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Postprint: Desorption Performance of Naphthalene from Typical Mesoporous Carbons

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Abstract

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Full Text

Study of Naphthalene Desorption Properties on Typical Mesoporous Carbons

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Abstract: This work investigates the desorption kinetics of naphthalene, a typical polycyclic aromatic hydrocarbon (PAH), on three typical mesoporous carbons (CMK-3, CMK-5, and FDU-15) and a conventional coconut-shell activated carbon (AC) through temperature-programmed desorption (TPD) thermogravimetric experiments. A combined thermogravimetric analysis method employing Kissinger equation estimation, Coats-Redfern equation calculation, and masterplot method verification was adopted to accurately determine the kinetic triplets (desorption activation energy E , pre-exponential factor $\ln A$, and reaction mechanism function) for naphthalene desorption on the four adsorbents. The results demonstrate that CMK-3, CMK-5, and FDU-15 exhibit significantly better desorption/regeneration performance than coconut-shell AC, with the desorption peak temperatures following the order: CMK-5 < CMK-3 < FDU-15 < coconut-shell AC. Naphthalene desorption on coconut-shell AC ($E = 101.5$ kJ/mol) encounters the greatest internal diffusion resistance, followed by CMK-3 ($E = 67.0$ kJ/mol); both are diffusion-controlled. Desorption on CMK-5 ($E = 58.3$ kJ/mol) and FDU-15 ($E = 46.5$ kJ/mol) is reaction-controlled. Although CMK-5 has a higher E than FDU-15 due to its greater microporosity, its more orderly distributed multi-type pore channels make it the optimal adsorbent for overall naphthalene desorption performance.

Keywords: Polycyclic aromatic hydrocarbons; Mesoporous adsorbents; Air

pollution; Desorption kinetics; Thermogravimetric analysis

Introduction

Polycyclic aromatic hydrocarbons (PAHs) are a class of persistent organic pollutants widely present in the environment and represent the earliest identified and most numerous group of carcinogens. With rapid development of human production and living activities, PAH emissions have increased annually, making the control and purification of PAHs in industrial exhaust gases critically important. Adsorption is considered one of the most promising PAH control technologies due to its high removal efficiency, low energy consumption, and simple operation, with adsorbent performance playing a key role.

Traditional adsorbents such as activated carbon exhibit large adsorption capacities due to their abundant micropore volume. However, they suffer from practical issues including slow mass transfer rates, low adsorption rates, and difficult regeneration when adsorbing large molecules like PAHs. The emergence of mesoporous adsorbents provides an effective solution to these problems, among which mesoporous carbon materials offer particular advantages for non-polar molecule adsorption due to their strong surface hydrophobicity and thermal stability. Ryoo et al. synthesized CMK-3 and CMK-5 mesoporous carbons using SBA-15 as a template through reverse replication, both materials well retaining ordered mesoporous structures. Zhao et al. synthesized the FDU series of mesoporous carbon materials through interfacial self-assembly technology, with FDU-15 being representative for its hexagonal structure similar to SBA-15 and excellent stability. All three mesoporous carbon materials possess coexisting mesoporous and microporous structures. Studies have shown that for adsorption of the bicyclic PAH naphthalene, all three exhibit superior performance compared to activated carbon, yet analysis of their desorption/regeneration properties has rarely been reported.

The ease of desorption and its kinetic characteristics are critical criteria for adsorbent selection and constitute an important aspect of PAH adsorption purification research. Temperature-programmed desorption (TPD) is a mainstream thermal analysis technique. In thermal analysis, the combination of multiple heating rate methods and mechanism function fitting to obtain kinetic triplets is currently the most recommended approach, widely applied in thermal decomposition fields, though reports on using thermal analysis to study desorption mechanisms of adsorbates on adsorbents are scarce.

This study optimizes the desorption thermal analysis method by incorporating the masterplot method, using typical PAH naphthalene as a representative to obtain desorption kinetic triplets (activation energy, pre-exponential factor, and mechanism function) on mesoporous carbons (CMK-3, CMK-5, and FDU-15) and coconut-shell activated carbon. The analysis compares the reasons for desorption difficulty and desorption behavior mechanisms on each adsorbent,

providing a comprehensive investigation of desorption kinetics to offer scientific reference for the design and application of high-performance PAH mesoporous adsorbents.

