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Study on activated carbon derived from sewage sludge for adsorption of gaseous formaldehyde

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ABSTRACT

The aim of this work is to evaluate the adsorption performances of activated carbon derived from sewage sludge (ACSS) for gaseous formaldehyde removal compared with three commercial activated carbons (CACs) using self-designing adsorption and distillation system. Formaldehyde desorption of the activated carbons for regeneration was also studied using thermogravimetric (TG) analysis. The porous structure and surface characteristics were studied using N_2 adsorption and desorption isotherms, scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR). The results show that ACSS has excellent adsorption performance, which is overall superior to the CACs. Adsorption theory indicates that the ACSS outperforms the CACs due to its appropriate porous structure and surface chemistry characteristics for formaldehyde adsorption. The TG analysis of desorption shows that the optimum temperature to regenerate ACSS is 75 °C, which is affordable and economical for recycling.

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1. Introduction

Formaldehyde, one of the most representative volatile organic compounds (VOCs), has brought a critical concern recently, since it brings serious harms to human health even at a very low concentration. Long term exposure to formaldehyde will result in eye, nose and throat irritation because it is pungent to the mucosa of them. Meanwhile, formaldehyde acts as a lachrymator to cause sneezing and coughing and sometimes leads to acute poisoning, dermal allergies and allergic asthma (Kim and Kim, 2005; Zhang et al., 2009). What is worse, formaldehyde is a human carcinogen and potential leukemogen (Tang et al., 2009). The International Agency for Research on Cancer (IARC) classified it as a Group 1 carcinogen to humans (Agents Classified by the IARC Monographs, 2010). Therefore, removal of formaldehyde is extremely urgent and meaningful.

Adsorption is a simple and effective method to remove formaldehyde from air. As many publications reported, activated carbons (ACs) were extensively used as highly effective adsorbent for adsorption of formaldehyde from air (Boonamnuayvitaya et al., 2005; Li et al., 2008; Lee et al., 2010). However, despite the outstanding performance of ACs, their adsorption remains an expensive treatment process because commercially available ACs are usually derived from non-renewable and relatively expensive raw materials such as coconut shell (Gratuito et al., 2008), bituminous coal (Sze and McKay, 2010) and wood (Yorgun et al., 2009). As a result, there is much monetary incentive for identifying alternative resources for manufacturing affordable activated carbon.

In recent years, many researchers have been paying a growing attention to ACs from industrial or agricultural by-products and waste materials, for example, wood particleboard wastes (Girods et al., 2009), sugar beet bagasse (Demiral and Gündüzoğlu, 2010), waste biomass (Nunes et al., 2009) and poultry litter (Qiu and Guo, 2010). Sewage sludge, the inevitable by-product of wastewater purification, has also been studied for beneficial disposal and reuse (Liu et al., 2010; Ros et al., 2006; Wang et al., 2008). Nevertheless, activated carbons derived from sewage sludge (ACSS) are mainly used for wastewater treatment (Smith et al., 2009), for instance, alkaline-black adsorption (Fan and Zhang, 2008), heavy metals adsorption (Rozada et al., 2008) and mercury removal from water (Zhang et al., 2005), as well as a few applications on gas purification such as NO treatment and H_2S removal (Cha et al., 2010; Seredych et al., 2008), but seldom even never used for formaldehyde removal.

Therefore, the authors optimized the preparation of ACSS in order to find a high-performance and affordable activated carbon

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from sewage sludge for adsorption of gaseous formaldehyde (Appendix A). The present paper investigated the adsorption and desorption performances of the optimum ACSS to evaluate its superiority compared with three commercial activated carbons (CACs) derived from coconut shell (ACCO), wood (ACWD) and coal (ACCA). The porous structure and surface characteristics of the four activated carbons were studied using N_2 adsorption and desorption isotherms, SEM and FTIR for result explanation.

2. Methods

2.1. Materials

The dewatered sewage sludge was collected from the Second Municipal Wastewater Treatment Plant of ChangSha, PR China. All of the chemicals were of analytical grades. Deionized water was employed for preparation of all required solutions.

