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Effect of preparation methods on the adsorption property of municipal solid waste-based carbon materials

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Abstract: Three different preparation methods including steam physical activation, catalytic carbonation and KOH chemical activation methods were used to prepare municipal solid waste-based carbon materials. The methylene blue (MB) adsorption value was applied to evaluate the adsorption capabilities of the prepared carbon materials. The effects of preparation methods on adsorption capability and yield of products were investigated. The yield of carbon materials with the catalytic carbonation method is the highest, and the KOH activation method is the second level. Considering the adsorption performance, the KOH activation method is much more favorable. Among the different components of municipal solid waste-based carbon materials, the adsorption properties of the single component of paperboard, the double components of tire and paperboard, the triple components of tire, paperboard and polyvinyl chloride (PVC), and the multi-component mixtures are better than those of other single-, double-, triple- and multi-component mixtures, respectively.

Key words: pyrolysis; preparation methods; waste; adsorption; activated carbon

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Activated carbon is the most widely employed adsorbent for its excellent adsorption properties. However, the high cost restricts its application. At the same time, the municipal solid waste (MSW) has increased greatly recently[1-2]. Now MSW has been used as low-cost alternative for producing activated carbon, which is a versatile adsorbent for pollutants removal. The utilization of wastes as a precursor for the preparation of carbon materials has been increasing comprehensively. The MSW has been investigated extensively by pyrolysis materials to evaluate the influences of the operating parameters[3-5].

The commonly used methods for the preparation of activated carbons are physical activation and chemical activation[6]. For the physical activation method, the prepared activated carbon has great adsorptive performance and wide distribution of pore size[7]. While for the chemical activation method, the impregnation with reagents is in favor of developing sufficient pore structures[8-9].

Most research investigates the influence of preparation methods and reaction conditions on single component based carbon. But few reports focus on the mixed components and their relationship with the adsorption capability. So the influence of preparation methods on the adsorption capability of mixed waste component-based carbon products is investigated in this study. Different wastes are used as precursors for the preparation of activated carbons by three different methods. The effects of preparation methods on adsorption performance and the yield of different component-based carbons are compared. In addition, their adsorption kinetic characterizations are analyzed.

1 Method

1.1 Materials

Paperboard, waste tire, toilet tissue, polyvinyl chloride (PVC) and acrylic textile are used as a precursor. Tab. 1 lists their proximate and ultimate analyses. The iron ore provided by the Shanghai Baosteel Co., Ltd. is selected as the catalyst. The iron ores are crushed and sieved to 120 mesh, dried at 120 ℃ for 2 h and then calcined in furnace in air for 4 h at 550 ℃.

1.2 Preparation methods

The preparation procedures for different methods are listed in Fig. 1. The detailed processes are described as follows[10-11].

1) Catalytic carbonation method: 10 g pretreated raw material mixed with the ironore catalyst at different mixed mass ratios is heated to a carbonation temperature of 10 ℃/min and keeps at 600 ℃ for 60 min under a nitrogen atmosphere of 0.5 L/min.

2) Steam activation method: 10 g raw material in a quartz pipe is heated, the same as process 1). Then the reactor goes on heating, and the steam is pumped into the reactor under the N_2 flow of 15 g/h as the temperature reaches 800 ℃, and kept for 90 min under a nitrogen atmosphere of 0.5 L/min.

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Tab. 1 The proximate and ultimate analysis of different precursors %

Components	Proximate analysis				Ultimate analysis			
	$w(V_{daf})$	$w(FC_{ad})$	$w(A_d)$	$w(M_{ad})$	$w(C_{ad})$	$w(H_{ad})$	$w(N_{ad})$	$w(O_{ad})$
Tire	54.54	18.59	24.73	2.14	51.21	4.88	0.48	43.43
Paperboard	71.37	13.47	7.28	7.52	40.82	5.7	0.12	53.37
Toilet tissue	75.14	9.76	8.65	6.45	41.52	5.95	0.09	52.44
PVC	94.06	5.63	0	0.31	51.50	6.37	0	42.13
Acrylic textile	88.34	8.68	1.74	1.24	61.76	4.37	0.18	33.69

Notes: ad—air dry; daf—dry ash free; d—dry basis.

