

Optimizing the preparation of activated carbon from lugi pressurized gasifier (LPG) slag by microwave-assisted potassium hydroxide activation

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ABSTRACT

Activated carbon (AC) was prepared from the carbon residue of Lugi pressurized gasifier (LPG) slag with a microwave-assisted activation process using potassium hydroxide (KOH) as the activating agent. The effects of various factors such as the heating behavior of the microwave, power of the microwave, activation time and ratio between LPG slag and KOH on the activation have been studied. The surface property of the AC was characterized by BET. The optimal activation conditions were 900 W of microwave power, 9 min of radiation time and 1:1 ratio of KOH and carbon. The surface area of prepared AC was 1061 m².g⁻¹, and a carbon yield of 42% could be reached. The iodine value was 1082.2 mg g⁻¹. When compared with the traditional method, the microwave-assisted activation process demonstrated faster rate of activation and higher carbon yield. Microwave-assisted AC removed phenols and chromaticity from the wastewater obtained from the Coking Plant in Kunming efficiently.

Keywords: Lugi pressurized gasifier (LPG) slag; Activated carbon; Microwave-assisted; Potassium hydroxide

1. Introduction

Activated carbon (AC), which has large surface area, microporous structure and high adsorptivity, has been widely used as an excellent adsorbent for the removal of various organic and inorganic pollutants as well as a cata-

lyst support. A wide range of carbonaceous materials such as coal, peat, wood and various agricultural byproducts can be used as the raw carbon precursors [1–5].

Generally, two methods are used for the preparation of AC namely physical and chemical activation [6]. In physical activation, first the raw material is carbonized at high temperature, and then it is activated by steam or CO₂ under pressure to produce AC with well-developed and accessible internal porosities. On the other hand, in chemical activa-

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tion process, both carbonization and activation are allowed to take place simultaneously, in which raw material is first impregnated with activating reagents such as potassium hydroxide (KOH), ZnCl_2 , and H_3PO_4 and then carbonized at desired temperature which varied according to the activating agent used [7–10]. Chemical activation is held in the presence of activating reagents which promote pyrolytic decomposition and inhibit the formation of tar. The carbon yield obtained is higher, and the temperature used in chemical activation is lower than that of physical activation. KOH is widely used as activating reagent since it resulted in high surface area and high yield. The AC had large surface area and more micropore structure [11–13]. For preparing AC, conventional heating method is usually adopted, in which the energy is produced by electrical furnace. Recently, microwave heating technology has been applied to fabricate AC due to its rapid and uniform heating. The main difference between microwave devices and conventional heating systems is the way of the heat is generated. In a microwave device, microwaves supply energy directly onto the carbon bed, and thus the energy from microwaves is readily transformed into heat inside the particles by dipole rotation and ionic conduction [1,3,14–18].

Ammonia was manufactured from low-quality coal with low calorific value by Lugi pressurized gasification process in a chemical fertilizer plant in Yunnan Province. However, the process will produce Lugi pressurized gas (LPG) slag, which contains 35–40% carbon. Therefore, efficient utilization of LPG slag has important economic and social benefits. In this research, activated carbon (AC_{LPG}) was prepared from the LPG slag by microwave-assisted activation method, which is used to remove phenol. The AC_{LPG} has significant effect on decolorizing and removing the odor from the wastewater emanating from the Coking Plant in Kunming.

2. Materials and methods

2.1. Materials

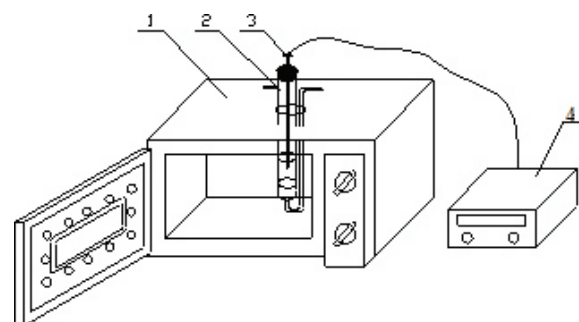
Raw material: LPG slag contains 35–40% carbon, 14.38% volatile matter; its low heating value is 10660–13850 kJ kg^{-1} . LPG slag particles which are smaller than 250 mesh were heated at 110°C and stored in desiccators. All chemical reagents except KOH used were of analytical grade. The KOH used was chemical reagent grade.

