Preparation and Optimization of Natural Composite Oil Absorbing Materials

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Abstract. In this paper, by using the graft copolymerization method, butyl methacrylate and styrene were used as grafting monomers, benzoyle peroxide as initiator, and methylene bisacrylamide as cross-linking agent, respectively, for natural waste bagasse. The powder (RMC: Natural waste bagasse powder) was chemically modified to determine the grafting monomer, initiator, amount of cross-linking agent, and the most suitable reaction time, temperature and other conditions through orthogonal tests, thereby obtaining two kinds of natural composite efficient oil absorption materials BMC (Natural oil absorption material).

1. Introduction
While China’s economy is pursuing high-speed GDP growth, it has also brought with it a series of ecological and environmental pollution issues. The problem of marine pollution is particularly acute. The cellulose in bagasse is a very important renewable resource. Therefore, it is of practical significance to carry out energy production of bagasse through an energy-saving and efficient method. The search for an economical, efficient, and environmentally friendly technology to remove oil pollutants is an urgent task facing the current treatment of offshore oil pollution. At present, there are many methods to deal with oil pollution. This paper regards adsorb oil.[1] Chemical modification of dried and ground bagasse powder by graft copolymerization. The modification conditions were optimized to determine the optimal time, temperature, graft monomer content and cross linker and other modification conditions, so as to study the preparation of highly efficient oil adsorption materials.

2. The modified experiment

2.1. Experimental reagents and equipment
The reagents and instruments used in the experiments are shown in Table 1 and Table 2:
Table 1. Reagents of experiment.

<table>
<thead>
<tr>
<th>Reagent</th>
<th>Technical level</th>
<th>Factory</th>
</tr>
</thead>
<tbody>
<tr>
<td>Butyl methacrylate</td>
<td>Analytical purity</td>
<td>Tianjin Damao Chemical Reagent Factory</td>
</tr>
<tr>
<td>Methylenebisacrylamide</td>
<td>Analytical purity</td>
<td>Tianjin Aolan Fine Chemical Research Institute</td>
</tr>
<tr>
<td>Benzyol peroxide</td>
<td>Analytical purity</td>
<td>Tianjin Fuchen Chemical Reagent Factory</td>
</tr>
<tr>
<td>Toluene</td>
<td>Analytical purity</td>
<td>Sinopharm Group Chemical Reagent Co., Ltd.</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>99.9% High purity</td>
<td>Dalian High Purity Nitrogen Plant</td>
</tr>
<tr>
<td>Acetone</td>
<td>Analytical purity</td>
<td>Tianjin Damao Chemical Reagent Factory</td>
</tr>
<tr>
<td>Anhydrous ethanol</td>
<td>Analytical purity</td>
<td>Sinopharm Group Chemical Reagent Co., Ltd.</td>
</tr>
<tr>
<td>Pure water</td>
<td>purity</td>
<td>Dalian Ocean University Chemical Analysis Laboratory</td>
</tr>
</tbody>
</table>

Table 2. Apparatus of experiment.

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Production model</th>
<th>Factory</th>
</tr>
</thead>
<tbody>
<tr>
<td>Collector type magnetic stirrer</td>
<td>DF-101S</td>
<td>Jiangsu Jintan Zhengji Instrument Co., Ltd.</td>
</tr>
<tr>
<td>Analytical electronic balance</td>
<td></td>
<td>METTLER TOLEDO Instrument Co., Ltd.</td>
</tr>
<tr>
<td>Electric blast drying box</td>
<td>101</td>
<td>Shanghai Experimental Instrument Factory</td>
</tr>
<tr>
<td>Diaphragm vacuum pump</td>
<td>GM-0.33Ⅱ</td>
<td>Tianjin Jinteng Experimental Equipment Co., Ltd.</td>
</tr>
<tr>
<td>Chinese medicine machinery grinder</td>
<td>RH-800</td>
<td>Zhejiang Ronghao Industry and Trade Co., Ltd.</td>
</tr>
<tr>
<td>Soxhlet extractor</td>
<td></td>
<td>Shenyang Chemical Instrument Factory</td>
</tr>
</tbody>
</table>

2.2. Experimental method

The waste bagasse after the juice was washed with tap water and pure water in this order, dried in an oven, and pulverized into a powder with a pulverizer. The desired material was screened using a 40 mesh (particle size approx. 0.425 mm) sieve and placed in a desiccator for use.

Weigh 3.00g of spare raw material bagasse (particle size is about 0.5mm) into a 500mL three-necked flask with an electronic balance, and pour 300mL of pure water into a three-necked bottle containing bagasse powder and put it into a thermostatic magnetic stirrer. Three bottles of high-purity nitrogen were introduced into the bottle for 10 minutes to drive off the air in the bottle.[2] The amide, as well as the quantitative grafting monomer, butyl methacrylate, react for a certain period of time. In the experiment, the entire set of experimental devices was placed in a fume hood. During the reaction process, the constant nitrogen flow was always maintained. The purpose was to avoid the presence of air, which interrupted the polymerization reaction and affected the smooth progress of the reaction. After the reaction was completed, the nitrogen valve and the thermostatic water bath stirrer power were turned off, the three-necked flask was taken out, and the membrane vacuum pump was used to
remove heat, while washing with ethanol and pure water several times. The filtered modified material was placed in a glass petri dish and placed in a 60°C oven for 24 hours to dry. The dried material was Soxhlet extracted with toluene for 12 hours in order to remove the homopolymer formed by the reaction and obtain a purified modified material. After washing several times with ethanol and pure water, it was dried in an oven at 60°C for 24 hours to obtain a purified modified material.