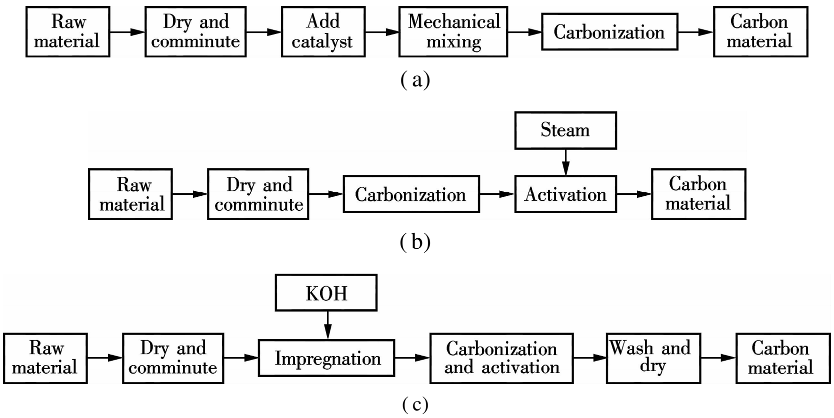


Fig. 1 Preparation procedure of different methods. (a) Catalytic carbonization process; (b) KOH activation process; (c) Stream activation process

3) KOH activation method: 10 g different components are added to a KOH aqueous solution with 1∶3 mass ratio, stirred 6 h at 40 ℃, and then dried at 105 ℃ for 24 h. Subsequently, the impregnated samples are put in a quartz pipe to be activated at 800 ℃ for 60 min under the nitrogen atmosphere. The prepared products are cooled, washed with water, and then dried.

1.3 The methylene blue (MB) adsorption

The adsorption capabilities of the prepared carbon materials are evaluated by China National Standards (GB/T12496.8—1999) to determine methylene blue (MB) adsorption values.

1.4 Adsorption isotherms procedure

0.1 g carbon samples are added into 50 mL MB solution with different concentrations, then stirred at 70 ℃ with different adsorption times. The equilibrium concentrations are determined by the UV-3200 spectrophotometer.

2 Results and Discussion

2.1 Effect of different preparation methods

2.1.1 Preparation conditions

The carbon adsorbents are manufactured by catalytic carbonation, KOH activation and steam activation methods, respectively, with five representative MSW components. A series of carbon adsorbents were prepared under different reaction conditions.

For the catalytic carbonation method, the catalyst ratio is a key factor, so the single component tire is selected as a sample to conduct a pre-experiment test and optimize the influence condition. The effects of the iron ore ratio on the methylene blue value and the yield of tire-based carbon material are investigated. Considering the adsorption capability, the optimum ratio of 15% is selected.

For steam activation and KOH activation methods, the preparation conditions take the previous study[10-11] as reference. The optimum conditions for different preparation methods are shown in Tab. 2, which will be used in the following study.

2.1.2 Comparison of different preparation methods

The single-, double- and triple-component MSW were further used for the preparation of carbon materials by three methods under the optimum conditions. Different numbers representing different samples are shown in Tab. 3. The adsorption values of methylene blue and the yield are taken as the index to illustrate their adsorption capability and production efficiency, respectively.

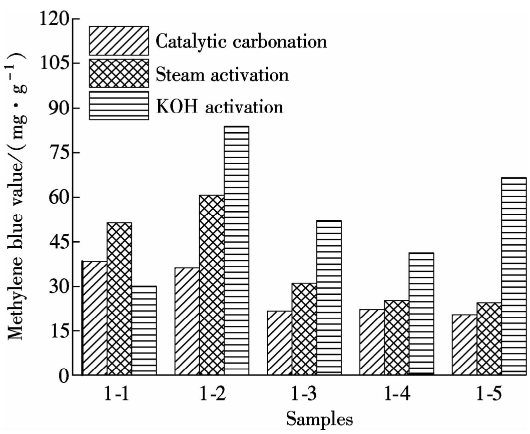
Tab. 2 The optimum conditions of different preparation methods