2.2. Heating behavior of LPG slag in the microwave oven

2.2.1. Experimental setup

The experimental setup was made of a microwave oven, a temperature control system and a quartz tube reactor as shown in Fig. 1.

The make and model of microwave oven used in the experiment were Guangdong Galanz Group and WD900 SL23 II, respectively. Its microwave frequency was 2450 MHz, and the export power was 900 W. There was a circular pore at the center of the microwave oven with a diameter of around 26 cm; the reactor was inserted into the microwave oven through this pore. The thermocouple in the inner part of the reactor was in contact with reactants; the thermocouple was connected to the temperature control system.



1. Microwave oven
2. Quartz tube reactor
3. Armoured thermocouple
4. Temperature indicator

Fig. 1. Experimental setup.

2.2.2. Temperature measurement

The nickel chrome-nickel silicon thermocouple (0–1100°C) was selected to measure the temperature in the reactor, and the temperature indicator is an XMZ digital temperature indicator whose precision is $\pm 1^\circ\text{C}$.

2.2.3. Microwave heat experiments

In order to understand the heating behavior of the microwave radiation on LPG slag, KOH, and the mixture of LPG slag and KOH, they were loaded into the reactor. Change in temperature was recorded at different applied power of the microwave radiation using the thermocouple system.

2.3. Preparation of AC_{LPG}

Chemical activation method with KOH as activation agent was used for the preparation of AC. LPG slag, with a particle size less than 250 mesh, was mixed with KOH by immerse method. The mass ratio of LPG to KOH mixture was 1:1. After dehydration at 120°C, activation parameters such as activation time, the LPG dosage, and the ratio of LPG to KOH were investigated.

2.4. Characterization of AC_{LPG}

2.4.1. Measurement method of iodine adsorption of AC_{LPG}

Iodine adsorption was used to study the adsorption capacity of AC. The adsorption values of various AC samples were measured according to the national standard: test methods of wooden AC—determination of iodine number (GB/T12496.8-1999) using the light photometer (721 cents, Shanghai, China).

2.4.2. Surface area of AC_{LPG}

The physical properties of the surface of AC were characterized by N_2 adsorption at -196°C (77 K) in a Micromeritics ASAP 2000 apparatus, USA). The surface area is calculated with the N_2 adsorption isotherms. The total pore volume was evaluated from the isotherms at a relative pressure of 0.98. The micropore area and volume were evaluated using the t-plot method.

3. Results and discussion

3.1. The effects of heating behavior of LPG

3.1.1. Temperature corrections to the thermocouple system

There was a lag when measuring temperature with thermocouple as the thermocouple has metal outer shell which would affect the heat transfer. In order to reduce the error (in measuring the temperature due to this effect) as much as possible, a correction test of the thermocouple measure system was carried out as follows: a mercury thermometer with a precision of 0.01°C and the thermocouple were placed in salt water; the salt water was heated gradually by an electric stove and the temperature measured both by the thermometer and the thermocouple. The recorded results are shown in Table 1.

The data shows that the discrepancy between the readings of the thermometer and the thermocouple measure system is about 4°C. Thus, the temperatures measured in the experiments were corrected accordingly. Although the thermocouple corrections were carried out in a lower temperature range, and in a different environment, they provided guide to correct the temperature readings obtained from the thermocouple; it is reasonable to expect that at higher temperatures the rate of heat transfer will be higher and thus the lag-time measuring the temperature will be lesser.

3.1.2. The heating behavior of LPG

A 10 g of LPG slag was heated in the oven at different microwave power. Rise in temperature under different power was measured by the thermocouple. The results are shown in Fig. 2.