2.2. Preparation of activated carbon derived from sewage sludge

Dewatered sewage sludge was used as raw material for preparation of ACSS. The sludge sample was first dried at 110 °C for 24 h, crushed and sieved into a granular size less than 20 mesh. Then the resulted particles were carbonized in muffle furnace at 450 °C for 1.5 h in the presence of N_2 with flow rate of 0.9 L/min. After that, a 10.5 g of sample was impregnated into 6 M $ZnCl_2$ solution at the solid–liquid ratios of 1:1 (w/w) for 5 h at 85 °C. After the supernatant liquid was removed, the sample was dried at 105 °C for 12 h and subsequently activated in a tube-type electronic heating furnace with purified nitrogen as ambient gas. In activation step, the temperature of the furnace rose with the rate of 10 °C/min to reach 750 °C. The sample was then kept at 750 °C for 120 min and the nitrogen gas flow was maintained at 0.5 L/min. After activation, the product was washed five times with 3 M HCl and then rinsed with deionized water until the pH of washing effluent reached 6–7. At last, the final product was dried at 105 °C to a constant weight and sieved into a uniform granular size from 60 to 20 mesh for use.

2.3. Characteristics

The proximate analysis was conducted according to ASTM D3173-3175 standards and the results were expressed in terms of moisture, ash, volatile matter and fixed carbon contents. The Elemental Analyzer vario EL cube III (Elementar Ltd. Corp., Germany) was employed for ultimate analysis. Porous structure characteristics of the activated carbons were determined from N_2 adsorption and desorption isotherms that were measured at 77 K using an automatic Micromeritics ASAP2020M+C volumetric sorption analyzer (Micromeritics Instrument Corp., USA). Surface physical morphology was observed by a scanning electron microscopy JSM-6700F (Japan Electron Optics Laboratory Ltd. Corp.). Fourier transform infrared spectroscopy (FTIR) was utilized to qualitatively identify the functional groups. The spectra were measured from 4000 to 400 cm^{-1} and recorded on a Nicolet 6700 FT-IR Spectrometer (Thermonicolet Ltd. Corp., USA) using the KBr pellets containing 1% of activated carbon samples. Prior to the measurement, the pellets about 12.5 mm in diameter and 1 mm in thickness were prepared in a manual hydraulic press at 10 ton and dried overnight at 100 °C before the spectra were recorded.

2.4. Adsorption and desorption performances of gaseous formaldehyde

The experimental apparatus for adsorption performances of ACSS and the CACs mainly consisted of a vapor generation system and a distillation system. The vapor generation system was composed of a compressor, a flow control valve, an air cleaner, an electric-heated thermostatic water bath, a formaldehyde vapor generator, a buffer flask, three rota meters, three adsorption columns, a tail gas treating unit, six t-type sampling ports and a four-way valve. The distillation system was mainly made up of a 200 mL distillation flask, a spirit lamp, a condenser tube and a 200 mL conical flask.

Flowing air purified by air cleaner passed through formaldehyde solution in the formaldehyde vapor generator at the flow rate of 1.5 L/min to generate formaldehyde vapor. Then, the formaldehyde vapor effluent was directed at the flow rate of 0.5 L/min to three adsorption columns, respectively, by switching a four-way valve. The columns had an inner diameter of 0.5 cm and a length of 5 cm. Fine meshes were installed at both ends of the columns to ensure a uniform gas distribution and prevent the carry-over of the adsorbent particles. Each column was loaded 0.40 g ACSS sample. The concentrations of formaldehyde vapor effluent at inlet and outlet of the adsorption columns were monitored at the t-type sampling port. Temperature of the columns was maintained at 30 °C throughout the experiment. Prior to each adsorption run, the formaldehyde vapor effluent was prepared, directed off-line until the formaldehyde vapor concentration remained stable for 60 min.

Removal efficiency of each adsorption column was determined with the following equation:

$$\eta = \frac{C_0 - C}{C_0} \times 100\% \quad (1)$$

where η = removal efficiency (%), C_0 = inlet concentration (mg/m^3), C = outlet concentration (mg/m^3).

It was defined that when the removal efficiency of three columns was all less than 5%, the adsorption reached equilibrium. Under the condition of equilibrium, the adsorption capacity was measured using the following method. Firstly, the saturated adsorbents coming from one of the three columns were put into 200 mL distillation flask and impregnated with 150 mL deionized water. Secondly, after the liquid was distilled, the condensate was collected from the conical flask. Thirdly, the condensate was diluted and the concentration of formaldehyde was determined using acetylacetone UV/VIS spectrophotometry at 413 nm (Chinese, GB/T 15516, 1995). At last, the adsorption capacity was calculated according to the formaldehyde content of the condensate and dosage of adsorbents in the column. Likewise, the measurements were replicated for the other two columns.