Preparation methods	Carbonation temperature/℃	Carbonation time/min	Activation temperature/℃	Activation time/min	Catalyst mass ratio/%	Alkali/carbon ratio
Steam activation method	600	60	800	90		
Catalytic carbonation method	600	60			15	
KOH activation method			800	60		3∶1

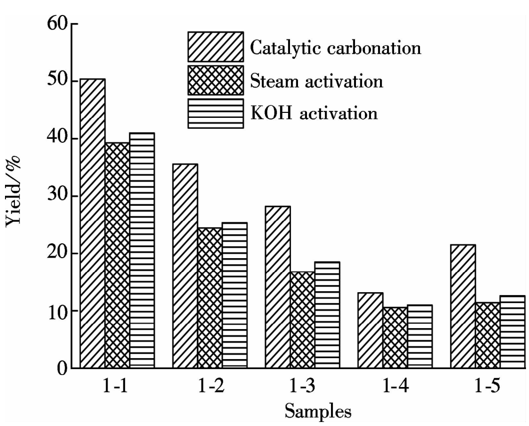
Tab. 3 The representative sample of different numbers

Component	Sample number	Sample name
Single component	1-1	Tire
	1-2	Paperboard
	1-3	Toilet paper
	1-4	Polyvinyl chloride (PVC)
	1-5	Acrylic textile
Double components	2-1	Paperboard + tire
	2-2	Toilet paper + tire
	2-3	PVC + tire
	2-4	Acrylic textile + tire
	2-5	Toilet paper + paperboard
	2-6	PVC + paperboard
	2-7	Acrylic textile + paperboard
	2-8	PVC + toilet paper
	2-9	Acrylic textile + toilet paper
	2-10	Acrylic textile + PVC
Triple components	3-1	Tire + paperboard + PVC
	3-2	Tire + toilet paper + PVC
	3-3	Tire + acrylic textile + PVC
	3-4	Paperboard + toilet paper + PVC

The effects of different preparation methods on adsorption values of methylene blue and the yield of the single component-based carbon materials are shown in Fig. 2. Different preparation methods exhibit different adsorption performances for the same carbon adsorbent. As for the tire, the adsorption performance can be listed as follows: steam activation, catalytic carbonation and KOH activation. While for other four raw materials, the sequence is different: KOH activation, steam activation and catalytic carbonation. Therefore, the steam activation is the optimum preparation method for the tire. The methylene blue adsorption value is 51.3 mg/g for the tire-based carbon materials. However, the KOH activation method exhibits the better adsorption performance for the other four raw materials. The methylene blue adsorption values of paperboard, toilet tissue, PVC and acrylic textile are 83.8, 52, 41.3, 66.6 mg/g, respectively.



(a)



(b)

Fig. 2 Effect of three preparation methods on different single component-based carbon materials. (a) Methylene blue adsorption value; (b) Yield

However, the catalytic carbonation method produces a much higher yield than the other two methods for all five single components. The sequence of the yield is: the catalytic carbonation method, the KOH activation method and the steam activation method. The yield is related to the reaction temperature, retention time, and methods, etc. In general, the yield decreases with the increase in the reaction temperature. Much more carbon element will react with the activating gas with the increase of temperature, leading to the decrease of yield. In our study, due to the presence of the catalyst, the reaction temperature of the catalytic carbonation method decreases to 600 ℃. While the activation temperature for other two methods is 800 ℃. Therefore, the yield of the catalytic carbonation method is higher than those of the other two methods. In addition, the yield will decrease with the increase of the retention time. With the prolonging of the reaction time, the residue substance will decompose and volatilize, resulting in the removal of the little random carbon atom. The activating gas will pass through the carbon materials to accelerate the overflow of the volatile substance with the prolonging of the reaction time, contributing to the decrease of the yield.

In addition, the two-component mixture-based carbons were prepared by three methods. The effects of the mixing ratio investigated to achieve the optimum ratio are shown in Tab. 4. Fig. 3 shows the effects of different preparation methods on the adsorption properties and yield of two-component mixture-based carbon materials under the optimum conditions. The KOH activation method demonstrates a unique advantage, particularly for the mixture of paperboard and tire, paperboard and PVC. However, the sequence of yield is the same as the results of single component-based carbon materials.