It can be seen from Fig. 2 that there is a marked difference under different microwave power. The tempera-

Table 1
Temperature measurements obtained by a mercury thermometer and the thermocouple used in this study from a gradually heated salt solution

Reading of mercury thermometer (°C)	20.0	45.5	65.0	74.0	85.5	92.0	100.0
Reading of thermocouple (°C)	16	41	61	70	81	88	94

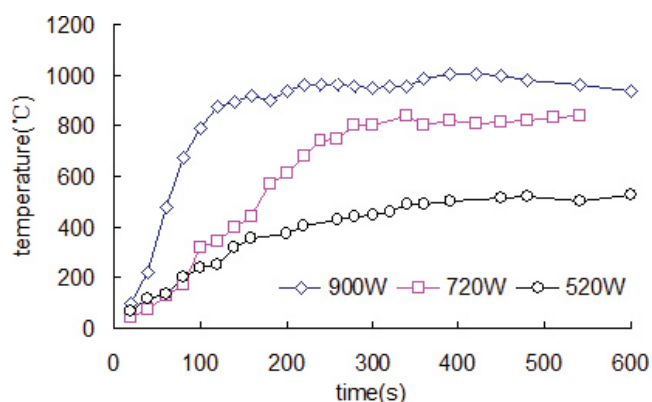


Fig. 2. The influence curve of microwave power.

ture change increases with the increase in the microwave power. At 900 W, the LPG slag was heated up quickly, and the temperature of the 10 g sample went up to 1006°C. The highest temperatures were 510°C and 820°C at 520 and 700 W, respectively. The changing trends in temperature will be unchanged going forward after the temperature at its highest point. Temperature is the most critical factor in the process of activation of AC. The extent of activation of AC has a lot to do with the microwave power and to obtain high-quality AC. It can be concluded that microwaves at 900 W are well suited for the preparation of AC_{LPG}.

3.1.3. Effect of LPG slag dosage

When microwave power was 900 W and no gas was provided, the temperature rise of the LPG slag at different dosages was measured by the thermocouple and recorded as shown in Fig. 3.

It can be seen from Fig. 3 that the rate of increase of temperature reduced obviously along with the increase of LPG slag dosage under the same microwave power (900 W). A 5 g sample of LPG slag was heated quickly, and its temperature increased to 1000°C in 3 min and to 1158°C in 4 min. The 10 g of sample of LPG slag is heated quickly and obviously; the highest temperature it could attain was 1006°C. The 15 g sample of LPG slag was also heated quickly; the highest temperature it could attain was 860°C. The temperature became steady after it reached its highest point. The LPG slag dosage has great influence on the temperature of LPG slag in the process of activation of the AC. The lesser the LPG slag dosage, the higher the microwave absorption and consequently the higher the temperature will be. The extent of activation of AC has a lot to do with the dosage of LPG. A dosage of 10 g of LPG slag is well suited for the preparation of AC_{LPG}.

3.1.4. The heating behavior of KOH

At 900 W, the temperature rise of 10 g KOH was measured and recorded as shown in Fig. 4. It can be seen from Fig. 4 that the temperature was 120°C after 90 s of heating at 900 W. The temperature went up to 160°C and became steady after 4 min. It shows that the absorption of microwave by KOH is weak and the dosage of KOH has a relatively small impact to rise of the temperature.

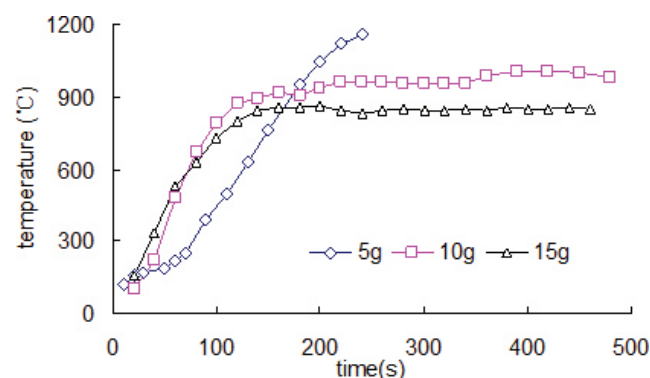


Fig. 3. The influence curve of LPG slag dosage.

