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Thermal characteristics and regeneration analyses of adsorbents by differential scanning calorimetry and scanning electron microscope

**Chung-Hwei Su · Sheng-Hung Wu · Sun-Ju Shen ·
Gong-Yih Shiue · Yih-Weng Wang · Chi-Min Shu**

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Abstract Volatile organic compounds (VOCs) are the main factors involved in pollution control and global warming in industrialized nations. Various treatment methods involving incineration, adsorption, etc., were employed to reduce VOCs concentration. Various absorbents, such as activated carbon, zeolite, silica gel or alumina, and so on were broadly used to adsorb VOCs in various industrial applications. Differential scanning calorimetry (DSC) was handled to analyze the thermal characteristics of absorbents. Typically, a scanning electron microscope (SEM) has been used to evaluate the structure variation of absorbents under high temperature situations. In view of pollution control and loss prevention, versatility and analysis of recycled adsorbents are necessary and useful for various industrial applications.

Keywords Volatile organic compounds (VOCs) · Adsorption · Zeolite · Differential scanning calorimetry (DSC) · Thermal characteristics · Scanning electron microscope (SEM)

Introduction

Volatile organic compounds (VOCs) are one of the major groups of atmospheric pollutants. It has been well known that VOCs have adverse effects on human health, cause odorous nuisances, and participate in photochemical reactions. Surveys of VOCs have shown that vehicle exhausts are the dominant sources of emissions, especially in urban areas, accounting for one-half to two-thirds of total emitted VOCs. The purification of industrial waste gases containing VOCs plays an important role in chemical engineering [1–4].

Activated carbon, zeolite, silica gel or alumina, and so on have been broadly used to adsorb VOCs in various industrial and household applications. Practically speaking, the ideal adsorbent characteristics for VOCs removal process are as follows:

(a) a large amount of reversible adsorption capacity (vast accessible pore volume);
(b) no catalytic activity;
(c) a hydrophobic property;
(d) high thermal and hydrothermal stability;
(e) an easy and flexible regeneration property.

C.-H. Su
Department of Fire Science, WuFeng Institute of Technology,
117, Chian-Kuo Rd., Sec. 2, Min-Hsiung, Chia-Yi 62153,
Taiwan ROC

S.-H. Wu · S.-J. Shen · C.-M. Shu (✉)
Doctoral Program, Graduate School of Engineering Science and
Technology, National Yunlin University of Science and
Technology, 123, University Rd., Sec. 3, Douliou, Yunlin 64002,
Taiwan ROC
e-mail: shucm@yuntech.edu.tw

S.-J. Shen
Department of Occupational Safety and Health, ChiaNan
University of Pharmacy and Science, 60, Erh-Jen Rd., Sec. 1,
Jen-Te, Tainan 71710, Taiwan ROC

G.-Y. Shiue
Department of Chemical Engineering and Biotechnology,
Hsiuping of Technology, 11, Gongye Rd., Dali, Taichung 41280,
Taiwan ROC

Y.-W. Wang
Department of Occupational Safety and Health, Jen-Teh Junior
College of Medicine, Nursing and Management, 1, Jen-Teh Rd.,
Houlong, Miaoli 35601, Taiwan ROC

Zeolite has been widely applied in various industrial fields and it has generally been used in the form of beads or pellets. Many studies have applied various types of zeolite to analyze the adsorption effect for VOCs chemicals. Many investigations have been employed to analyze the adsorption capability with various formats of zeolite [5–9]. This study was used to appraise the thermal characteristic of self-made Y-type zeolite in the chemical industries. Figure 1 demonstrates the self-made Y-type zeolite manufacturing flowchart in Taiwan. VOCs were assimilated in the zeolite rotor-wheel system. The zeolite rotor-wheel system was composed of three parts that include the adsorption area, desorption zone, and the recuperative thermal oxidizer (RTO).