In this work, the 20% (v/v) and 1% (v/v) formaldehyde solution was used to generate 498 mg/m^3 and 0.41 mg/m^3 formaldehyde vapor for adsorption performances of ACSS and the CACs measurement, respectively. The obtained results of three columns were averaged and then reported for comparison. The relative standard deviation (RSV) of the results was less than 5%.

After saturation adsorption of 498 mg/m^3 formaldehyde, desorption of formaldehyde from the ACSS and the CACs was investigated using thermogravimetric analyzer STA409PC/4/H (NETZSCH Ltd. Corp., Germany). In this experiment, about 10 mg adsorbent was put into the instrument, and then the ambient temperature was raised from 25 °C to 250 °C by the ratio of 5 °C/min. The weight loss of adsorbent was recorded for thermogravimetric (TG) and derivative thermogravimetric (DTG) curves.

3. Results and discussion

3.1. Proximate analysis and ultimate analysis

The granular ACSS prepared on the condition mentioned above was black and inodorous with the bulk density of 0.45 mg/cm^3 and the final yield of 32.6 wt.%. The results of proximate analysis and ultimate analysis for ACSS are listed in Table 1. For comparison, sewage sludge dried at 110 °C (SS) and CACs are also listed. As can be seen from Table 1, SS had high volatile matter which was a key requirement for justifying its suitability as activated carbon precursor because the volatile matter would be ejected by high temperature treatment during which a rudimentary pore structure was formed. The resulting char could then be activated further to increase adsorption capacity. Though the fixed carbon content of SS was very low, after activation, the percentage of fixed carbon of ACSS increased greatly and the ACSS was observed to have high carbon content which was almost the same as the three CACs. That was attributed to activating agent serving as dehydrating agents which influenced the pyrolytic decomposition and inhibited the formation of ash, thus enhancing the carbon yield (Lu (Max) and Lau, 1996). Other elements (N, H, S and O content) were various, which might result in different chemical characteristics of the activated carbons by forming various functional groups.

3.2. Physical morphology of the activated carbons

The scanning electron microscopy (SEM) images of the ACSS (1#) and the commercial activated carbon derived from wood (ACWD, 2#), coconut shell (ACCO, 3#) and coal (ACCA, 4#) were analyzed and compared using statistics. The results showed that the surface physical morphology of the activated carbons (ACs) was various: from 1# to 4#, the average pore diameter on the surface was 20.18 nm, 26.84 nm, 22.77 nm and 14.23 nm according to a statistics of 100 pores, respectively; the pore shape of ACSS and ACWD was irregular and that of the ACCO was almost ellipse while the pores of the ACCA were few even no somewhere. Those pores with different size and shape might affect the adsorption performances of ACs in different degrees because the adsorbate should firstly pass through them before arriving at the adsorptive site.

3.3. Characteristics of the porous structure

The N_2 adsorption and desorption isotherms of the ACs are shown in Fig. 1. The lower branch of each isotherm represented measurements obtained by adsorption and the upper branch was obtained by desorption. As can be seen from Fig. 1, the ACSS, ACWD and ACCA exhibited a type I with somewhat type IV character nitrogen isotherm in the BDDT classification, because they had both a sharp rise at low relative pressure and a hysteresis loop. That indicated they were microporous carbons with a considerable development of mesoporosity because the sharp rise lied in the micropore filling and the hysteresis loop was associated with capillary condensation of mesoporous solids (Brunauer et al., 1940). All of the three isotherms showed a B-type hysteresis loop in de Boer's classification where the adsorption branches were steep at saturation vapor pressure while the desorption branches were steep at the middling relative pressure, which was the characteristic of the parallel slit-shaped pores (de Bore, 1958). The isotherm of ACCO exhibited type I character with a small hysteresis loop, which indicated that micropores were the major component of ACCO. Because the micropore filling occurred significantly at relatively low partial pressures <0.1 P/P_0 and the adsorption process completed at 0.5 P/P_0.