1.1 Adsorbent Characterization

The mesoporous carbons CMK-3, CMK-5, and FDU-15 were purchased from Zibo Huatong Chemical Reagent Co., Ltd., and coconut-shell AC was obtained from Jiangsu Nantong Activated Carbon Co., Ltd. Small-angle X-ray powder diffraction (XRD) patterns of the three mesoporous carbons were characterized using a Dmax-2500 X-ray diffractometer (Rigaku, USA) with Cu K radiation source, tube voltage of 40 kV, tube current of 200 mA, diffraction angle scanning range of $0.5\text{--}10^\circ$, and scanning speed of $0.5^\circ/\text{min}$. An Autosorb-1 physical adsorption instrument (Quantachrome, USA) was used to test the Ar adsorption-desorption isotherms (87 K) of the four adsorbents, yielding pore structure parameters including pore size distribution, pore volume, and specific surface area. Total pore volume was calculated from the adsorption amount at relative pressure $P/P = 0.99$, pore size distribution was calculated using the NLDFT method, and specific surface area was determined from the desorption curve via the BET equation.

1.2 Desorption Sample Preparation

The adsorbate naphthalene (99.7%, GC) was purchased from Sigma-Aldrich. A quantity of 0.20 ± 0.01 g naphthalene was added to 10 ± 0.1 g methanol solution, followed by addition of 0.12 ± 0.01 g adsorbent. The mixture was placed in a 298 K constant-temperature water bath for 12 h, then filtered. The sample and filter paper were together placed in a 333 K drying oven for 2 h to obtain the desorption samples for each adsorbent, which were sealed after drying. Additionally, samples with only methanol attached and blank samples were prepared for each adsorbent for blank experiments.

1.3 TPD Experimental Conditions

A Q50 thermogravimetric analyzer (TA Instruments, USA) was used to record weight changes (thermogravimetric, TG, curves) of samples during TPD experiments at different heating rates. Four samples of each adsorbent (20 ± 1 mg each) were placed in crucibles inside the thermogravimetric chamber. High-purity N₂ at 50 mL/min was used as carrier gas, and TPD experiments were conducted at three heating rates: 8 K/min, 16 K/min, and 20 K/min. The initial equilibrium temperature was 293 K, held for 5 min before heating began and weight loss curves were recorded. Heating continued until sample mass no longer changed significantly, at which point data recording stopped. After maintaining for 30 min, heating was terminated. TG curves at different heating

rates were converted to derivative thermogravimetric (DTG) curves. Additionally, two sets of repeatability experiments were conducted for each sample under identical conditions, with TG curves showing good reproducibility. Blank experiments verified that the small amount of residual methanol on adsorbents had negligible effect on desorption.

2.1 Calculation of Desorption Kinetic Triplets

Based on measured weight loss during desorption, TG curves can be obtained. The desorption conversion rate, α , is calculated by:

$$\alpha = \frac{m_0 - m_t}{m_0 - m_f}$$

where m_0 is the initial sample weight, m_t is the sample weight at time t , and m_f is the final sample weight after desorption completion. The desorption rate under non-isothermal conditions can be expressed by the general equation:

$$\frac{d\alpha}{dT} = \frac{A}{\beta} \exp\left(-\frac{E_a}{RT}\right) f(\alpha)$$

where β is the heating rate, T is absolute temperature, R is the gas constant, E_a is desorption activation energy, A is the Arrhenius pre-exponential factor, and $f(\alpha)$ is the differential form of the reaction mechanism function—these three parameters constitute the kinetic triplet.

Before mechanism function fitting analysis, E_a and A must be estimated. The Kissinger equation is one of the most common and convenient methods:

$$\ln\left(\frac{\beta}{T_{\max}^2}\right) = \ln\left(\frac{AR}{E_a}\right) - \frac{E_a}{RT_{\max}}$$

where T_{\max} is the temperature corresponding to the DTG curve peak. Plotting $\ln(\beta/T_{\max}^2)$ versus $1/T_{\max}$ yields the estimated activation energy E_a , from the slope and pre-exponential factor A from the intercept.