Figure 2 shows the cross-sectional area of the zeolite rotor-wheel. The zeolite rotor-wheel was divided into two zones that involve the adsorption area and desorption zone. The zeolite rotor-wheel system is continuous equipment for VOCs handling. Here, the green zone is an adsorption area for process gases. The red zone is a desorption area that is penetrated for hot air under the boiling point of wastes. The thermal stability of the zeolite is an important factor under the best VOCs handling.

First step, zeolite and activated carbons were used to test the stable of its structure processing 200 to 600 °C by chamber furnace. Second, differential scanning calorimetry (DSC) was employed to evaluate and to assess the thermal stability of zeolite and activated carbons (after first step). Results showed that the zeolite is a quite stable material in the VOCs adsorption, compared with other adsorbents, such as activated carbon.

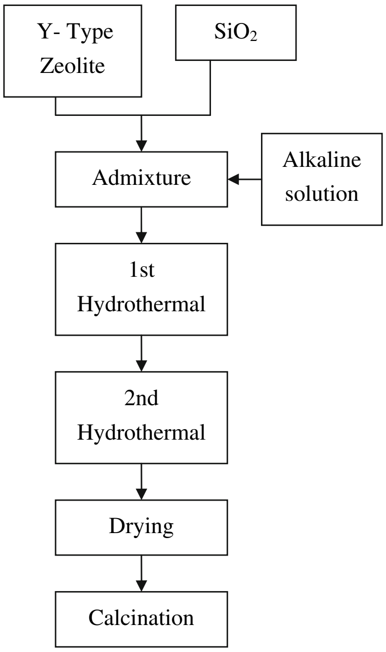


Fig. 1 The self-made Y-type zeolite manufacturing flowchart from our laboratory

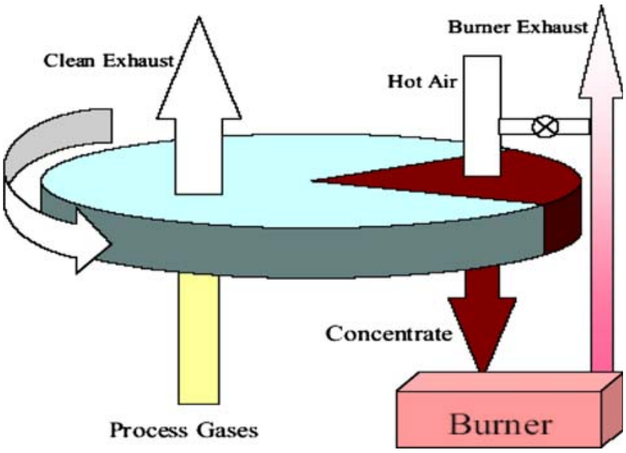


Fig. 2 The cross-section of self-made Y-type zeolite rotor-wheel

Experimental design and methods

This study attempted to evaluate the thermal stability of self-made Y-type zeolite and activated carbon. The experimental processes involved sample preparation, structure tests under previously isothermally treated (200 to 600 °C) by chamber furnace, and the DSC thermal scanning tests for 30 to 640 °C.

Samples

Zeolite was made in five steps: mixing, gelling, forming, drying, and calcination, in the manufacturing process. First, we used SiO_2 mixed with sodium silicoaluminate powder (Si/Al mixed mass ratio was 30) and sodium hydroxide (NaOH) solution in the well stirred machine. Secondly, zeolites were put into the extruding forming machine. Finally, zeolite was dried at 100 °C for 8 h and calcined at 450 °C in an oven (10 h). To develop sample stability, zeolite was employed three times to determine stable analysis. Figure 3 delineates the sample repeated tests of zeolite under heating rate (β) at 4 °C min^{-1} by DSC. Initial reaction behavior of Y-type zeolite was an endothermic reaction at 30 to 200 °C. The final reaction behavior has an exothermic reaction at 550 to 640 °C. Heat of endothermic reaction was calculated as displayed in Table 1. Sample reconstruction tests of zeolite under β at 4 °C min^{-1} by the DSC were calculated at about 150–200 J g^{-1}. An adsorbent such as activated carbon and zeolite adsorbs a few wastes in the air. To adsorb a large amount of wastes, the adsorbent must be desorbed at high temperature.