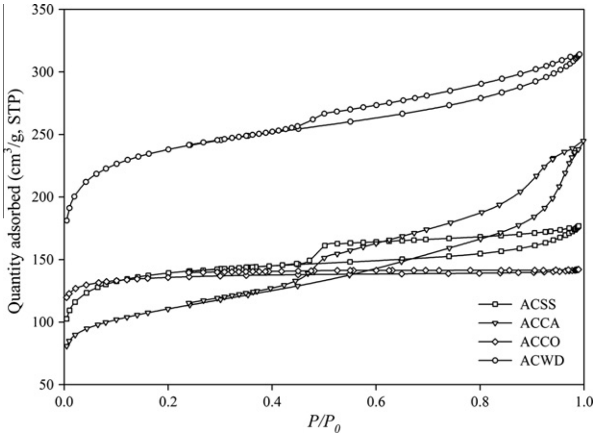

Fig. 1. N_2 adsorption and desorption isotherms of the ACs.

The structural parameters calculated from nitrogen isotherms are listed in Table 2, including total BET surface area (S_{BET}), t-plot micropore area (S_{Micro}), percentage of micropore area (PS_{Micro}), single point adsorption total pore volume calculated at $P/P_0 = 0.98$ (V_{Total}), t-plot micropore volume (V_{Micro}), percentage of micropore volume (VP_{Micro}) and BJH desorption average pore diameter (D_{Ave}). It can be seen that the BET surface area of ACSS was 509.88 m^2/g, which was next to that of ACWD but higher than ACCA and ACCO. The ACCO had the highest percentage of micropore area followed by ACSS, ACWD and ACCA, and the percentage of micropore volume was also in this order. The sequence of average pore diameter of the ACs was ACCO < ACSS < ACWD < ACCA.

3.4. Surface chemistry characteristics

The FTIR spectrum of the ACs presented the similar shape but different strength, which indicated that the ACs had almost the same species but different content of functional groups on their surface. The main functional groups of the ACs were deduced as follows: 3450 cm^{-1}, —OH and —NH$_2$; 1650 cm^{-1}, C═O; 1560 cm^{-1}, —NO$_2$; 1385 cm^{-1}, —CH.

Firstly, a strong and broad peak was displayed on the spectrum near 3450 cm^{-1}, which might be due to O—H or N—H stretching vibration. Generally, for free —OH groups, the band appeared sharply at approximately 3700 cm^{-1}, but in the presence of the hydrogen bonding, the band shifted to lower wave numbers and broadens (Jung et al., 2001). Meanwhile, the weak band appearing in the 615–620 cm^{-1} region confirmed the presence of —NH$_2$. Thus,

Table 1
Results of proximate analysis and ultimate analysis.

Sample	Proximate Analysis (wt.)				Ultimate Analysis (wt.)				
	Moisture (%)	Volatiles (%)	Fixed carbon [a] (%)	Ash (%)	C (%)	N (%)	H (%)	O [a] (%)	S (%)
SS	6	49.4	30.7	13.9	66.7	9.3	9.2	14.3	0.5
ACSS	2.9	12.5	77.2	7.4	89.8	1.2	3.1	5.1	0.8
ACWD	2.2	9.8	85.3	2.7	92.1	1.6	3.3	2.6	0.4
ACCO	1.8	8.9	87.7	1.6	90.7	1.3	3.7	3.6	0.7
ACCA	2.7	10.5	78.2	8.6	87.2	2.1	5.3	2.1	3.3

[a] By difference.

Table 2
Characteristics of the porous structure of activated carbons.

AC samples	S_{BET} (m^2/g)	S_{Micro} (m^2/g)	PS_{Micro} (%)	V_{Total} (cm^3/g)	V_{Micro} (cm^3/g)	PV_{Micro} (%)	D_{Ave} (nm)
ACSS	509.88	353.83	69.39	0.301	0.161	53.49	4.85
ACCO	453.79	393.06	86.62	0.219	0.184	84.02	3.71
ACCA	381.32	184.87	48.48	0.365	0.085	23.029	6.20
ACWD	811.80	532.93	65.65	0.482	0.247	51.24	5.24

it was sure that there were —OH and —NH_2 on the surface of the ACs. Next, there were two peaks displayed at band 1650 and 1560 cm^{-1}, which were related to the C═O and —NO_2, respectively. The peaks at 1458 and 845 cm^{-1} were also assigned to the -NO_2 and the peak at 1130 cm^{-1} confirmed the presence of C═O (Daifullah and Girgis, 2003). In the end of the characteristic spectrum band, a strong peak at band 1385 cm^{-1} detected the presence of C—H whose stretching vibration appeared overlaid by the 3450 cm^{-1} band of —OH and —NH_2 (Bio-Rad Laboratories Inc., 1978). In conclusion, there were mainly hydrophilic groups such as —OH, —NH_2, —NO_2 and C═O on the surface of the ACs and the major hydrophobic group was —CH.

