Free-Standing Activated Carbon Made from Melamine- Impregnated Paper

Yaokun Zhang
South China Agricultural University
Xiuyi Lin
South China Agricultural University
Chuanshuang Hu
South China Agricultural University
Hong Yun (hongy@scau.edu.cn)
South China Agricultural University

Article

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Abstract

Aiming at the high value-added utilization of waste scraps of Melamine impregnated paper, also known as “melamine” paper, is a plain base paper or printing decorative paper impregnated with amino resin (melamine formaldehyde resin and urea formaldehyde resin) and dried to a certain extent, with a certain resin content and volatile content of the adhesive paper, after hot pressing can be bonded with each other or with artificial board substrate., this study adopts high temperature pyrolysis method to carbonize the waste impregnated paper to prepare melamine impregnated paper-based activated carbon. The effects of different carbonization temperatures and NaOH/C on the properties of activated carbon materials were studied. When the carbonization temperature is 400℃ and the ratio of alkali to carbon is 2:1, the highest removal rate of methylene blue (MB) by activated carbon is 99.45%, and the specific surface area reaches 608.548 m^2/g. Langmuir isotherm, Freundlich isotherm models were used to fit the experimental dye adsorption data, with the Langmuir providing the best fit. This activated carbon adsorbent based on melamine impregnated paper is shown to be highly promising for water decontamination applications.

Introduction

Melamine impregnated paper (MIP) is a kind of facing material in which paper is used as the base material and the surface paper is impregnated with melamine formaldehyde resin. It is currently widely used in the surface decoration of wood-based panels. The world produces about 9 million square meters of melamine impregnated paper every year, and the cutting volume reaches 380,000 cubic meters per year. Since melamine is a thermosetting resin, it is difficult to recycle the waste MIP and can only be treated as industrial waste, which not only increases production costs, but also pollutes the surrounding environment.

In this study, Melamine impregnated paper (MIP) as a waste material has received more attention, because these are accessible in huge amounts in all areas of the world.

At present, some companies pulverize melamine impregnated paper and use it as a filler for wood-based panels[1]. Some researchers used artificial board hot pressing process to make melamine impregnated paper powder into composite particleboard[1]. However, this method does not significantly improve the performance of the board, and the added value of the product is low. Therefore, the development of high value-added waste melamine impregnated paper utilization technology is of great significance.

Environmental issues such as water and air pollution have become a great concern to the scientific community in recent years. Among them, water pollution has drawn more attention to the world because of its devastating and catastrophic effects[2]. The direct discharge of industrial waste (from textile, paper, dye synthesis, printing, leather, electroplating and cosmetics industries) containing different types of pollutants like heavy metals, crude oils, pigments, synthetic dyes and organic solvents cause severe water pollution[3–5].

Activated carbon is made of solid carbonaceous material through high temperature carbonization and activation under the condition of isolating air. It has a developed pore structure, a large specific surface area[6] and abundant surface chemical groups, so it has a specific adsorption capacity. Researchers have used agricultural waste and by-products[7] or industrial waste and by-products to produce activated carbon with excellent performance. Camellia husk, sucrose, etc. can be made into activated carbon with excellent performance under the activation of alkali[8,9]. Factors such as pyrolysis temperature and activator type will also affect the performance of activated carbon materials. The research results of Wang et al. showed that the adsorption performance of methylene blue reached the maximum when the pyrolysis temperature reached 800℃. And the adsorption performance of activated carbon is controlled by the charge, chemical structure and size of organic pollutants [10]. In another study, Jawad treated coconut shells with sulfuric acid to prepare activated carbon[11]. The results of the study showed that the amount of adsorbent was 0.1g/100mL, the pH value of the solution was 8, and the adsorption effect was best when the temperature was 303k. Sun et.al., studied a work on lignin-based biological activated carbon was modified with iron ions, and the removal rate of 100 mg/L methylene blue (MB) by the adsorbent reached 100% within 15 minutes, which is not suitable for industrial intention because more energy[12].

Min Wang et al. used the mesoporous zeolite-activated carbon composite material to remove the ammonia nitrogen and methylene blue in the aqueous solution. The results showed that the composite activated carbon material has adsorption capacities of 754.75 mg/g and 9 mg/g (298K) for MB and NH_3-N[13]. Yadav et al., synthesized activated carbon from eucalyptus with carbon nanotubes to make a new type of activated carbon adsorbent[14]. Studies have shown that when the dye concentration is 8 ppm, the composite activated carbon is less effective for methylene blue (MB) and eosin yellow (EY). The best amount of adsorbent is 1.5g/L and 0.5g/L. Consequently, there is a strong global urge for designing and development of advanced activated carbon materials that have high dye removal efficiencies.

The ratio of base paper to dipping amount in Melamine impregnated paper is 1:1.1. The paper fiber has a disordered porous structure, which provides a porous precursor material for activated carbon. However, it is easy to be destroyed during the carbonization process, and the carbon structure formed by the melamine resin at high temperature provides a hard skeleton, which makes up for the shortcomings of low physical strength of paper fiber carbon materials. In this study, waste melamine impregnated paper was used as raw material, and activated carbon materials were prepared by NaOH chemical activation method, and the effect of carbonization temperature and activation method on the performance of activated carbon was studied. Use it to produce an activated carbon material with superior adsorption performance, realize the high value-added utilization of waste materials, and achieve the purpose of protecting the environment.

The sorption behavior of dyes on an activated carbon composite surface has been examined utilizing batch adsorption method. The physicochemical characteristics of the adsorbent are investigated using TG, SEM, and BET. The adsorption tests are performed and the impact of working conditions like NaOH-to-C ratio, dye concentration, carbonization temperature[15], and adsorbent-adsorbate contact timing are

explored on the adsorption proficiency of adsorbent. A specific goal of the examination is to estimate the kinetic of adsorption of MB on adsorbent. The data is fitted to Isotherm study and Kinetic study to decide the best-fit equation.[16–20]

Experiments

1 Material

The reagents used in the experiment are of analytical grade that are purchased from McLean (China). Melamine impregnated paper (Guangdong Yaodonghua Decorative Material Technology Co., Ltd). The stock solutions of the dyes, methylene blue (MB) having chemical formula $C_{16}H_{18}C_lN_3S$ (MW: 319.87) were prepared by dissolution of 25 mg in 500 ml distilled water (DW). And were prepared by consecutive dilution for achieving the required concentrations (2-50 mg/L) of the dye for the adsorption experiments. The solution was prepared by dissolving the required amount of dye in distilled water.