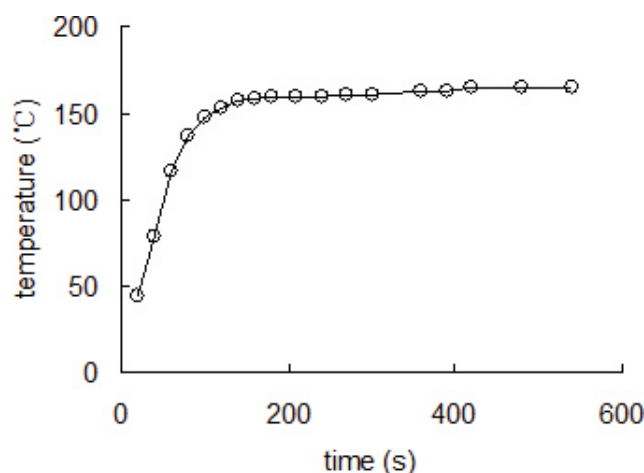


Fig. 4. The heating behavior curve of KOH.

3.1.5. The heating behavior of the mixture of LPG slag and KOH

At the microwave power of 900 W and a mass ratio of LPG slag to KOH of 1:1, the temperature rise in different dosages of mixture were measured and recorded as shown in Fig. 5.

As shown in Fig. 5, the 10 g mixture heated up quickly, and its temperature was 1180°C within 60 s. The 20 g mixture also heated up quickly and went to its highest temperature 940°C in 180 s. The heating rate of the 30 g mixture was the lowest among the three mixtures, and its highest temperature reached to 740°C. The results show that the heating behavior between the mixture and LPG slag alone was very similar because of the weak absorption of microwave by KOH.

From the above experimental results, we could conclude that the microwave power and the LPG dosage are the most important factors affecting the temperature for the activation of AC. When the microwave power is 900 W and the LPG dosage is 10 g, the microwave heating can receive a satisfactory temperature condition for the preparation of AC from LPG slag by microwave-assisted KOH activation.

3.2. Selection of preparation conditions for activated carbon from LPG slag

3.2.1. Determination of activation time

LPG and KOH were mixed using immersed method, and the mass ratio of LPG to KOH was kept at 1:1. After dehydration at 120°C, the mixture was activated for different time periods. The results are shown in Table 2.

The adsorption capacity of iodine value increased from 575.3 to 1114.9 mg g⁻¹ when the microwave radiation time was increased from 0 to 15 min. A possible explanation for this effect is that the microwave heating is uniform with a rapid heating rate, so the reaction between KOH and the char of the LPG is greater at higher-radiation power and thereby promoting the growth of the pore structure and causing the formation of more active sites in a shorter time [9–10]. That will be effective in creating well-developed pores on the surface of LPG and can result in its well-developed and uniform surface. So the iodine value of AC is high, and its adsorption ability is better. From this study, the activation time was determined to be 9 min.

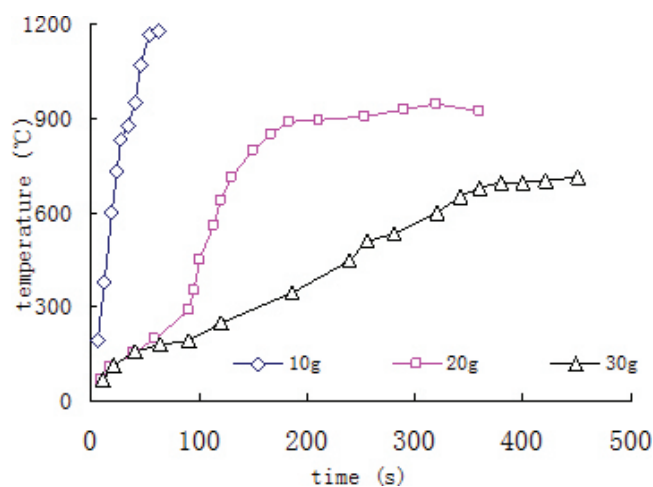


Fig. 5. The heating behavior curve of the mixture of LPG slag and KOH at 1:1 weight ratio.