3.5. Adsorption performances of formaldehyde

Adsorption capacity and initial removal efficiency of 498 mg/m^3 and 0.41 mg/m^3 formaldehyde were tested respectively and the results are shown in Fig. 2. As it is shown in Fig. 2(A), at concentration of 498 mg/m^3, ACWD had the largest adsorption capacity of formaldehyde followed by ACSS, ACCO and ACCA. The initial removal efficiency was in the same order. From Fig. 2(B), at concentration of 0.41 mg/m^3, it can be found that the highest initial removal efficiency still belonged to ACWD, and ACSS showed the second highest removal efficiency. However, the largest adsorption capacity belonged to ACCO, and ACSS exceeded that of ACWD and ACCA. In order to find out the reason of adsorption capacity change at concentration of 0.41 mg/m^3, the reduction of removal efficiency with time was investigated and Fig. 3 illustrates the adsorption profiles of 0.41 mg/m^3 formaldehyde. From the curves, it can be observed that though the initial removal efficiency of ACWD was the highest, about 12 h later, it was lower than that of ACCO and ACSS. Especially, after 25 h, there was a remarkable decrease which would significantly reduce the adsorption capacity of ACWD. The removal efficiency of ACSS could keep at a relatively high level for at least 30 h, so its adsorption capacity was higher than that of ACWD and ACCA.

At last, the adsorption performances mentioned above could be summarized as follows: ACWD showed the best adsorption performance at formaldehyde concentration of 498 mg/m^3, while, at concentration of 0.41 mg/m^3, it was inferior to ACCO and ACSS; ACSS performed excellently both at concentrations of 0.41 mg/m^3 and 498 mg/m^3. Though the adsorption performance of ACSS was invariably at the second place, the overall adsorption performance was optimum comparing with that of ACWD, ACCO and ACCA with consideration of all conditions. The adsorption performance of ACCA was always the worst no matter in which concentration. Those phenomena will be explained and further confirmed in the following text.

Normally, surface area is an important attribute while considering the adsorption of organic compounds and large surface area signifies high adsorption of organic compounds (Bansode et al., 2003). Meanwhile, due to high concentration of adsorbate, the vacant surface sites available for adsorption may be rapidly and completely occupied and multilayer sorption occurs subsequently, so adsorption performance may mainly depend on the

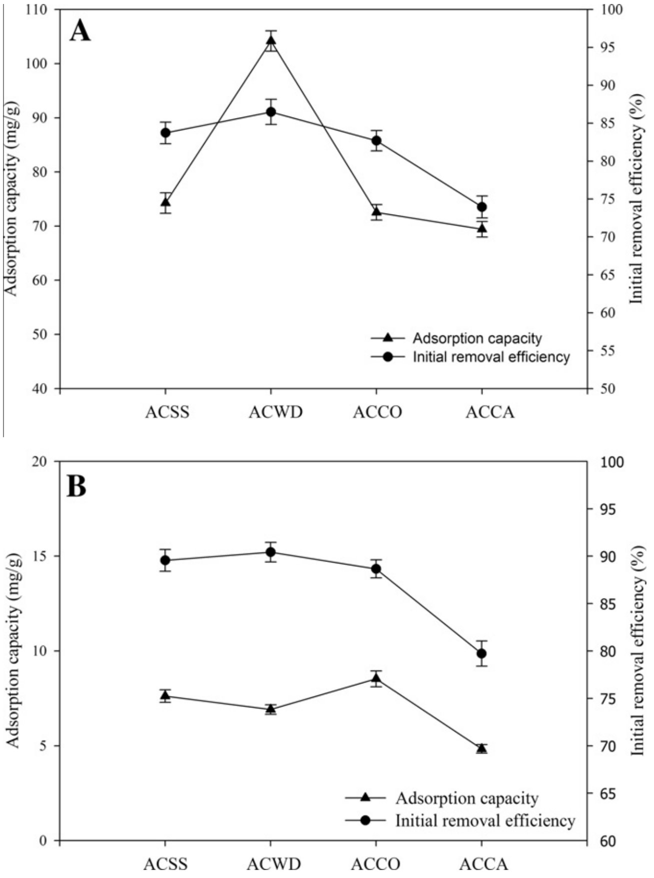

Fig. 2. Formaldehyde adsorption performances of the activated carbons: (A) formaldehyde concentration, 498 mg/m^3; (B) formaldehyde concentration, 0.41 mg/m^3.