Furthermore, the triple-component MSWs are mixed according to the optimum mixing ratio of double components. The mixing ratio are 1∶1∶2, 2∶1∶2, 1∶2∶1, and 1∶1∶2 for sample 3-1, 3-2, 3-3 and 3-4, respectively. The mixtures are prepared by three different methods, which have the same as above experimental conditions. Fig. 4 shows the MB adsorption properties and the yield

Tab. 4 The effect of mixing ratio of double components on methylene blue adsorption value and yield of carbon materials

Samples	Mixing ratio	Methylene blue value/($mg \cdot g^{-1}$)	Yield/%	Optimum mixing ratio
2-1	1∶2	27.6	43.4	1∶1
	1∶1	35.2	41.1	
	2∶1	21.3	39.3	
2-2	1∶2	34.1	41.8	1∶2
	1∶1	25.7	36.1	
	2∶1	32.8	32.2	
2-3	1∶2	31.6	35.5	2∶1
	1∶1	34.8	30.2	
	2∶1	38.7	23.9	
2-4	1∶2	28.3	39.0	1∶2
	1∶1	19.5	33.8	
	2∶1	21.3	25.6	
2-5	1∶2	31.5	25.4	1∶2
	1∶1	23.8	23.9	
	2∶1	23.0	21.7	
2-6	1∶2	19.8	28.7	2∶1
	1∶1	16.6	24.1	
	2∶1	31.0	19.3	
2-7	1∶2	25.4	31.1	1∶2
	1∶1	12.6	26.3	
	2∶1	15.8	22.6	
2-8	1∶2	27.8	20.9	1∶1
	1∶1	31.2	18.1	
	2∶1	19.2	16.2	
2-9	1∶2	21.8	17.9	1∶2
	1∶1	11.0	18.1	
	2∶1	19.0	15.6	
2-10	1∶2	35.7	11.6	1∶2
	1∶1	29.4	13.5	
	2∶1	30.9	16.3	

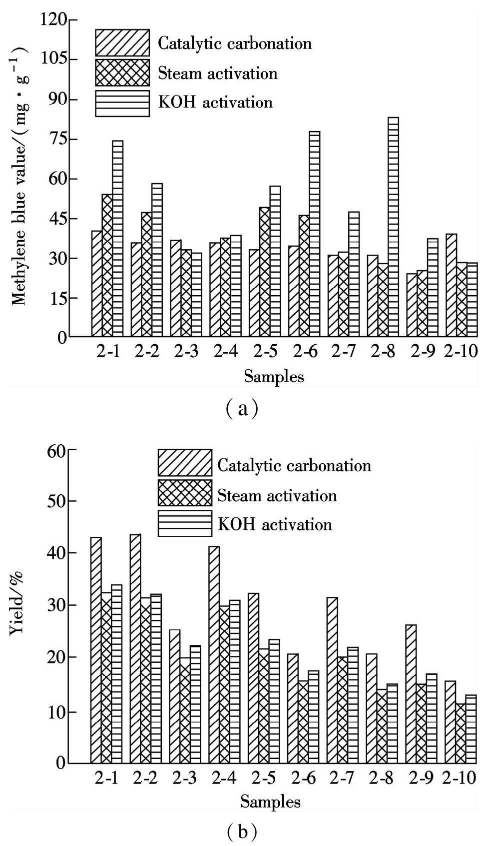


Fig. 3 Effect of three preparation methods on different double component-based activated carbon. (a) Methylene blue adsorption value; (b) Yield

of triplecomponent-based carbon, illustrating the effect of the preparation method.

For different triple-component mixtures, the preparation methods have different effects on their adsorption characteristics. For sample 3-1 and sample 3-2, the carbon materials prepared with the catalytic carbonation method show the highest adsorption value. For sample 3-3, the steam activation method is better than the other two methods. However, the KOH activation method is the best method for sample 3-4. The preparation method has little effect on the adsorption of the other three mixture-based carbon materials. However, the yield of carbon materials prepared by the catalytic carbonation method is higher than that of the other two methods, which is the same as the single and double component-based carbon materials.