2 Materials synthesis

2.1 Preparation of activated carbon

As experimental sections, a powder of 40-60 mesh was prepared from melamine impregnated paper (MIP). The powder is placed in an oven at 105 °C and dried for 2 hours to remove moisture from the material. The material is placed in a tube furnace for carbonization, conjunction with selected carbonization temperature is 300°C, 350°C, 400°C, and 450°C. Then four carbonized materials at different temperatures were activated by NaOH. The NaOH-to-C ratio was 2:1,3:1,4:1,5:1, mixed with carbonized samples and add appropriate amount of deionized water, in magnetic mixer for two hours, then placed in 105 °C ovens to remove moisture, finally placed in the tube furnace, at 600 °C activation treatment. After activation, the sample is washed to neutral by deionized water and hydrochloric acid. The whole process is shown in Fig.1.

Schematic diagram showing the carbonization process of MIP

2.2 Adsorbability of activated carbon

The Methylene blue reagents were used to prepare the standard solution of different solubility of Methylene blue at the concentrations of 100 mg/L, 200 mg/L, 300 mg/L, 400 mg/L, and 500 mg/L respectively. 30 mg of 80-100 mesh activated carbon and 50 ml of 100 mg/L Methylene blue solution were placed in a 250 ml conical bottle and then placed in a constant temperature oscillator at 30 °C and 150 rpm until adsorption equilibrium. The adsorption amount is calculated by the formula (1) by measuring the MB absorbance before and after the adsorption through the ultraviolet photometer.

2.3 Performance test

TG and DTG analysis

Take 5-15 mg of the sample into an alumina crucible (ø6.8×4.0mm, Shanghai Jingyi Chemical Material Co., Ltd.), and place it in a thermogravimetric analyzer (STA449C, NETZSCH-Gerätebau GmbH, Germany) for pyrolysis performance testing. The nitrogen flow rate is 30 mL/min, the heating rate is 10 °C/min, and the target temperature is 800 °C.

Results And Discussion

3.1 Analysis of adsorbability results

The prepared 100 mg/L Methylene blue was diluted to 2 mg/L, 4 mg/L, 6 mg/L, 8 mg/L, 10 mg/L, and 12 mg/L. After decantation and filtration, the equilibrium concentrations of dye in the solution were measured at 665 nm using UV-visible spectrophotometer, $y = 0.07727x + 0.05234$. According to the equation, the adsorption capacity of activated carbon at different Methylene blue concentration was calculated. The fitted equation is shown in Fig. 2, R^2= 0.9941.

The adsorption capacity and removal rate of activated carbon for methylene blue are carried out according to formulas (2) and (3), thus measured data is shown in the Table.1.

$$q_e = \frac{\left(C_0 - C_e\right)V}{W}$$

2

$$R = \frac{C_0 - C_e}{C_0} \times 100\%$$

3

Table.1 The influence of carbonization temperature and NaOH-to-C ratio on the adsorption capacity and removal rate of activated carbon

Temperature	NaOH/C	q_e (mg/g)	R(%)
300℃	2: 1	482.23	96.45
	3: 1	395.22	88.64
	4: 1	468.78	93.76
	5: 1	457.95	92.59
350℃	2: 1	492.92	98.58
	3: 1	403.82	94
	4: 1	475.5	97.76
	5: 1	470	95.1
400℃	2: 1	497.23	99.45
	3: 1	406.62	93.14
	4: 1	495.92	99.32
	5: 1	465.7	99.18
450℃	2: 1	496.43	99.29
	3: 1	405.65	91.13
	4: 1	489.91	97.98
	5: 1	486.28	97.26
*$[MB]_{initial}$=300mg/L			

3.1.1 The effect of carbonization temperature on adsorption

According to Fig. 3, the amount of methylene blue adsorbed by activated carbon increases with the increase of carbonization temperature, reaches a peak when the temperature reaches 400°C, and then begins to decrease.

The main function of the carbonization process of MIP is to enrich the fixed carbon in the raw materials, reduce the volatile components and moisture in the raw materials, thereby improve the strength of the carbonized materials, and at the same time generate initial porosity, which helps the activation process. If the carbonization temperature is too low, the components will not be completely volatilized, so that sufficient initial porosity will not be generated, which will affect the adsorption. If the temperature is too high, the graphite crystallites in the carbonized product will change in an orderly manner, reduce the gaps between the crystallites, and affect the activation process.

3.1.2 The effect of NaOH-to-C ratio on adsorption

According to Fig. 4.The ratio of NaOH-to-C ratio has a significant effect on the adsorption capacity of activated carbon. When the ratio of NaOH-to-C ratio is 2:1, the adsorption capacity reaches 497.23 mg/g.

With different NaOH-to-C ratio, the properties of the prepared materials are different, which is mainly reflected in the difference in specific surface area.

The chemical activation method is used to prepare more pore structures. With the increase in the proportion of the activating reagent NaOH, the precursor of the material is brittle and cannot withstand severe chemical changes, resulting in larger pores or through-holes. Because of surface area is reduced, so that the adsorption capacity of activated carbon is not well.

3.2 TG results and analysis

According to Fig. 5. From room temperature to about 250 ℃ is the initial stage of pyrolysis temperature rise. MIP raw material sample absorbs heat and evaporates water, thereby reducing the weight. The temperature is in the range of 250 ~ 400 ℃, which is the pyrolysis stage of MIP, mainly hemicellulose and cellulose pyrolysis, so the thermal weight loss is the most, and the weight loss rate is the fastest. When temperature rises to 400 ℃, the main ingredients of the melamine facing paper enter the end of pyrolysis, the structure gradually stabilizes and the formation of a carbon layer, and finally, the carbon residue rate of MIP is 25.06%, which is similar to that of ordinary agricultural waste.