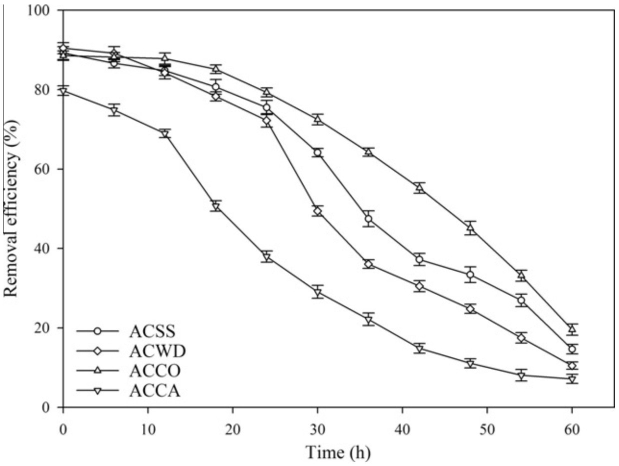

Fig. 3. Formaldehyde removal profiles of the ACs.

surface area. As a result, it was logical that ACWD had the best adsorption performance at concentration of 498 mg/m^3 followed by ACSS, ACCO and ACCA. However, at concentration of 0.41 mg/m^3, adsorption performances of ACs may be significantly affected by their porous characteristics and surface chemistry because the monolayer adsorption may be in the highest flight. Firstly, micropore is superior in adsorbing low concentration adsorbate because of micropore filling which occurs significantly at relatively low partial pressures (Kondo et al., 2005). From Table 2, it can be seen that the percentage of micropore area and volume were in the following order: ACCO > ACSS > ACWD > ACCA, as a result, ACCO had the best performance of 0.41 mg/m^3 formaldehyde adsorption followed by ACSS, ACWD and ACCA. However, ACWD still showed the highest initial removal efficiency, because its average pore diameter was larger than that of ACCO and ACSS and it was easier for formaldehyde passing through the larger pores and arriving at the adsorption sites. However, despite ACCA showed the largest average pore diameter, its initial removal efficiency was still the lowest. That was because the average pore diameter on its surface was the smallest, which greatly reduced the removal efficiency. Secondly, the hydrophilic groups can increase the adsorption of formaldehyde (Boonamnuayvitaya et al., 2005). According to strength of the spectrum mentioned in Section 3.4, the content of each hydrophilic group were all in the following order: ACCO > ACSS > ACWD > ACCA, which further ensured the superior adsorption performance of ACCO and ACSS at concentration of 0.41 mg/m^3. Nevertheless, at the concentration of 498 mg/m^3, because the surface of the adsorbents was overlaid with multilayer formaldehyde, the influence of functional groups was slight.

3.6. Thermogravimetric analysis of formaldehyde desorption

Ordinarily, desorption of ACs should be studied for regeneration. In this section, thermogravimetric analysis was employed for desorption of formaldehyde and its TG and DTG curves were made. Firstly, the result showed that there was only one range from 60–125 °C on each of the TG curves presented a significant weight loss, which was mainly due to formaldehyde desorption. Meanwhile, desorption of formaldehyde from the ACs mostly finished after 100–125 °C, therefore, heating-up for formaldehyde regeneration was feasible. Secondly, though the DTG curves showed different peaks in the range of weight loss temperature, all of the peaks presented before 100 °C, which indicated the fastest desorption temperature was lower than 100 °C and it was affordable to regenerate the ACs using heating-up method at that temperature. Besides, it was worth mentioning that the fastest desorption temperature of ACSS was 75 °C which was more economical than ACCO and ACWD.

4. Conclusions

In the present study, the ACSS was prepared and its adsorption performances were measured. At formaldehyde concentration of 498 mg/m^3 and 0.41 mg/m^3, the adsorption capacity could achieve 74.27 mg/g and 7.62 mg/g and the initial removal efficiency was 83.72% and 89.56%, respectively. Compared with another three commercial activated carbons, ACSS exhibited excellent adsorption performances at both concentrations of 498 mg/m^3 and 0.41 mg/m^3 owing to its higher surface area and percentage of micropores combined with hydrophilic functional groups of $-OH$, $-NH_2$, NO_2 and $C{=}O$. Its thermogravimetric analysis of formaldehyde desorption indicated that reuse of ACSS was economical and affordable by heating-up method.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.biortech.2010.09.042.

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