Table 2
Determination of activation time

Time (min)	0	6	9	10	15
Iodine value (mg g ⁻¹)	575.3	1017.8	1082.2	1114.9	1072.3
yield (%)	65	36	40	40	38

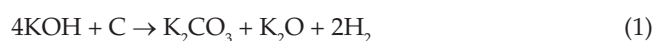
Note: LPG slag dosage = 10 g; mass ratio of potassium hydroxide (KOH) and Lugi pressurized gasifier (LPG) slag = 1:1; microwave power = 900 W

3.2.2. Determination of the mass ratio of LPG slag and KOH

At an activation time of 9 min, the iodine values under different ratios of LPG to KOH are shown in Table 3.

From Table 3, the mass ratio of LPG slag to KOH has remarkable influence on the iodine value AC_{LPG}. When mass ratio increases (between the mass ratio of 1:0.8 to 1:1.2), the iodine value reaches the national standard (GB/T12496.8-1999).

With the increasing of KOH dosage, more potential sites could be penetrated and occupied by the activating agent. However, excessive KOH could not promote the activation further since an insulating layer might be formed due to the presence of K₂O and K₂CO₃ or K residues on the pore surface of char [9]. This will lead to the decline in surface area and decrease in the iodine absorption value. Thus, the activation ratio was chosen as 1:1. General chemical reaction between KOH and carbon material is as follows:



3.2.3. Determination of the LPG slag dosage

At a mass ratio of LPG to KOH 1:1 and an activation time of 9 min, the influence of LPG slag dosage on absorption ability was investigated in Table 4, and the results are shown in Table 4.

Table 3
Determination of the ratio of LPG slag and KOH

Ratio of raw material with activation quality ($M_{LPG}:M_{KOH}$)	0	1:0.6	1:0.8	1:1	1:1.2	1:1.4
Iodine value (mg g^{-1})	632.6	785.2	1039.2	1082.5	1054.5	860.0
yield (%)	61	38	40	42	38	40

Note: LPG slag dosage = 10 g; microwave power = 900 W; activation time = 9 min.

Table 4
Determination of the LPG slag dosage

LPG dosage(g)	5	10	15	20	25
Iodine value (mg g^{-1})	1082.2	1015.4	830.1	785.2	756.4
yield (%)	36	40	43	38	44

Note: Mass ratio of KOH and LPG slag = 1:1; microwave power = 900 W; activation time = 9 min.

From Table 4, it can be seen that there is a great influence of LPG slag dosage on the adsorption ability of AC. The lesser the LPG dosage, the higher the temperature rise and larger the iodine value will be. When the LPG mass is 10 g, the iodine value can reach the national standard (GB/T12496.8-1999). So the dosage of the LPG slag is selected as 10 g.

In short, the optimal conditions for preparing AC_{LPG} by microwave-assisted were determined by single-factor experiments: activation time is 9 min; the dosage of the LPG slag is 10 g; and ratio of LPG to KOH is 1:1.

3.2.4. Contrast with the traditional method

In a previous study, we used the traditional method for AC_{LPG} that was prepared from LPG slag with KOH as activat- ing agent at high temperature. The most suitable conditions

for the traditional method were: activation temperature of 850°C, activation time of 20 min and ratio of LPG to KOH of 1:1 [19]. The experimental data for the traditional method and microwave-assisted method are shown in Table 5.

From Table 5, the properties such as iodine value and yield were comparable with the two methods because the two methods can provide the temperature required for the preparation of AC_{LPG} . But the microwave-assisted activation process had faster activation rate and higher carbon yield than that of the traditional method. Therefore, the micro- wave-assisted method has a bright future for its applications.

3.3. The surface characteristics of activated carbon

The surface area and characteristics of AC_{PLG} prepared by two methods and the raw material (LPG slag) were measured by the BET method. The results are shown in Table 6. The surface areas of AC_{LPG} were in the range of 993–1061 $\text{m}^2 \cdot \text{g}^{-1}$, which is comparable. But the surface area of AC_{LPG} is four times as much as the raw material (LPG slag). There is a large exalta- tion in other indices, such as the micropore area and pore vol- ume, which is consistent with the result of iodine value.

But with the increase in the activation time, the pore wall would collapse due to excessive burning of carbon, and thus its pore volume and surface area will decrease. So activation time is key to prepare AC which has higher sur- face area and more micropore.