3.3 SEM – morphological studies

A SEM study was explored to reveal microstructural morphological features of activated carbon. Figure 6a-d as shown in prepared carbonization/pyrolysis processes revealed morphological features of activated carbon. Small and large particles microstructures view with different magnifications explored at 20 – 10μm. The major part of the samples NaOH is chemically treated and thermally activated like pyrolysis/carbonization effects of 3D–porous carbon. The carbonization temperature is 400℃, when the ratio of NaOH-to-C ratio is 2:1, the surface of activated carbon is relatively dense, and the pore structure is rich, and the pore size ranges from 0.1nm to 4nm; when NaOH-to-C ratio to 3:1, there are fewer micropores on the surface of activated carbon. when the ratio of NaOH-to-C ratio is 4:1 and 5:1, the pore structure of activated carbon is not uniform, and the minimum pore size is about 0.2 nm. Clearly, shown that Fig. 6a lower magnification micro-structural images of 3D-porous fully inter-connected network combine of Cellular Structure forming an interconnected porous network.

3.4 BET analysis

To investigate the textural features (surface area and pore size distribution) of the adsorbent, the nitrogen adsorption-desorption isotherm was determined by using surface area analyzer. BET(Fig. 7) survey curve showed major contribution of activated carbon which is clearly observed about N_2 adsorption-desorption isotherm and pore size distribution. The curve is mainly s-shaped, which is a type II isotherm. There is an inflection point in the low-pressure zone, indicating that the single-layer adsorption is saturated at this

time, and the way to continue the adsorption is multi-layer adsorption. As seen in Fig. 6b when the NaOH-to-C ratio is 3:1 that the adsorption capacity is the lowest.

As shown in Table.2, shows the pore structure changes in the activated carbon caused by temperature variation during the preparation process. The maximum specific surface area of activated carbon is 608.55 m^2/g, the average pore diameter is mainly between 1.93 nm to 3.04 nm, the micropore volume is small, and the pore diameter distribution is mainly concentrated between micropores and mesopores, and minimum pore volume is 0.131 cm^3/g.

Table.2 Specific surface area and pore structure parameters of activated carbon under different conditions

Sample	BET Surface Area(m^2/g)	Total pore volume (cm^3/g)	Micropore volume(cm^3/g)	Average pore size(nm)
400°C, 2: 1	608.548	4.4309	0.221061	1.9295
400°C, 3: 1	364.329	8.4167	0.13134	2.345
400°C, 4: 1	602.828	3.7402	0.205702	2.3277
400°C, 5: 1	442.619	4.9573	0.144457	3.0432

Therefore, based on the understanding and control strategy of activated carbon pore structure formation process, creating more micropores is conducive to the improvement of MB adsorption performance.

3.2 Isotherm study

Interpreting the isotherm information is significant for originating an equation that can be utilized. The Langmuir (4) and Freundlich (5) models were used to analyze the adsorption thermodynamics of MIP activated carbon. (see Fig. 8)

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e}$$

4

$$q_e = K_f C_e^{\,1/n} \quad (5)$$

In the formula, C_e(mg/L) is the concentration of adsorbate in the solution at equilibrium, q_e(mg/g) is the adsorption capacity per unit mass of adsorbent at equilibrium, q_m(mg/g) is the maximum single-layer adsorption per unit mass of adsorbent. K_L(L/mg) is the Langmuir isotherm adsorption energy constant.

When R_L>1.0, it means that it is not a single-layer adsorption; when R_L=1.0, it means that the relationship is linear; when R_L<1.0, it means that it is a single-layer adsorption; when R_L=0, it means that it is irreversible adsorption. K_f is the Freundlich isotherm adsorption empirical constant, which is related to the adsorption capacity and the adsorption strength.

The correlation coefficients obtained by the nonlinear fitting of Langmuir and Freundlich isotherm models are 0.96233 and 0.94119, respectively, indicating that the adsorption of methylene blue by activated carbon is more consistent with the Langmuir model, and the adsorbent surface is uniform and is a single-layer adsorption. Calculated parameters of Isotherms for MB removal are given in Table.3.

Table.3 Langmuir and Freundlich equation parameters

Isotherm	Langmuir			Freundlich		
parameters	$q_m(mg/g)$	$K_L(dm^3/mg)$	R^2	$K_f(mg/g(L/mg)^{1/n})$	n	R^2
	269.45	0.1143	0.96233	275.49	7.9246	0.94119

3.3 Kinetic study

In order to further explore the adsorption state of activated carbon in the Kinetic models, the typical pore size models were combined.

The adsorption kinetics of methylene blue on activated carbon was investigated under the conditions of 30 ℃, not pH value adjustment, and 0.03 g of activated carbon. The nonlinear fitting of the quasi-first-order kinetics model (6) and the quasi-second-order kinetics model (7) was performed respectively. (Fig. 9)

$$q_t = q_e\left(1 - exp^{-k_1 t}\right)$$

6

$$q_t = \frac{{q_e}^2 k_2 t}{1 + q_e k_2 t}$$

7

In the formula, t (h or min) is the adsorption time, q_t, q_e (mg/g) are the adsorption capacity at time t and when equilibrium is reached, k_1 (h^{-1} or min^{-1}) is the quasi-first order kinetic rate constant, k_2(mg·g^{-}

^{1}·min^{-1}) is the quasi-second-order kinetic rate constant.

Table.4, the correlation coefficient of the experimental data obtained by the non-linear fitting of the quasi-second-order kinetic model is 0.9961, and the correlation coefficient of the quasi-first-order is 0.70495. To a certain extent, the adsorption amount $q_{e,cal}$ value calculated by the quasi-second-order kinetic equation is relatively close to the experimental data. It showed that the adsorption kinetics of methylene blue on melamine facing paper-based activated carbon conforms to the quasi-second-order kinetic model, and chemical adsorption is dominant.

Table.4 Equation parameters

C_0	$q_{e,exp}$	Pesudo-first-order kinetic			Pesudo-second-order kinetic		
(mg/g)	(mg/g)	$q_{e,cal}(mg/g)$	$k_1(h^{-1})$	R^2	$q_{e,cal}(mg/g)$	k_2(g/mgh)	R^2
200	198.7	196.58	0.5019	0.70495	201.2	0.0065	0.9961

Conclusion

Activated carbon is prepared with melamine impregnated paper scraps as raw materials, which has good adsorption of methylene blue. When the carbonization temperature is 400℃ and the ratio of alkali to carbon is 2:1, the average pore diameter of activated carbon is mainly between 1.93nm and 3.04nm, and the specific surface area is 608.548 m²/g, which has the best performance. The adsorption capacity of methylene blue reaches 497.23 mg/g, decolorization rate reaches 99.45%. The Langmuir isotherm fitted well with the equilibrium data, which confirms that the sorption occurs via single-layer adsorption interaction. The adsorption kinetics shows the process follows the pseudo second order model. This new adsorbent is very useful for efficiently removal of dyes from waste water.