To further screen appropriate mechanism functions, the Coats-Redfern (C-R) equation was used to fit several common mechanism functions (Table 1) based on TPD experimental data:

$$\ln\left[\frac{g_i(\alpha)}{T^2}\right] = \ln\left(\frac{AR}{\beta E_{a,i}}\right) - \frac{E_{a,i}}{RT}$$

where “i” represents a specific reaction mechanism function and $g(\alpha)$ is the integral form of the mechanism function. Based on Equation (4), each mechanism function was used to fit TPD experimental data individually. Plotting $\ln[g(\alpha)/T^2]$ versus $1/T$ yields E , and $\ln A$ from the slope and intercept, respectively. The mechanism function with the highest linear fitting degree (maximum correlation coefficient R^2) and whose E , and $\ln A$ values are close to the Kissinger estimates was selected as the most probable mechanism function, yielding the final kinetic triplet.

2.2 Masterplot Method Verification

Gotor et al. proposed the masterplot method to determine kinetic models for isothermal and non-isothermal solid-state decomposition reactions. This method introduces the concept of generalized time τ , using conversion rate $\alpha = 0.5$ as a reference point to establish masterplot criteria based on generalized kinetic equations. By reconstructing kinetic curves to determine the kinetic model function, this method can verify the accuracy of the most probable mechanism function for desorption processes.

The masterplot represents the relationship $g(\alpha)/g(0.5) \sim \tau$. Comparing experimental masterplots derived from TPD data with standard masterplots based on kinetic mechanism functions allows direct judgment of the appropriate mechanism function. Standard masterplots can be obtained by substituting the integral forms of various mechanism functions (Table 1) into the $g(\alpha)/g(0.5) \sim \tau$ relationship. Experimental masterplots can be derived through transformation of the $g(\alpha)$ expression:

$$\frac{p(x)}{p(x_{0.5})} = \frac{g(\alpha)}{g(0.5)}$$

where E is the desorption activation energy obtained from the Kissinger equation, and $p(x)$ is the Arrhenius temperature integral function with $x = E/RT$, which can be obtained from the Tang Wanjun approximation:

$$p(x) = \frac{\exp(-x)}{x} \cdot \frac{x^3 + 18x^2 + 88x + 96}{x^4 + 20x^3 + 120x^2 + 240x + 120}$$

From Equation (5), based on the reference point $\alpha = 0.5$, the following relationship can be obtained:

$$\frac{p(x)}{p(x_{0.5})} = \frac{g(\alpha)}{g(0.5)}$$

where $x = E/RT$, and $T_{0.5}$ is the temperature corresponding to 50% desorption conversion. Experimental masterplots are obtained by calculating x values at different conversion rates using E and T , then plotting $p(x)/p(x_{0.5})$ versus τ .

3.1 Adsorbent Characterization Results

The small-angle XRD characterization results for the three mesoporous carbons are shown in Figure 1 [Figure 1: see original paper]. CMK-3, CMK-5, and FDU-15 all exhibit significant diffraction peaks at $2\theta = 0.5\text{--}2.5^\circ$, demonstrating long-range ordered two-dimensional hexagonal structures (p6mm). The inverse-replicated structure CMK-3, based on SBA-15 as a hard template, shows similar peak patterns to FDU-15, indirectly reflecting the structural similarity between FDU-15 and SBA-15, though the former has smaller interplanar spacing (larger diffraction angles) than the latter.

The pore size distributions and corresponding Ar adsorption-desorption isotherms of the three mesoporous carbons and coconut-shell AC are shown in Figure 2 [Figure 2: see original paper], with the calculated BET specific surface area (S_{BET}), micropore volume ($V_{\text{micropore}}$), mesopore volume (V_{mesopore}), and most probable pore diameter (d_{p}) summarized in Table 2. The isotherms of the three mesoporous carbons all show Type IV characteristics with hysteresis loops at $P/P^* = 0.4\text{--}0.8$, indicating ordered mesoporous structures. CMK-3 and CMK-5 have relatively narrow mesopore distributions, while FDU-15 shows a broader mesopore distribution; all three exhibit micropore distributions. Coconut-shell AC primarily displays irregular micropore distributions.

CMK-5 possesses significant ultramicropore (<0.7 nm), micropore (0.7–2 nm), and mesopore ($d = 3.32$ nm) distributions, with the highest S_{BET} (1522 m²/g). CMK-3 has larger mesopore distribution ($d = 4.88$ nm) and minor ultramicropore distribution ($V_{\text{micropore}} = 0.03$ cm³/g), with the second-highest S_{BET} (1440 m²/g). FDU-15's main mesopore diameter (~ 3.64 nm) is close to that of CMK-5, but its V_{mesopore} and S_{BET} (1026 m²/g) are lower than the former two. All three mesoporous carbons have higher S_{BET} values than coconut-shell AC.