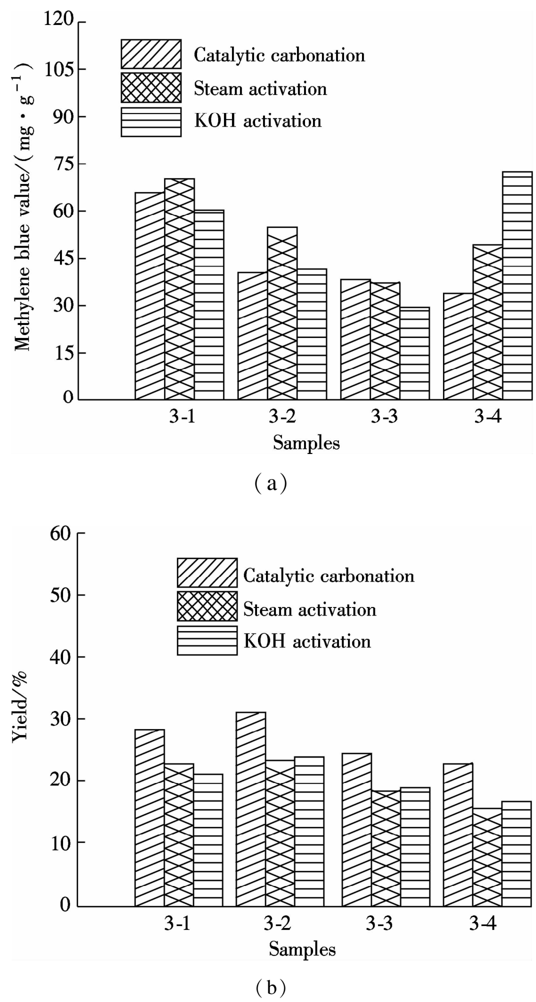


Fig. 4 Effect of three preparation methods on different triple component-based activated carbon. (a) Methylene blue adsorption value; (b) Yield

According to the survey of the real proportion of municipal solid waste, the multi-component raw materials are mixed with the ratio of 1∶8∶8∶12∶3 for tire, paperboard, toilet paper, PVC and acrylic textiles. The above multi-component raw materials are further used to prepare carbon by three methods. Fig. 5 shows the comparison of methylene blue adsorption values and yield. It is found that the carbon materials exhibit the highest adsorption value by the KOH activation method. However, the sequence of yield is the catalytic carbonation method, the KOH activation method and the steam activation method. Based on the above three preparation methods, the KOH activation method shows the best performance with 54.7 mg/g and 21.4% for their methylene blue adsorption value and the yield, respectively.

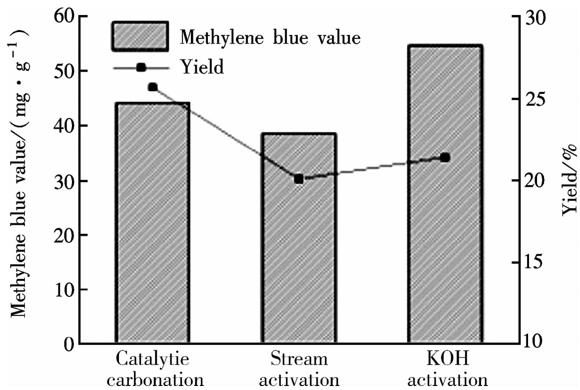


Fig. 5 Effect of three preparation methods on the methylene blue adsorption value and yield of multi-component-based carbon

2.2 The methylene blue adsorption kinetic of different component-based carbon materials

The methylene blue adsorption rate curves of different components based carbon materials are presented in Fig. 6. The results indicate that the different components-based carbon materials exhibit similar dynamic behavior. The adsorption processes are composed of three stages: rapid adsorption, slow adsorption and approaching an equilibrium. As shown in Fig. 6, the methylene blue adsorption capacity of carbon materials increases with the increase of time. However, their adsorption equilibrium times are different. The equilibrium time of AC2 is 3 h; the others based carbon materials are all 2 h approximately.

