Preparation and Identification of Carbon Materials from Coffee Grounds

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Abstract. Carbon materials have been reported to applying extensively on many biological researches. In this paper, the organic carbon component in coffee grounds was extracted to convert the activated carbon, and the adsorption activity of the activated carbon was studied. It was noticed that the two steps of oil extraction and sieving gave a great influence for adsorption activity. Oil extraction in the treatment process made the surface of the coffee grounds form a loose and porous structure and thus the adsorption areas were expanded, which was the important influencing factor. Screening could control the particle diameter of the final product. It was found that the large particles of the unscreened sample formed activated carbon of coffee grounds with adsorption properties. It showed the stronger adsorption activity of the activated carbon than the original coffee grounds, which showed only weak adsorption. The final product obtained after oil extraction and sieving was about 70% higher than the adsorption capacity of commercial activated carbon.

1. Introduction
The improvement of the quality of life has led to an increasing demand for coffee from the Chinese people. While meeting the demand for drinking, it also produces a large amount of coffee grounds. The warm, humid and organic environment can easily allow bacteria to multiply without cleaned in time. Activated carbon has excellent adsorption and is widely used to improve water quality, purify air, prevent and control environmental pollution. However, the price of raw materials is high, and it is necessary to find a low-cost alternative that also has adsorption performance as soon as possible. Therefore, we decided to take coffee grounds as the research object, extract the biochar and make it into activated carbon, explore its adsorption strength.

2. Scheme Design and Implementation

2.1 Adsorption Standard
Adsorption capacity and adsorption rate are the two main parameters for measuring adsorption performance. Methylene blue or neutral red solutions are commonly used as reference reagents. In order to exclude the influence of the pH of the solution in this test, the methylene blue solution was used as the standard to measure only from the aspect of the adsorption rate.
2.2. Experimental Variable

2.2.1 Whether or not sieved
To ensure an extremely high specific surface area, the particle diameter must be minimized. However, during the experiment, it was found that the carbonized and ground products had no difference from the naked eye. Therefore, when the experiment was repeated, whether or not to pass through a 50 mesh sieve was added as the first variable.

2.2.2 Whether to raise oil
The ingredients in coffee grounds are extremely complex. Except for caffeine, polysaccharides, water, etc. more of them are lipids (34.79% of linoleic acid, 9.85% of oleic acid, 42.12% of palmitic acid, and 8.49% of stearic acid). To make coffee grounds biochar, oil must be removed. In order to compare the effect of grease on the porous structure, this was set as the second variable. Using the petroleum ether reflux method, heating the water bath at 70°C for 4h to extract the oil and fat in the coffee grounds, the dosage ratio is: 50g coffee grounds: 300mL petroleum ether.

2.2.3 Experimental sample

|                    | 80 mesh sieve is not used | 80 mesh sieve is used |
|--------------------|--------------------------|----------------------|
| Not to mention oil | No.1                     | No.2                 |
| mention the oil    | No.4                     | No.3                 |

In addition to the above four experimental groups, in order to compare the activation effect, the following three groups are set up.
No. 5: Unground coffee grounds
No. 6: Ground coffee grounds
No. 7: Commercial activated carbon
(Note: The above seven groups are all weighed after drying)

2.3 Experiment Process

2.3.1 Sample preparation
According to the grouping of 1.2.3, weigh 3.0g of No. 1, 2, 3 and 4, respectively, and soak in 15mL of 40% ZnCl₂ solution for 48h (adjust the pH to 1). Activate for 200 minss in a drying cabinet at 180 °C. After activation, carbonization was carried out in a microwave reactor under a nitrogen atmosphere: carbonization at 550 °C for 12 minss, temperature increase rate of 5 °C / s, and temperature decrease rate of 4 °C / minss. After crushing, it was washed three times with 1% hydrochloric acid, then rinsed with distilled water until neutral, and finally treated with a centrifuge with a rotating speed of 7000 rpm / minss for 10 minss. The supernatant was discarded and the precipitate was dried.

2.3.2 Performance testing
The methylene blue solution with a concentration of 0.2 mg / mL and 7 bottles labeled No. 1-7 were prepared. Add 45mL of the prepared methylene blue solution and 0.02g of the corresponding activated carbon sample to each bottle. And put the bottles in the shaker to continue shaking. Measure the absorbance (665nm) of the methylene blue solution in each bottle at different times using a spectrophotometer. Observe the surface characteristics of the product using an electron microscope.
3. Results and Discussion

Table 1 Methylene blue absorbance at 665nm

| Methylene blue | the first time | the second time | the third time | average |
|----------------|---------------|----------------|---------------|---------|
| Absorbance     | 3.877         | 3.872          | 3.867         | 3.872   |