Declarations

CRediT authorship contribution statement

Yaokun Zhang: Methodology, Investigation, Formal analysis, Visualization, Writing - original draft. **Xiuyi Lin**: Validation, Formal analysis. **Chuanshuang Hu**: Project administration. **Hong Yun**: Conceptualization, Validation, Supervision, Writing -review & editing.

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Declaration of competing interest

This work did not have competing financial interests or personal relationships.

Data availability

The datasets generated during or analysed during the current study are available from the corresponding author on reasonable request.

Formatting of funding sources

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Figures

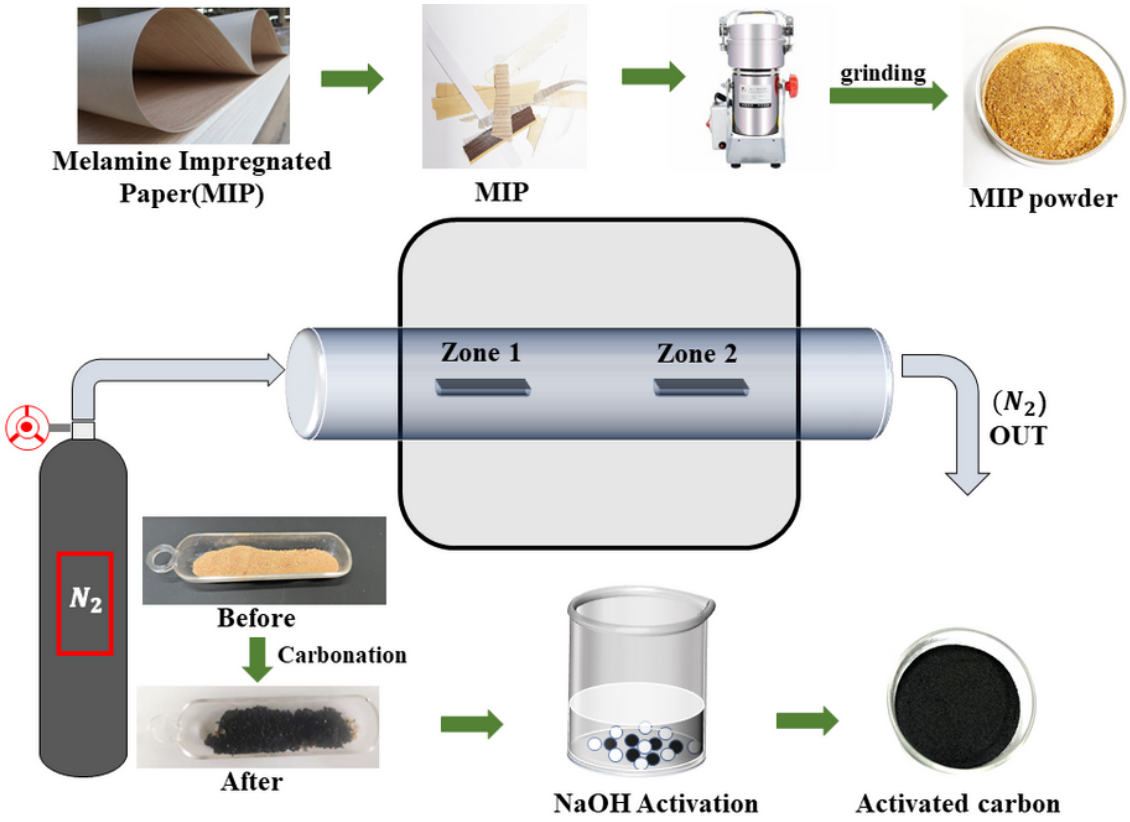

Figure 1

A schematic representation of experimental set-up and their related photographs

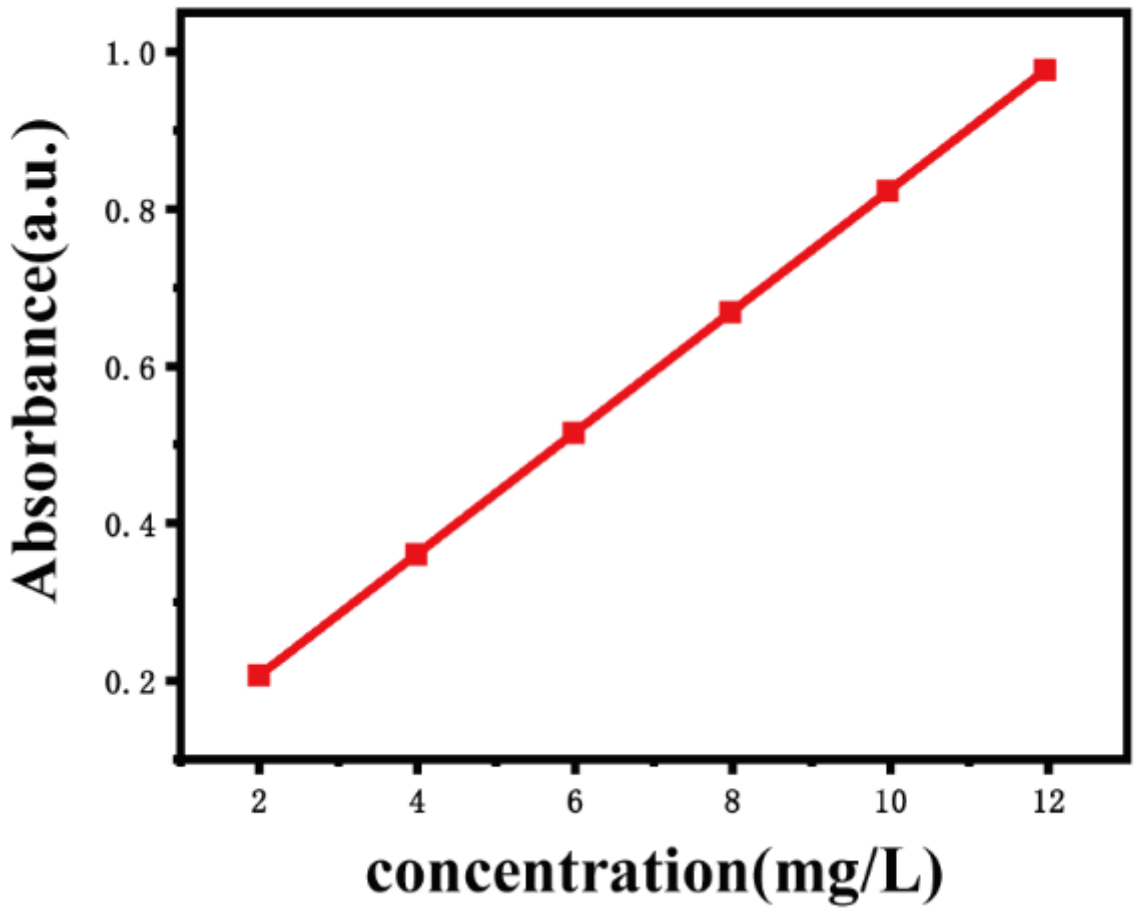

Figure 2

Standard curve of methylene blue

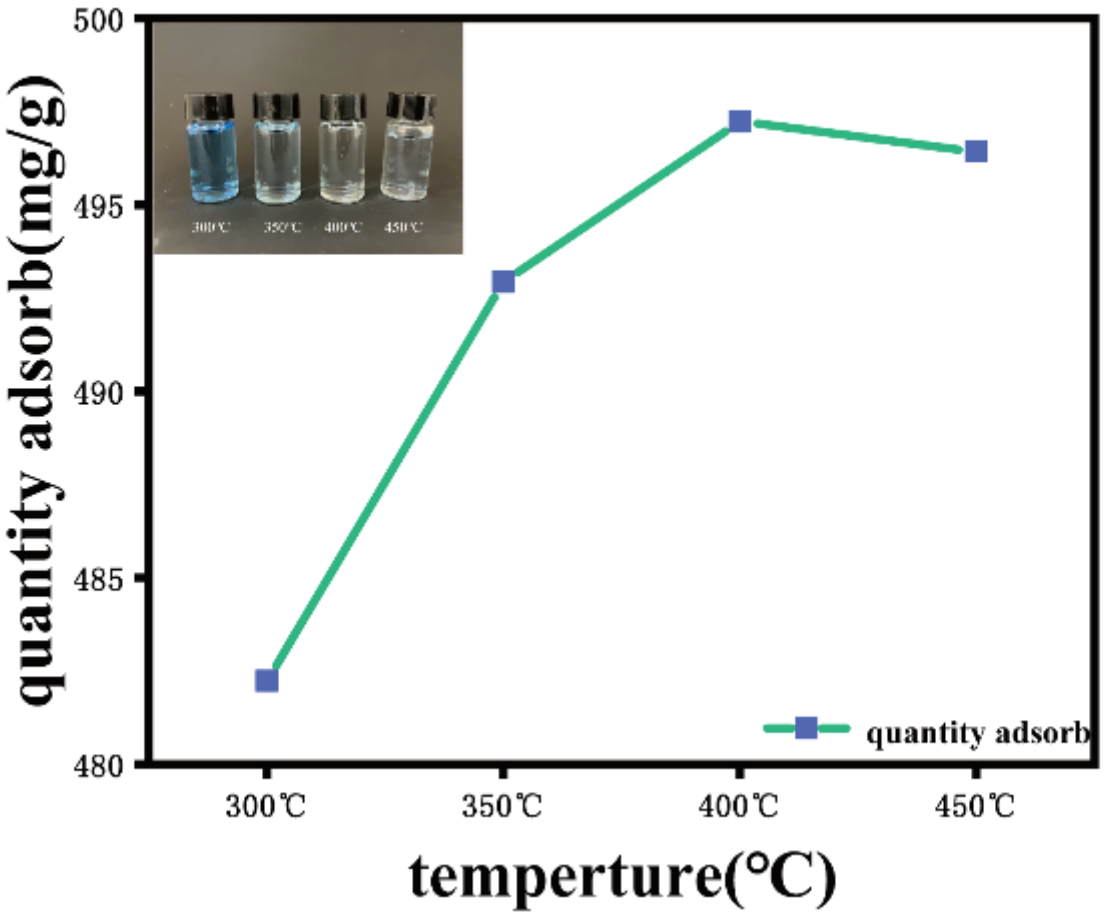

Figure 3

Effect of carbonation temperature on MB adsorption

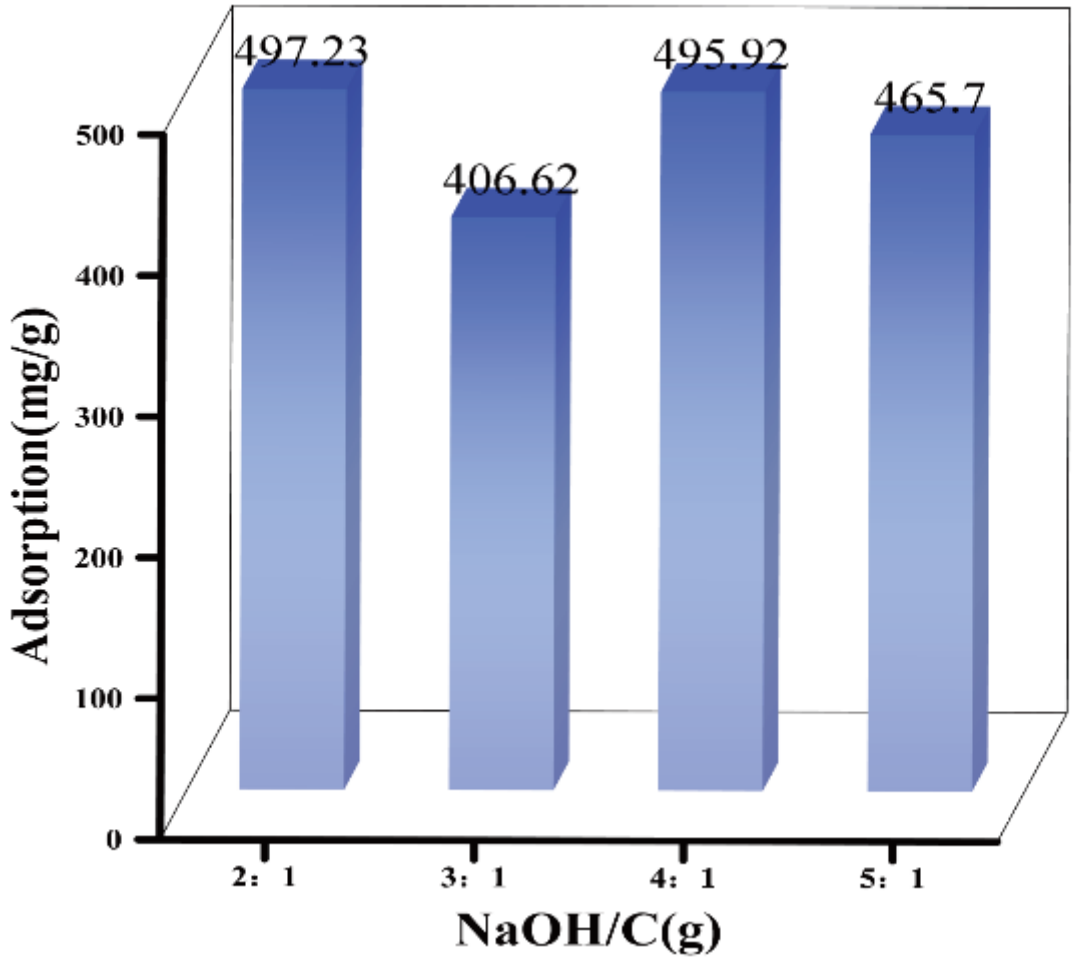

Figure 4

The effect of different NaOH-to-C ratio on the adsorption capacity of activated carbon