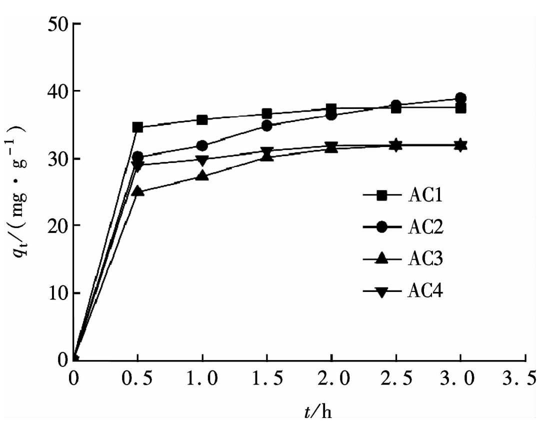


Fig. 6 Methylene blue adsorption rate curves of different component-based carbon materials

The methylene blue adsorption process is a dynamic process that includes the adsorption and desorption processes. The methylene blue molecules that crashed on the activities' sites on the surface of carbon materials are entrapped in the adsorption process. Then, methylene blue molecules are adsorbed on the surface divorce from carbon materials in the desorption process. Generally, the two processes continue during the whole adsorption. The Langmuir adsorption isotherm is used as the adsorption isotherm model to study the adsorption equilibrium. The Langmuir equation is described as

$$\frac{C_e}{q_e} = \frac{1}{q_0 b} + \frac{C_e}{q_0} \tag{1}$$

where q_e is the adsorption amount in equilibrium, mg/g; C_e is the concentration in equilibrium, mg/L; b is the Langmuir constant; q_o is the single saturated adsorption quantity, mg/g. Fig. 7 shows the Langmuir isotherms' plots of different carbon materials. The results demonstrate that the Langmuir adsorption isotherm model can accurately describe the adsorption phase equilibrium of methylene blue on carbon materials. The adsorption is monolayer adsorption and it is dominated by physical adsorption. The maximum methylene blue adsorption capacity of AC1, AC2, AC3, AC4 is 85.84, 74.52, 61.84, 57.67 mg/g respectively. The Langmuir fitting parameters for methylene blue adsorption data on different component-based carbon materials are presented in Tab. 5. The AC1 exhibits the highest adsorption capacity than the other three kinds of carbon. The reason may be ascribed to the decrease of the surface area due to the block of micropores after mixing with other components[12]. In particular, the adsorption values decrease significantly after the addition of PVC.

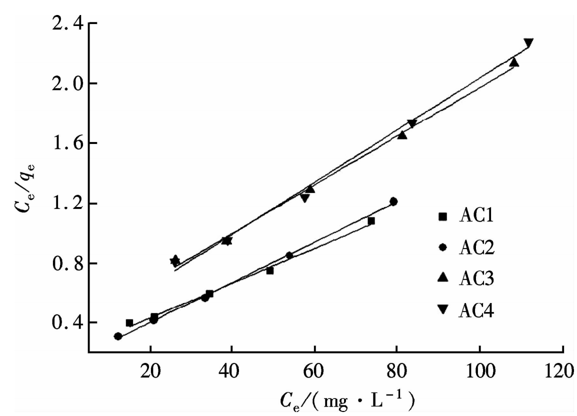


Fig. 7 Langmuir isotherms plots of different carbon materials

Tab. 5 Langmuir fitting parameters of methylene blue adsorption isotherm for different carbon materials

Sample	q_o/(mg · g^{-1})	b	R^2
AC1	85.84	0.057	0.994 4
AC2	74.52	0.097	0.998 8
AC3	61.84	0.046	0.995 0
AC4	57.67	0.057	0.991 9

In order to explore the methylene blue adsorption kinetics of different components-based carbon materials, the pseudo-first order, pseudo-second order and intra particles diffusion models are employed to fit methylene blue adsorption of carbon materials. The related parameters obtained by fitting are shown in Tab. 6.