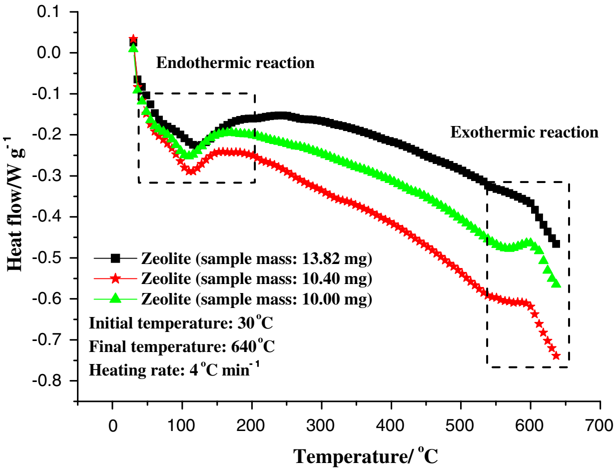


Fig. 3 Sample reproducible tests of Y-type zeolite under β at 4 °C min^{-1} by DSC

Table 1 Heat of endothermic reaction for three tests of self-made Y-type zeolite under β at 4 °C min^{-1} by DSC

Test no.	Sample mass/mg	ΔH/J g^{-1}
1	13.82	209
2	10.40	190
3	10.00	148

Differential Scanning Calorimetry (DSC)

Scanning experiments were performed on a Mettler TA8000 System coupled with a DSC821^{e} measuring cell that can withstand pressure up to about 100 bars. STAR^{e} software was applied for acquiring curve traces. An aluminum standard pan was employed to avoid evaporation of the zeolites and activated carbons during the scanning experiment. For better accuracy in testing, the scanning rates chosen for the temperature range were from 30–640 °C with 4 °C min^{-1} under atmospheric air [10–14]. The experimental conditions were set as follows:

(a) Heating rates: 4 °C min^{-1}.
(b) Materials mass: 1–20 mg.
(c) Test cell: An aluminum standard pan was used to avoid evaporation of the Y-type zeolites and activated carbons during scanning experiments.

Results and discussion

Analysis of zeolite

This study was used to analyze the thermal capacity of zeolite and activated carbon for the chemical industries. Figure 4 shows the thermal capacity of zeolite under previously isothermally treated (previously isothermally treated temperature at 200–600 °C by chamber furnace) by DSC. Zeolite has two reaction behaviors. One is an endothermic reaction at low temperature; the other is an exothermic reaction under high temperature. The operating index, such as desorption temperature, adsorption time, adsorption surface area, etc., was used to adsorb the VOCs and waste as important parameters in the chemical industries. The best adsorbent was tested as disclosed in Fig. 4. Here, heat of endothermic reaction for Y-type zeolite was determined as given in Table 2. Heat of endothermic reaction for zeolite that was tested under room temperature (holding time was 10 h) was about 209 J g^{-1}, indicating a relatively unstable material.

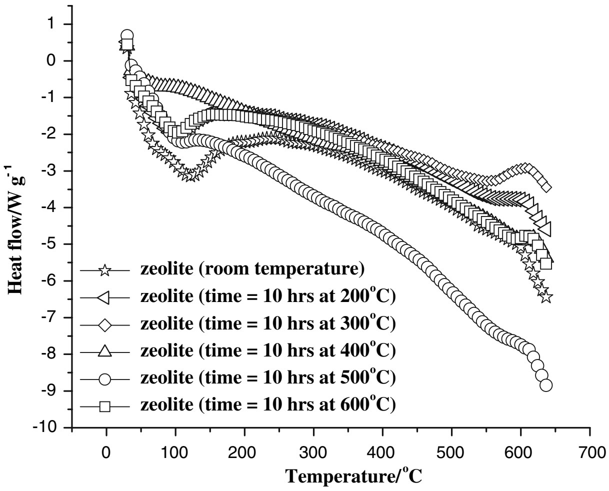


Fig. 4 The best adsorption temperature test of Y-type zeolite under various previously isothermally treated (200–600 °C) by DSC