3. Results and discussion

3.1. Effect of reaction temperature on chemical modification of materials.

In this group of experiments, the reaction time was set to 3 h, 4 ml of butyl methacrylate graft monomer was added, 0.2 g of initiator benzoyl peroxide, and 0.02 g of crosslinker methylene bisacrylamide. Then the graft copolymerization reaction was carried out at the reaction temperature of 45°C, 55°C, 65°C, 75°C, 85°C, and 95°C, and the oil absorption of the modified material in pure diesel fuel was measured at different reaction temperatures in order the amount.

![Figure 1. Effect of temperature on BMC.](image)

It can be seen from Figure 1 that the oil absorption of the modified material BMC changes with the temperature of the graft copolymerization reaction. When the reaction temperature is 45°C, the oil absorption of the modified material is 5.50 g/g. With the gradual increase of the temperature, the oil absorption of the modified material gradually increases. When the temperature is 85°C, the oil absorption of the modified material reaches a maximum of 12.48 g/g. When the reaction temperature continues to increase, the oil absorption of the modified material not only does not increase, but gradually decreases. From this, it can be seen that the reaction temperature increases and the graft copolymerization reaction rate increases, but when the reaction temperature is too high, the reaction rate decreases, so that the oil absorption rate of the prepared modified material decreases.

3.2. Effect of reaction time on chemical modification of materials

In this group of experiments, the reaction temperature was set to 85°C, 4 mL of butyl methacrylate grafting monomer, 0.2 g/g of initiator benzoyl peroxide, and 0.02 g of crosslinker methylene bisacrylamide were added. Then at the reaction temperature of 1h, 2h, 3h, 4h, 5h, 6h, the graft copolymerization reaction was carried out and the oil absorption of the modified material pure diesel oil under different conditions was determined in order.
As can be seen from Figure 2, the oil absorption of the modified material changes with the graft copolymerization reaction time. When the reaction time is 1 h, the modified material has an oil absorption of 6.85 g/g. With the gradual increase of time, the oil absorption of the modified material also gradually increased. When the time was 4 hours, the oil absorption of the modified material reached a maximum of 13.02 g/g. When the reaction time continues to increase, the oil absorption of the modified material remains basically unchanged, indicating that the graft copolymerization reaction has reached its maximum equilibrium.

3.3. Effects of grafting monomers on chemical modification of materials
In this group of experiments, the reaction temperature was set at 85°C, the reaction time was 3 hours, 0.2 g of benzoyl peroxide was added as initiator, and 0.02 g of methylene bisacrylamide was used as the crosslinking agent. Then the graft copolymerization reaction was performed under the conditions of 1 ml, 2 ml, 4 ml, 6 ml, 8 ml, and 10 ml of graft monomer, and the oil absorption of the modified material in the pure diesel oil was determined sequentially under the grafting monomer condition.

As can be seen from Figure 3, when the grafting monomer butyl methacrylate was added in an amount of 1 mL, the oil absorption of the modified material was 7.85 g/g. When the amount of
grafting monomer is 2 mL, the maximum balance of the oil absorption of the modified material is 11.85 g/g. When the amount of grafted monomer continues to increase, the oil absorption of the modified material BMC begins to gradually decrease.

3.4. Effect of initiators on chemical modification of materials

In this group of experiments, the reaction temperature was set to 85° C, the reaction time was 3 hours, and 4 mL of the grafting monomer butyl methacrylate and 0.02 g of the cross-linking agent methylene bisacrylamide were added. Then, the reaction was carried out under the conditions of 0.1 g, 0.2 g, 0.3 g, 0.4 g, and 0.5 g of initiator benzoyl peroxide, and the oil absorption of different modified materials in pure diesel oil was measured sequentially.

![Figure 4. Effect of initiator’s amount on BMC.](image)

As can be seen from Figure 4, when the amount of initiator benzoyl peroxide is 0.1 g, the oil absorption of the modified material is 12.20 g/g, with the increase of the dose, the modified material BMC. The oil absorption rate gradually decreases. The reason is that an appropriate amount of initiator can accelerate the reaction rate. However, when the amount of initiator is increased, the amount of primary radicals generated per unit time will be increased, thereby increasing the probability of termination reaction of the activity of the fiber branches, resulting in grafting. The rate of copolymerization declines. Since benzoyl peroxide is in the form of small crystal particles, the amount of 0.1 g when weighed is already very small, so no study has been conducted on the effect of initiating a dose of less than 0.1 g.

3.5. Effect of crosslinking agents on chemical modification of materials

In this group of experiments, the reaction temperature was set to 85° C, the reaction time was 3 hours, and 4 mL of the grafting monomer butyl methacrylate and 0.2 g of the initiator benzoyl peroxide were added. Then, under the conditions of the amount of crosslinker methylenebisacrylamide dosing 0.01 g, 0.02 g, 0.03 g, 0.04 g, 0.05 g, the graft copolymerization reaction was carried out and measured sequentially under different conditions. Oil absorption in pure diesel.
As can be seen from Figure 5, when the crosslinking agent methylenebisacrylamide dosing amount is 0.01 g, the modified material has an oil absorption of 11.93 g/g. With the increase of the amount of crosslinker, the oil absorption rate of the modified material BMC gradually decreased.

4. Experimental results and discussion
Chemical modification of the bagasse cellulosic material was carried out with butyl methacrylate to obtain a highly effective composite oil absorption material BMC. The optimal reaction conditions for the preparation of BMC high-performance composites were as follows: reaction temperature 85 °C, reaction time 4 h, raw material bagasse 3.00 g, graft monomer butyl methacrylate 2 ml, initiator benzoyl peroxide. It is 0.1 g and the crosslinking agent methylenebisacrylamide is 0.01 g.

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