The difference between CMK-3 and CMK-5 obtained from the SBA-15 hard template is that CMK-3 forms a spatial carbon rod array, while CMK-5 forms a spatial carbon tube array, with the interior space of the latter's carbon tubes composing a significantly uniform micropore distribution. FDU-15 exhibits a hexagonal channel arrangement similar to SBA-15 materials, with numerous coarse and fine mesopores and micropores of various sizes formed during the carbonization process, resulting in a broader pore size distribution. The coexistence of fine mesopores and certain micropore distributions alongside ordered main mesopores in the three mesoporous carbons contributes substantially to surface area, all higher than the unordered micropore-dominated coconut-shell AC.

3.2 TPD Experimental Curve Analysis

The TG and DTG curves of naphthalene on the four adsorbents are shown in Figures 3 and 4 [Figure 3: see original paper][Figure 4: see original paper], respectively. The consistent total weight loss across different heating rates indicates essentially complete desorption within the corresponding temperature range. From TG curves, the adsorption capacity (desorption amount) of naphthalene on the four adsorbents follows the order: CMK-5 > CMK-3 > FDU-15 > coconut-shell AC, consistent with the S_{mes} values.

DTG curves show that T_{max} (the temperature corresponding to the desorption rate peak) increases with heating rate. The T_{max} values of all three mesoporous carbon samples are lower than those of conventional coconut-shell AC, indicating better regeneration performance of mesoporous carbon materials for naphthalene. The adsorbents follow the order: CMK-5 < CMK-3 < FDU-15 < coconut-shell AC. CMK-5 shows significantly lower desorption temperatures than other adsorbents with narrower peak shapes, achieving complete desorption at 450 K, reflecting easier and faster desorption characteristics. CMK-3 and FDU-15 have similar desorption temperatures, but CMK-3 shows more asymmetric DTG peak shapes with obvious tailing, with possible desorption still occurring above 600 K. For the traditional adsorbent coconut-shell AC, T_{max} reaches as high as 520 K, with severe DTG curve tailing requiring temperatures up to 800 K for complete desorption.

These macroscopic desorption characteristics, judged from desorption temperatures and DTG peak shapes, reflect intrinsic desorption behaviors and mechanisms related to kinetic characteristics. Therefore, further analysis requires evaluation of desorption kinetic triplets.

3.3 Calculation of Desorption Kinetic Triplets

Based on TPD experimental results, characteristic values from DTG curves were extracted to estimate E and $\ln A$ for each sample using the Kissinger equation (3). The results show that for naphthalene desorption on CMK-3, CMK-5, FDU-15, and coconut-shell AC, the E (kJ/mol) and $\ln A$ (min^{-1}) values are 63.7 and 14.9, 57.2 and 13.0, 47.1 and 8.4, and 99.7 and 18.2, respectively, with linear fitting R^2 values all exceeding 0.99. Further, based on the C-R equation (4), mechanism functions listed in Table 1 were used to fit each TPD dataset. According to fitting results, several mechanism functions with high fitting degrees were selected for desorption on the four adsorbents: A1, A2/3, A1/3, A1/4, and F2. Through further comparison with Kissinger-estimated E and $\ln A$ values, the desorption mechanism functions for naphthalene on CMK-3, CMK-5, FDU-15, and coconut-shell AC were determined as A1/3, A2/3, F2, and A1/4, respectively.

Figure 5 [Figure 5: see original paper] compares standard and experimental masterplot values for naphthalene desorption on the four adsorbents, with standard values based on A1, A2/3, A1/3, A1/4, and F2 masterplots. The compari-

son reveals that experimental masterplots for naphthalene on CMK-3, CMK-5, FDU-15, and coconut-shell AC closely match standard masterplots based on A1/3, A2/3, F2, and A1/4, respectively—completely consistent with C-R equation results and confirming their reliability. The desorption kinetic triplets for naphthalene on the four adsorbents are summarized in Table 3, where E and $\ln A$ are averages across three heating rates obtained by the C-R method based on respective mechanism functions.