3.4. Treatment of coked wastewater using AC_{LPG}

The wastewater from the Coking Plant at Kunming con- tains total phenols of 368.25 mg L^{-1} , a chromaticity concentra- tion value of 1000°, ammonia nitrogen value of 89.65 mg L^{-1} and a COD value of 4663.5 mg L^{-1} . The data were obtained from Environmental Monitoring Department in Kunming. The AC_{LPG} was used to treat this coked wastewater. A 50 mL of this coked wastewater sample was poured into a 250 mL Erlenmeyer flask; known amount of AC_{LPG} was added to the flask, and the contents (25°C) were shaken using an incu- bator shaker for 2 h. The results are shown in Table 7. The removal efficiency of total phenols and chromaticity reached

Table 5
Experimental data obtained by the traditional method and microwave-assisted method

Activation method	$M_{LPG}:M_{KOH}$	Activation time (min)	Activation temperature	Iodine value ($\text{mg} \cdot \text{g}^{-1}$)	Yield (%)
High temperature [19]	1:1	20	850°C	1009.0	38
Microwave-assisted	1:1	9		1082.2	42

Note: LPG slag dosage = 10 g.

Table 6
Surface area characteristics of activated carbon ratio

Activation time (min)	Iodine value ($\text{mg} \cdot \text{g}^{-1}$)	Surface area ($\text{m}^2 \cdot \text{g}^{-1}$)	Total volume ($\text{CC} \cdot \text{g}^{-1}$)	Average diameter (Å)	Micropore area ($\text{m}^2 \cdot \text{g}^{-1}$)	Micropore volume ($\text{CC} \cdot \text{g}^{-1}$)
9	1082.2	1061	0.72	20	481	0.22
15	1072.3	993	0.68	20	459	0.21
LPG slag	350.15	249.7	0.25	29	83	0.038

Note: LPG slag dosage = 10 g; mass ratio of KOH and LPG slag = 1:1; microwave power = 900.

Table 7
Removal rate of contaminants in the coked wastewater by AC_{LPG} adsorption

The activated carbon dosage (g L ⁻¹)	The removal rate (%)			
	Total phenols	Chromaticity concentration	Ammonia nitrogen	COD
0.25	100	96	38.1	58
0.5	100	100	44	85
0.75	100	100	60	78
1	100	100	53	70

almost 100%, and the removal of COD was more than 70%. The removal of ammonia nitrogen was 43.4%.

4. Conclusion

This study has brought out the potential of LPG slag as an efficient raw carbon precursor for the preparation of AC with noticeable adsorption capacity; the study presents the heating behavior of microwaves and the synthesis of AC from LPG (AC_{LPG}) with a microwave-assisted activation process. The results are as follows:

The microwave power and the LPG slag dosage were significant factors affecting the temperature for the activation of the AC. When the microwave power was 900 W and the LPG slag dosage was 10 g, the temperature of LPG slag went up to 1006°C, and the LPG slag heated up quickly. In this case, the microwave heating can provide satisfactory activation temperature for the preparation of AC from LPG slag by microwave-assisted method.

The optimal activation conditions were obtained at microwave power of 900 W, radiation time of 9 min and KOH/LPG slag ratio of 1:1. The obtained iodine value of AC_{LPG} is 1082.2 mg.g⁻¹. The BET surface area was 1061 m².g⁻¹, and the yield was 42%. The AC_{LPG} has higher surface area and larger pore volume, and already exceeds the requirement of national standards (GB/T12496.8-1999). Microwave provides uniform and rapid heating as its energy is readily transformed into heat inside the LPG slag particles by dipole rotation and ionic conduction.

Comparisons with the traditional method process demonstrated that the microwave-assisted activation has faster activation rate and higher carbon yield. Microwave-assisted activation has successfully reduced the heating period which will reduce the costs associated with energy. Its mechanism is basically the same as the traditional heating activation method. AC_{LPG} prepared by microwave-assisted activation method was used to remove total phenols and chromaticity presented in wastewater from the Coking Plant at Kunming.

Microwave heating technique with low-energy and short activated time is one of the most prospective methods to obtain AC.

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