Table 2 Heat of endothermic reaction for Y-type zeolite under various previously isothermally treated (200–600 °C) (holding time was 10 h) by DSC with 4 °C min^{-1} of heating rate

Under previously isothermally treated temperature/°C	Sample mass/mg	Holding time/h	ΔH/J g^{-1}
Room temperature	13.82	10	209
200	11.00	10	128
300	9.50	10	146
400	8.80	10	21
500	10.00	10	104
600	14.30	10	99

Analysis of activated carbon

In general, activated carbon is a traditional adsorbent that is widely used to adsorb the wastes in water or air, but it has a flame problem in the chemical industries. Activated carbon is cheaper than zeolite. To prevent a fire accident, this

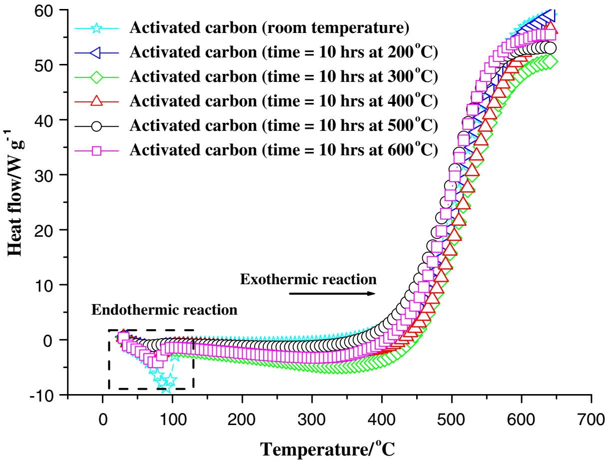


Fig. 5 The endothermic behavior of activated carbon under various previously isothermally treated (200–600 °C) by DSC

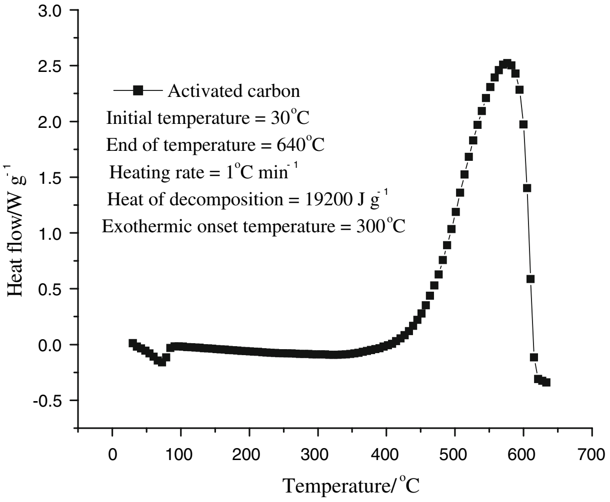


Fig. 6 The peak and curve of activated carbon under heating rate at 1 °C min^{-1} by DSC

study was employed to discover the exothermic behavior under previously isothermally treated temperature (200–600 °C) by DSC. Figure 5 shows the exothermic onset temperature of activated carbon under previously isothermally treated (200 to 600 °C) by DSC. The endothermic behavior of activated carbon was determined by DSC as illustrated in Fig. 5.