3.4 Desorption Mechanism Analysis

During naphthalene desorption from each adsorbent, naphthalene molecules diffuse from surfaces to the bulk gas phase, exhibiting different desorption kinetic mechanisms depending on pore structure. Figure 6 [Figure 6: see original paper] shows schematic diagrams of pore structures for the three mesoporous materials. Table 3 reveals that except for FDU-15, desorption kinetics on all other adsorbents follow the Johnson-Mehl-Avrami (JMA) rate equation based on nucleation-growth models, $g(t) = [-\ln(1 - \dots)]$. This equation commonly describes reaction kinetics in phase transformation and decomposition processes, where exponent n is closely related to reaction type: $n < 0.5$ indicates diffusion-controlled reactions; $0.5 < n < 1$ indicates interface reaction-controlled processes; $n > 1$ indicates nucleation and growth-controlled processes.

Naphthalene desorption kinetics on coconut-shell AC and CMK-3 both exhibit JMA diffusion-controlled mechanisms. Given their large E values (101.5 kJ/mol and 67.0 kJ/mol), the disordered ultramicropores and micropores within coconut-shell AC and CMK-3 carbon rod walls create substantial internal diffusion resistance for naphthalene (molecular kinetic diameter = 0.66 nm), making diffusion the rate-controlling step. The smaller n value (A1/4) for coconut-shell AC indicates greater diffusion resistance.

Naphthalene desorption on CMK-5 follows JMA interface reaction-controlled kinetics. The carbon tube array structure of CMK-5 (Figure 6) provides interconnected diffusion spaces both inside and outside carbon tubes, resulting in low desorption diffusion resistance. The rate-controlling step is primarily interfacial interaction between naphthalene and carbon surfaces. Meanwhile, CMK-5's multi-type pore distribution (ultramicropores, micropores, mesopores) provides more optimal matching sites for naphthalene adsorption-desorption processes, with uniform pore size distributions further promoting desorption diffusion, making its overall desorption performance optimal (Figure 4).

The mechanism function F2 for naphthalene desorption on FDU-15 represents a second-order reaction-controlled mechanism, suggesting the desorption process tends toward a thermal decomposition reaction. In fact, Figure 5 shows FDU-15's experimental masterplot is also close to the A2/3 standard masterplot, indicating similarity to CMK-5's interface reaction-controlled mechanism. However, FDU-15 likely has weaker overall binding to naphthalene, with a slightly different rate-controlling form following a reaction-order model. This is primarily

related to FDU-15's special pore structure. The pore size distribution (Figure 2) shows FDU-15 has a broad distribution, with pore structure mainly consisting of primary mesopores ($d = 3.64$ nm) accompanied by coarse and fine mesopores and micropores. These primary mesopore walls are either open or closed, forming open cross-linked channels that result in low naphthalene desorption diffusion resistance. Compared to CMK-5, FDU-15's richer mesopore configurations and lower micropore distribution provide more suitable mesoporous adsorption sites for naphthalene, with weaker overall binding and consequently lower E (46.5 kJ/mol). However, due to far inferior pore channel ordering compared to CMK-5, macroscopic naphthalene desorption from FDU-15 remains relatively difficult, manifested by higher desorption temperatures. Comprehensive analysis reveals that the existence form of micropores and fine mesopores, along with the ordering of matching mesopores, are key factors determining superior naphthalene desorption performance.

4 Conclusions

1. The three mesoporous carbons CMK-3, CMK-5, and FDU-15 exhibit significantly better naphthalene desorption/regeneration performance than traditional coconut-shell AC, with T following the order: CMK-5 < CMK-3 < FDU-15 < coconut-shell AC.
2. A combined thermal analysis approach using Kissinger equation estimation, C-R equation calculation, and masterplot method verification accurately obtained naphthalene desorption kinetic triplets on the four adsorbents.
3. Naphthalene desorption on coconut-shell AC ($E = 101.5$ kJ/mol) encounters the greatest internal diffusion resistance, followed by CMK-3 ($E = 67.0$ kJ/mol); both are diffusion-controlled.
4. Both CMK-5 ($E = 58.3$ kJ/mol) and FDU-15 ($E = 46.5$ kJ/mol) are reaction-controlled. Although CMK-5 has higher E than FDU-15 due to greater microporosity, its more orderly distributed multi-type pore channels make it the best adsorbent for overall naphthalene desorption performance.

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