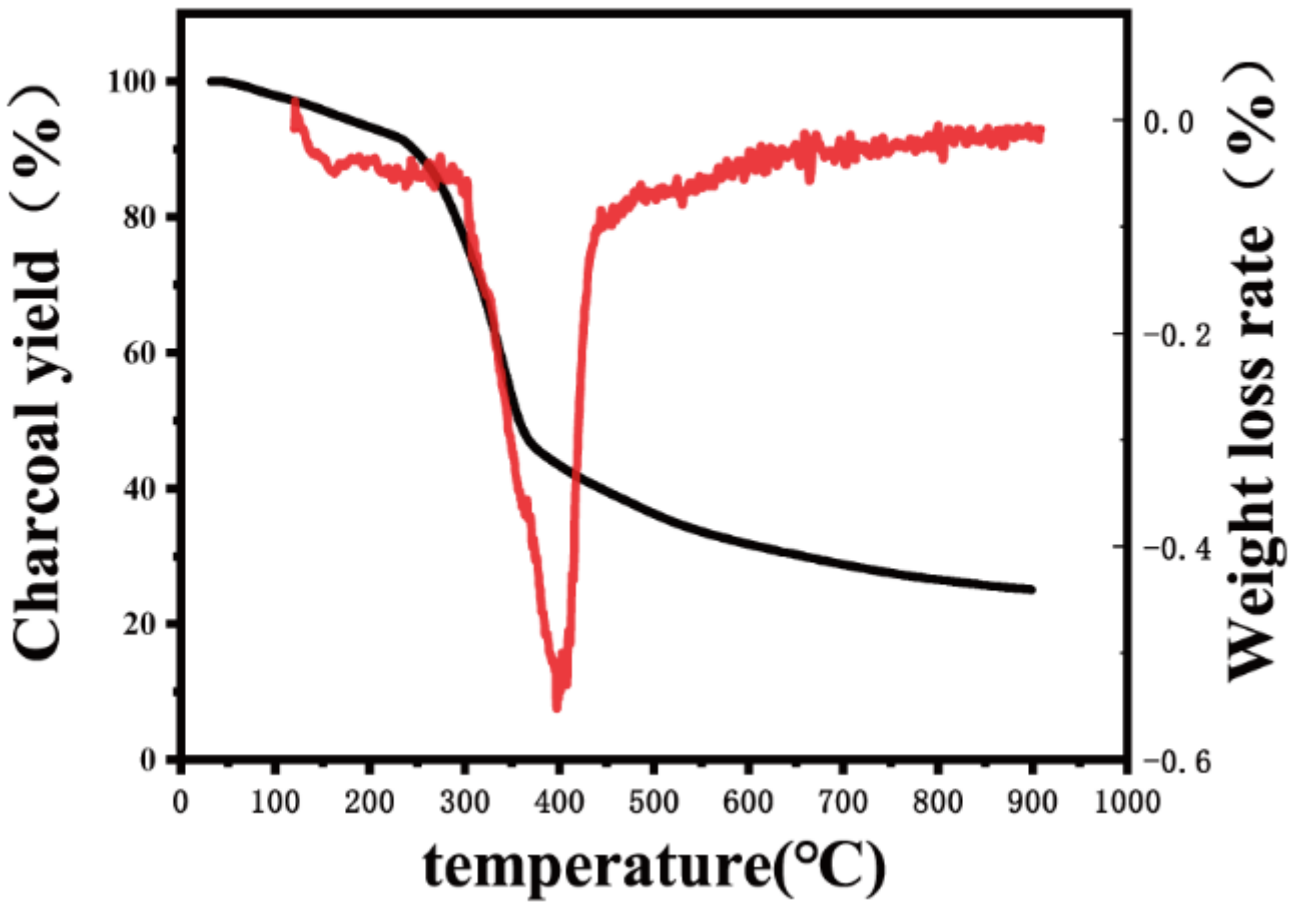

Figure 5

TG analysis of Melamine impregnated paper

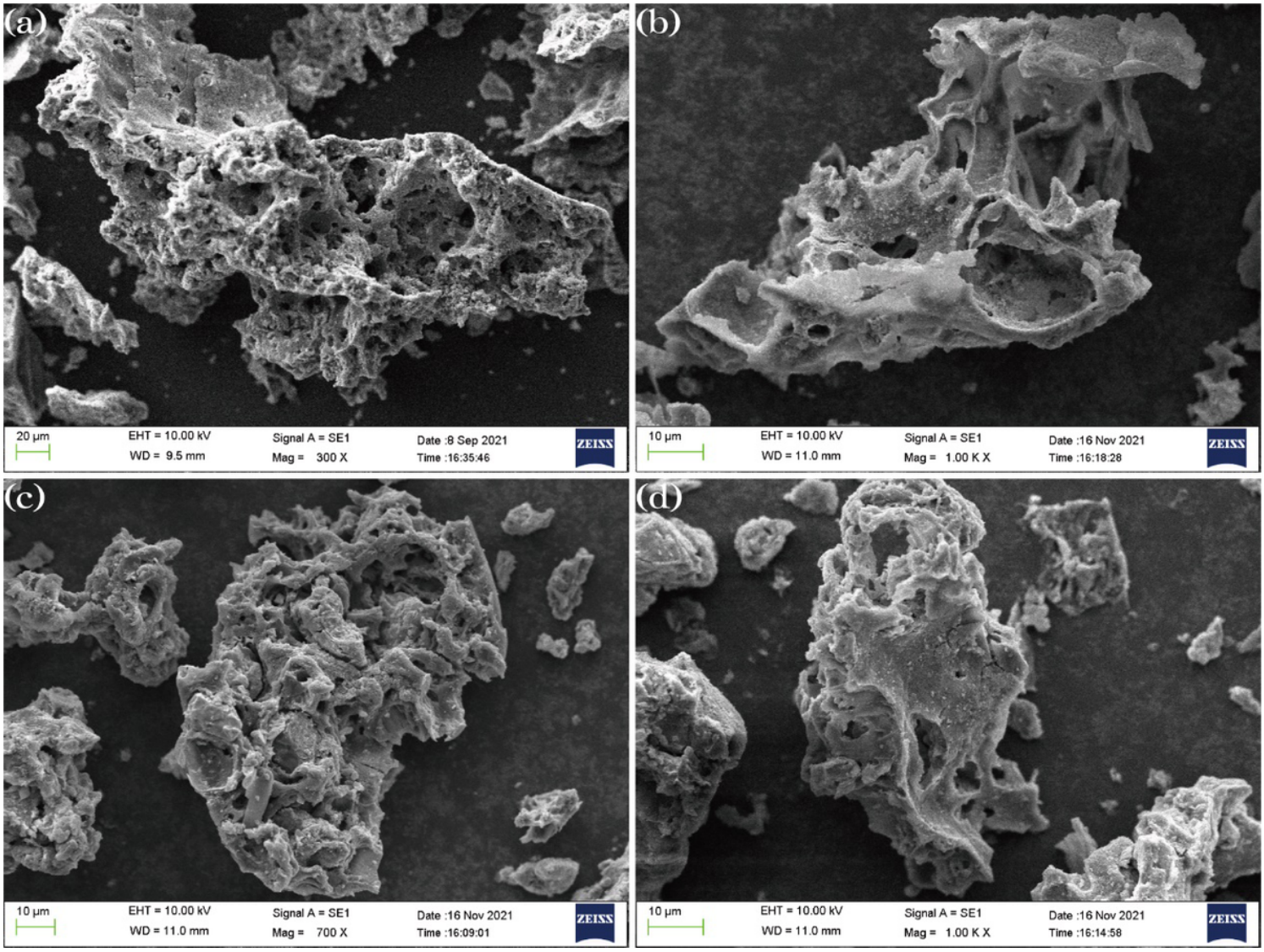

Figure 6

SEM images of activated carbon with different NaOH-to-C ratio (a: NaOH-to-C ratio is 2:1, b: NaOH-to-C ratio is 3:1, c: NaOH-to-C ratio is 4:1, d: NaOH-to-C ratio is 5:1)