Tab. 6 The kinetic parameters of methylene blue adsorption of carbon materials

Sample	$q_{e,exp}$/ (mg · g^{-1})	The pseudo-first order			The pseudo-second order			Intra particles diffusion	
		$q_{e,cal}$/ (mg · g^{-1})	k_1/ min^{-1}	R^2	$q_{e,cal}$/ (mg · g^{-1})	k_2/ (g · mg^{-1} · min^{-1})	R^2	k_p/ (mg · g^{-1} · min$^{-0.5}$)	R^2
AC1	37.5	12.31	2.16	0.81	38.12	0.540	0.999 6	3.02	0.92
AC2	38.9	18.07	1.07	0.94	41.96	0.088	0.997 6	8.93	0.99
AC3	31.9	20.94	1.77	0.94	34.52	0.130	0.998 4	7.22	0.91
AC4	32.0	13.76	2.21	0.81	32.71	0.470	0.999 8	3.21	0.91

The pseudo-first order kinetics model equation is described as

$$\ln(q_e - q_t) = \ln q_e - k_1 t \tag{2}$$

The pseudo-second order kinetics model equation is described as

$$\frac{t}{q_t} = \frac{1}{(k_2 q_e^2)} + \frac{t}{q_e} \tag{3}$$

The intra particles diffusion model is described as

$$q_t = k_p t^{0.5} + C \tag{4}$$

where t is the adsorption time, h; q_t, q_e are the adsorption amount in time t and equilibrium separately, mg/g; k_1 is the adsorption rate constant, min^{-1}; k_2 is the secondary reaction kinetics rate constant, g/(mg · min); $k_2 qe^2$ is the initial adsorption rate, mg/(g · min); k_p is the particle diffusion rate constant, mg/(g · min$^{0.5}$); and C is the constant.

By the comparison of experimental equilibrium adsorption values with the calculated adsorption values, the results illustrate that the adsorption amount at equilibrium of the pseudo-first order model exhibits a large difference with actual adsorption capacity. While the difference between the adsorption amount at equilibrium of the pseudo-second order and the experimental values is small, it fits well. So, the methylene blue adsorption of AC1, AC2, AC3, and AC4 are more consistent with the pseudo-second order model.

Furthermore, the pseudo-second order model has a higher correlation coefficient R^2. All the coefficients are greater than 0.997, which is higher than the correlation coefficient of the intra particles diffusion model and the pseudo-first order model. All the processes of adsorption are included in the quasi two stage dynamic model, such as external film diffusion, intra particle diffusion and surface adsorption etc[13]. Therefore, the methylene blue adsorption mechanism of carbon materials can be demonstrated truly.

3 Conclusion

The carbon materials were prepared by KOH chemical activation, catalytic carbonation and steam physical activation methods with different components. In general, the effects of the three preparation methods on methylene blue adsorption values and yield of the single, double, triple and multi-component-based carbon materials are compared comprehensively, suggesting that the KOH activation method is the relatively advantageous method compared with the other two methods. Among different components, the paperboard-based carbon (AC1), the double components of tire and paperboard (AC2), the triple components of tire, paperboard and PVC (AC3), and the multi-component mixture of tire, paperboard, toilet paper, PVC, acrylic textile (AC4) are better than the other components among the single-, double-, triple- and multi-component mixture, respectively. Their methylene blue adsorption capacities are 85.84, 74.52, 61.84, 57.67 mg/g, respectively, which illustrates their potential application.

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制备方法对城市固体废弃物基炭材料吸附性能的影响

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摘要:采用水蒸气物理活化法、催化炭化法和 KOH 活化法制备了城市固体废弃物基炭材料. 利用亚甲基蓝吸附值来评价制备碳材料的吸附特性,研究了不同制备方法对碳材料得率和吸附容量的影响. 其中催化炭化法制备的碳材料的得率最高;KOH 活化法次之. 就吸附性能而言,KOH 活化法是一种更好的活化方法. 在不同组分固体废弃物基碳材料中,单组分的纸板,双组分的轮胎和纸板,三组分的轮胎、纸板. PVC 及多组分混合物混合制备的碳材料的吸附特性要分别优于其他单组分、双组分、三组分及多组分混合物.

关键词:热解;制备方法;废弃物;吸附;活性炭

中图分类号:TK09