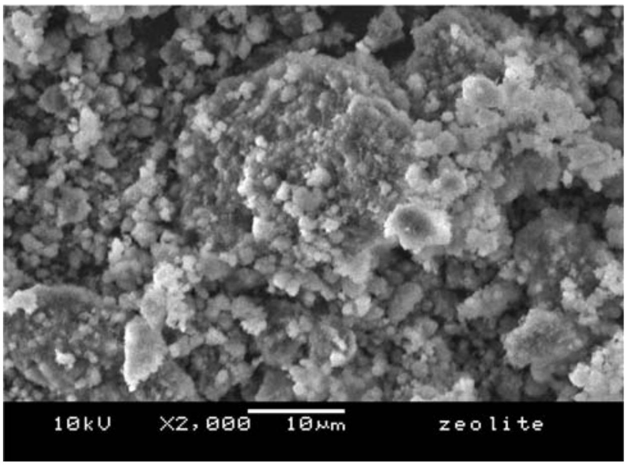
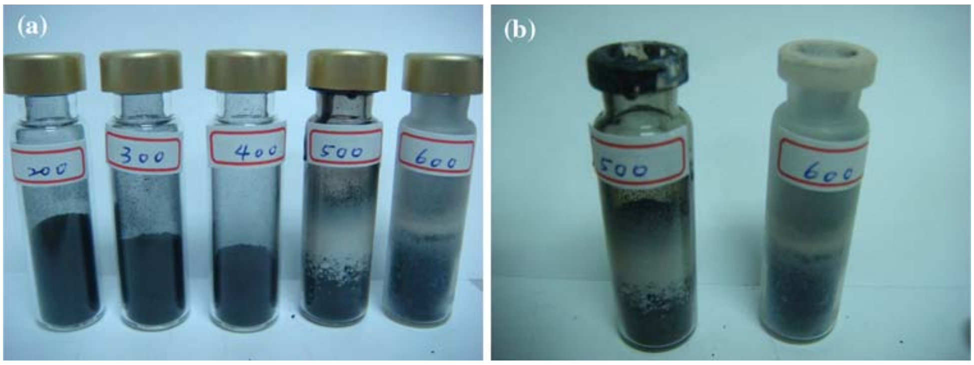


Fig. 8 The zeolite structure using amplification facture at 2000 by SEM

The results show the ideal adsorption temperature at 500 °C that has a low endothermic profile. Figure 5 shows unfinished reactions of activated carbon under heating rate at 4 °C min^{-1} by DSC. Figure 6 reveals the peak of activated carbon under heating rate at 1 °C min^{-1} by DSC. The exothermic onset temperature (T_0) of activated carbon was produced at 300 °C by DSC (β is 1 °C min^{-1}). Thus, heat of decomposition (ΔH_d) from 300–640 °C in activated carbon decomposition behaviors was integrated at about 19,200 J g^{-1} by DSC. Figures 7a and b show the thermal decomposition situation of activated carbon under previously isothermally treated (200 to 600 °C) by chamber furnace. Activated carbon was flame under 500 to 600 °C by chamber furnace (Fig. 7b). Figures 7a and b were applied to display the flame hazard under various temperature. Activated carbon was burst as shown in Figures 7a and b. Figures 8 and 9 display the zeolite structure by scanning electron microscope (SEM). SEM was used to evaluate the structure variation of adsorbents under the high temperature situations. As we know, zeolite structure by SEM scanning was noted by the uniform phase and aperture.

Fig. 7 **a, b** The thermal decomposition situation of activated carbon under various previously isothermally treated by chamber furnace



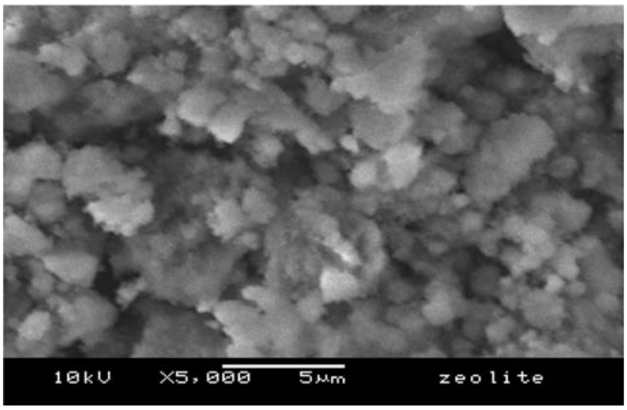


Fig. 9 The zeolite structure using amplification facture at 5000 by SEM

Conclusions

Zeolite is a quite stable substance from room temperature to 650 °C. Activated carbon decomposition behavior was integrated about 19,200 J g^{-1}. Thus, activated carbon is a dangerous adsorbent that shows flame characteristic over 500 °C. Essentially, the best desorption temperature of zeolite is 500 °C.

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