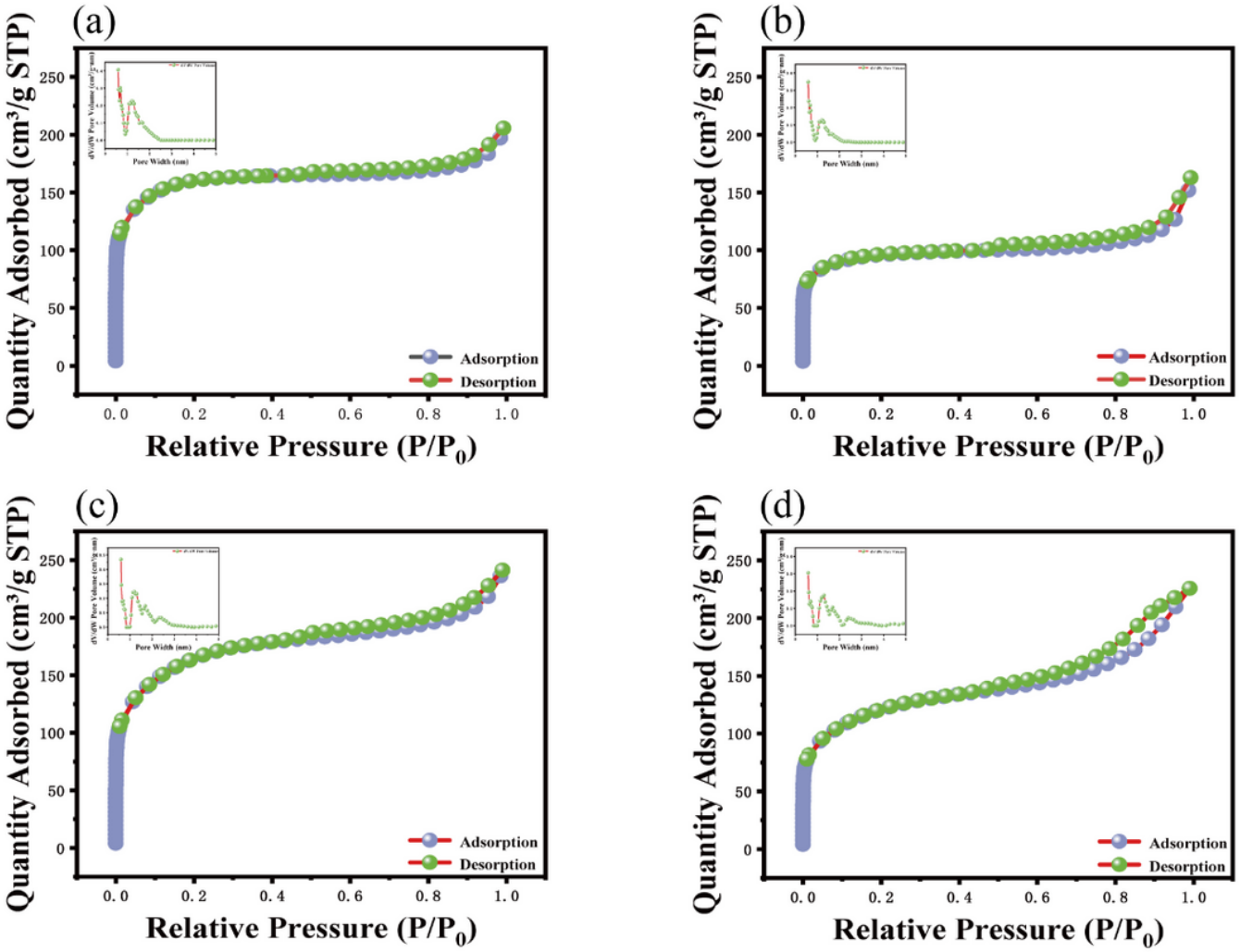

Figure 7

N_2-desorption isotherm and pore size distribution diagram (a: Carbonized at 400℃, NaOH-to-C ratio is 2:1. b: Carbonized at 400℃, NaOH-to-C ratio is 3:1. c: Carbonized at 400℃, NaOH-to-C ratio is 4:1. d: Carbonized at 400℃, NaOH-to-C ratio is 5:1)

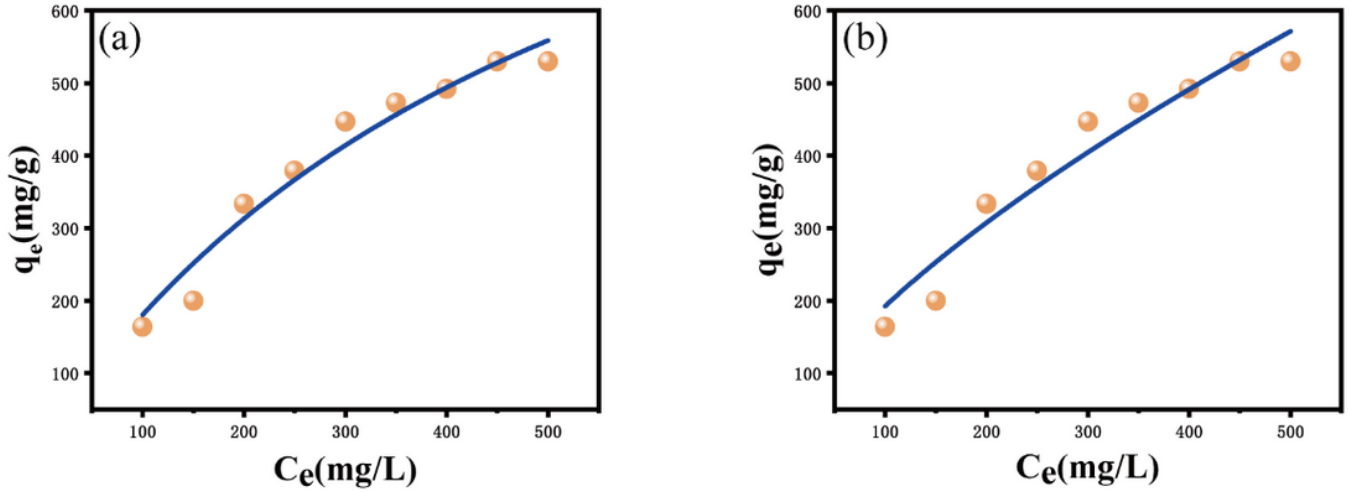

Figure 8

(a) Langmuir and (b) Freundlich

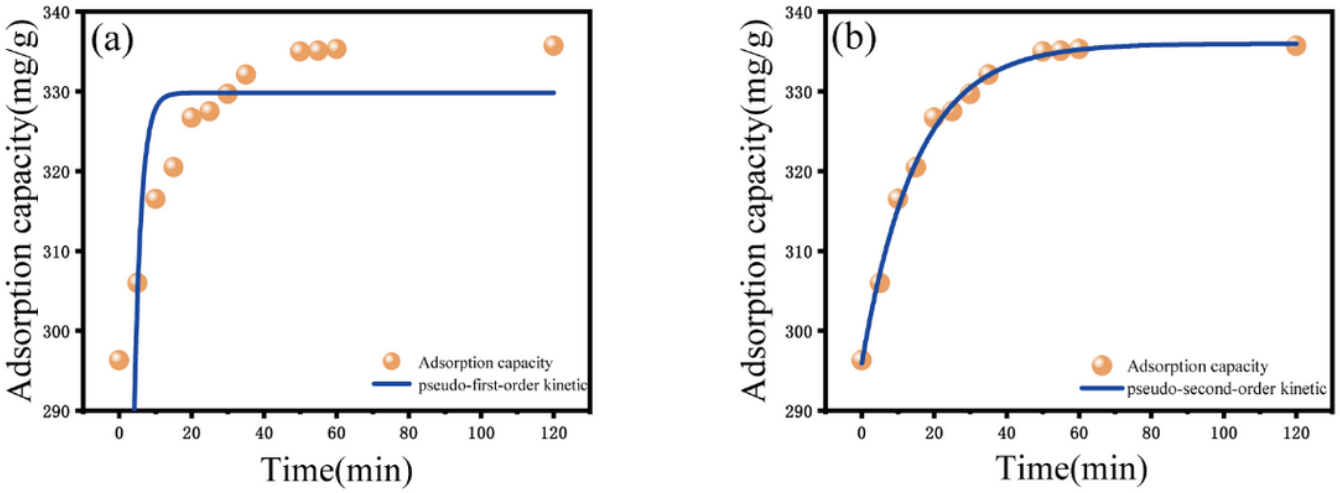

Figure 9

(a) the quasi-first order kinetic and (b) the quasi-second-order kinetic