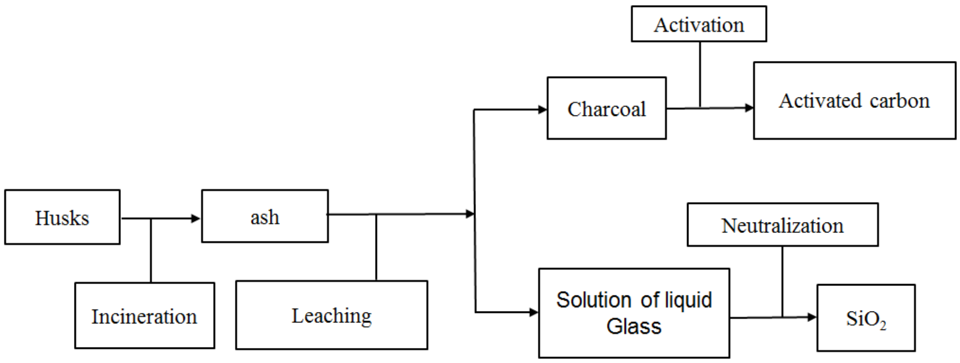

Fig. 1. Schematic diagram of the production of activated carbon and silica for rice husk

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/cm^3, 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 (M_1). 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 (M_2). Comparison of carbon content before and after the separation in the products M_1

and M_2 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:

$$SiO_2 + 2Na(OH) \rightarrow Na_2SiO_3 + H_2O \quad (1)$$

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 1. Classification of ash particles by diameter

Particle diameter (mm)	1	0.63	0.43	0.32	0.25	0.16	0.1	<0.1	Total
Mass percentage (%)	0.11	0.91	0.29	2.44	0.04	6.36	5.87	83.98	100

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 2. Dependence of the content of separated products on the air flow

Airflow rate (m^3/hr.)	0.23	0.5	1
Input mass (g)		100	
Mass M_1 (g)	18	21	23
Mass M_2 (g)	82	79	77

Table 3. Dependence of the carbon content of the products M_1 and M_2 on the air flow before and after separation

Airflow rate (m^3/hr.)	Initial carbon content (%)	M_1	M_2
0.23	55	62.5	44
0.5	55	57.72	51
1	55	59.7	49

From Tables 2 and 3 it is seen that the carbon content of the product M_1 increased in all modes in comparison with the original content, and carbon content decreased in the product M_2. With increasing air flow M_1 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 M_2.

2.2. Chemical method separation

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

T,°C	C, M	Ratio alkali/charcoal	Content of SiO_2 before separation % mass	Content of SiO_2 after separation, % mass.	Degree of separation , %
100	3	0.3	45	40	11
133	3	0.3	45	15	67
100	6	0.3	45	38	16
133	6	0.3	45	22	51
100	3	0.6	45	36	20
133	3	0.6	45	18	60
100	6	0.6	45	23	48.9
133	6	0.6	45	2	95.6

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:

$$y=b_0+b_1X_1+b_2X_2+b_3X_3+b12X_{12}+b_{13}X_{13}+b_{23}X_{23}+b_{123}X_{123} , \tag{2}$$

where: y- degree of separation; X_1- temperature; X_2- alkali concentration; X_3- ratio of alkali/ash.

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

$$y=46.1+22.22X_1+6.67X_2+10X_3+9.44X_{23}-3.333X_{123} . \tag{3}$$

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 C_{NaOH}=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 (ΔH = 55.5 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 SiO_2 requires 80g NaOH. The separation of 45g of SiO_2 (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%.

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