Table 2 Absorbance of methylene blue after different time

| number | 1   | 2   | 3   | 4   | 5   | 6   | 7   |
|--------|-----|-----|-----|-----|-----|-----|-----|
| 1h     | 3.836 | 3.814 | 3.758 | 3.825 | 3.852 | 3.837 | 3.813 |
| 2h     | 3.690 | 3.684 | 3.622 | 3.661 | 3.713 | 3.687 | 3.721 |
| 3h     | 3.650 | 3.636 | 3.571 | 3.614 | 3.693 | 3.664 | 3.694 |

Note: Original methylene blue absorbance: 3.872

According to the absorbance curve, it can be known that the four products have faster adsorption rates than the commercially available activated carbon, so samples 1-4 were selected to be placed under an electron microscope to observe the pore diameter, and as a control, untreated raw coffee grounds were added. The electron micrograph is as follows:

![Figure 1](image1.png)

**Figure 1** electron micrograph of No.1(from 1.1 to 1.4)

Observation results: The sample size of this group of samples is very uneven, and the particle diameter ranges from 2 μm to 100 μm. Most of them show irregular multi-faceted blocks (see Figures 1.1 and 1.2). In addition, there are smooth, crystal-like particles: spherical (Figure 1.3) and polyhedron (Figure 1.4). The diameter of such particles is between 2-5 μm. Presumed to be the product of oil and fat in coffee grounds at high temperatures.

![Figure 2](image2.png)

**Figure 2** electron micrograph of No.2(from 2.1 to 2.4)
Observation results: The particles in this group of samples are relatively uniform in size, most of which are 80 μm to 100 μm in diameter. Most of the particles are triangular prism-shaped and have a smooth surface (Figure 2.2). One of the most complete particles was zoomed in and found that there were attachments on the surface (Figure 2.3). After zooming in again, it was found that an ideal pore size had been formed. The number of pores was small and the diameter was small, about 2 μm, spreading across the particle surface (Figure 2.4).

![Figure 3 electron micrograph of No.3(from 3.1 to 3.4)](image1)

Observation results: It was found under the electron microscope that although the samples of this group were not uniform in size (Figures 3.1 and 3.2), after careful observation it was found that both large and small particles had a large number of small holes on the surface (Figures 3.3 and 3.4) Is a loose and porous irregular block. It can be seen that the small particles are the products obtained after the large particles are broken, and they still have adsorption properties. In summary, this group is consistent with the characterization of the expected product of the experiment.

![Figure 4 electron micrograph of No.4(from 4.1 to 4.4)](image2)

Observation results: The activated carbons obtained in group 4 are all irregular blocks and still have a loose and porous structure, but only for larger particles with a diameter of 80 μm or more (Figure 4.3). The enlarged pore size is similar to that of group 3 (Figure 4.4). No pores appear on the surface of the small particles.

![Figure 5 electron micrograph of No.5(from 5.1 to 5.4)](image3)
Observation results: The original coffee grounds sample without any treatment has a rich morphology under the electron microscope, which is flaky (Figure 5.2), lumpy (Figure 5.3), or cotton wool (Figure 5.4). It is completely different from the first four samples.

4. Conclusions

It is completely feasible to prepare activated carbon from coffee grounds. Comparing the absorbance, the following conclusions can be drawn:

(1) Even if the coffee grounds are not activated or carbonized, they still have a certain degree of adsorption, but they are very weak, and they rise to a certain extent after oil extraction. Coffee grounds are cheaper than activated carbon, and have strong practicability in some special scenarios: for example, you can place coffee grounds indoors for air purification after new house decoration;

(2) For the 7 groups of samples tested in this experiment, the adsorption rates are ranked as follows: sieved oil > sieved oil > sieved oil > sieved oil > coffee grounds > activated carbon > Ground coffee.

The two variables designed in this experiment were tested for importance: oil extraction > sieving, except for the untreated raw coffee grounds samples, the other five groups had faster adsorption rates than commercially available activated carbon, of which oil extraction The sieve-treated group No. 3 has the best performance, with about 70% higher adsorption capacity than commercially available activated carbon in the same time.

Comparing the SEM photos of 1, 2, 3, 4, 5, oil extraction can make the surface of the coffee grounds form a loose and porous structure so as to have adsorption, which is the first influencing factor. The sieve can control the particle diameter of the final product. Only the large particles of unscreened samples can form coffee ground activated carbon with adsorption properties.

(3) Activated carbon is widely used in environmental pollution prevention and other fields due to its excellent adsorption performance, but the taste, price and supply of activated carbon raw materials restrict its mass production. At present, studies have shown that waste biomass can be activated and activated carbon can be prepared with superior properties. This experiment uses cheap coffee grounds as the raw material for processing. The final product has better adsorption and faster adsorption rate than activated carbon, and can be used as a substitute for activated carbon. Value-added applications have good environmental, economic and social benefits.

A variety of fast food restaurants or drink shops in the society can completely guarantee the supply of coffee grounds. If the technology is mature, it can not only reduce environmental pollution, but also use the extracted coffee charcoal to generate a new economy.

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6. References

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