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Loading redistribution of NOM competitors?

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Mitigating NOM competition against micropollutant adsorption through staged dosing of activated carbon: Loading redistribution of NOM competitors?

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A R T I C L E   I N F O

A B S T R A C T

Powdered activated carbon (PAC) adsorption is widely applied for the removal of organic micropollutants in drinking water treatment. However, conventional single-dose PAC application requires high dosages to overcome competitive adsorption from natural organic matter (NOM). This study evaluates a multi-stage PAC dosing strategy to mitigate NOM competition and enhance the removal efficiency of representative odorous micropollutants. Experiments with NOM-rich surface water showed that multi-stage dosing increased adsorption capacity and achieved up to 48% PAC savings at an 80% micropollutant removal benchmark, compared to single-stage application. In contrast, *no improvement was observed in NOM-free water*. Two-stage PAC dosing considerably improved the removal of weakly adsorbing compounds such as 2-methylisoborneol (MIB) and 2-ethyl-5,5-dimethyl-1,3-dioxane (EDD), while incremental gains were observed for strongly adsorbing micropollutants (e.g., 2-butyl-5,5-dimethyl-1,3-dioxane) with three- or four-stage configurations. Mechanistic analysis indicated that early-stage PAC doses preferentially adsorbed competitive NOM components, preserving high-affinity sites for micropollutants in later stages. PACs enriched in narrow mesopores outperformed microporous PACs in the staged dosing configuration. The proposed strategy, as compared to existing designs summarized from the literature, requires minimal infrastructural modifications and offers a cost-effective, scalable approach for improving micropollutant removal under NOM-rich conditions.

1. Introduction

Odorants are a critical subset of organic micropollutants for drinking water production, as they can trigger widespread public complaints and lead to adverse social and economic consequences [1]. Emerging odorants, including cyclic acetals (e.g., 2-ethyl-5,5-dimethyl-1,3-dioxane) and 2-chloro-1-methylethyl compounds, have been increasingly detected in raw and treated water across various global water supply systems [2]. These compounds pose challenges due to their extremely low odor thresholds—often below 10 ng/L—and resistance to conventional treatment processes [3]. Their presence not only impairs the sensory acceptability of drinking water but may also raise toxicological

concerns, highlighting the urgent need for effective removal strategies. For odorants that are also resistant to oxidative processes (e.g., cyclic acetals and terpenes among odorants), powdered activated carbon (PAC) adsorption remains essential in treating source water to drinking water, effectively removing a wide range of odorous compounds [4,5]. However, the high consumption of PAC adsorbents is primarily due to competitive adsorption by high concentrations of natural organic matter (NOM, in mg/L) against trace-levels of odorants (in ng/L) as the target adsorbates [6,7]. PAC demand for mitigating odorants in NOM-rich source water exceeds that of NOM-free water by approximately tenfold [8,9], as most of the effective adsorption sites are occupied by NOM competitors instead of the target adsorbates.

The adsorption of odorants and background NOM onto activated carbon primarily involves physicochemical effects/interactions such as size exclusion, van der Waals forces, hydrophobic interactions, electrostatic interactions, and π–π stacking. The molecular weight of NOM constituents exhibits a wide range (10^2 —10^6 Da). Low molecular weight (LMW) NOM components can penetrate micropores and compete with similarly sized odorants via direct site competition, particularly through hydrophobic interactions [10]. In contrast, high molecular weight NOM tends to adsorb on the external surface or mesopores of PAC, potentially blocking access to internal adsorption sites and prolonging diffusion pathways [11]. Additionally, aromatic-rich NOM can engage in π–π interactions, further interfering with odorant uptake [12]. Therefore, variations in the composition of NOM constituents—such as differences in molecular size, hydrophobicity, and aromaticity—as well as changes in NOM concentration, considerably modulate the overall performance of odorant adsorption during water treatment processes. In this context, a clear understanding of the distinct adsorption and desorption behaviors of NOM and odorants is essential for devising effective strategies to mitigate NOM competition and improve odorant removal efficiency.

While physical adsorption of organic micropollutants, including odorants, is often cited as the primary mechanism for activated carbon adsorption, and associated with (complete) reversibility, NOM compounds usually exhibit high irreversibility [13,14]. Various forces, such as hydrogen bonding, electrostatic interactions, and π–π interactions, contribute to NOM adsorption on activated carbon, complicating desorption from activated carbon surfaces in aqueous solutions or polar solvents [15]. Previous studies explored the impact of NOM on micropollutant adsorption/desorption dynamics of micropollutants including odorants [11,13,16,17]. Their findings indicate a considerably lower desorption of micropollutants in the presence of NOM (~20 % vs. ~ 80 % under single-solute conditions), suggesting that pore blocking by NOM reduces adsorbed trace-level micropollutant to release into aqueous solutions [13].

Considering the distinctive adsorption–desorption characteristics of NOM and targeted micropollutants, a multi-stage adsorption process could be considered as an upgrade of the conventional single-stage PAC dose strategy to improve the removal of the targeted micropollutants [18,19]. Owing to the inadequate desorption of adsorbed NOM, the NOM competitors against, e.g., odorants may selectively accumulate in the early stage of PAC adsorption, thus not persistently impairing the odorant elimination in later stages. By fine-tuning the PAC loadings for odorants and NOM compounds at different phases, the multi-stage adsorption approach could thus effectively alleviate NOM competition, boost the exploitation of PAC adsorption sites, and diminish PAC usage in drinking water treatment practices. Prior studies on multi-stage adsorption processes predominantly focused on biologically refractory organics (evaluated as bulk organics level) from domestic/industrial wastewater treatment [20–22]. The further adaptation of multi-stage adsorption to enhance micropollutant removal is expected to yield operational cost savings in PAC utilization in the context of the increasingly stringent global drinking water quality guidelines, which impose low thresholds for odorants and per-/polyfluoroalkyl substances [23,24].

Therefore, this study was conducted to verify the hypothesis that multi-stage PAC treatment could improve odorant adsorption by alleviating NOM competition. The odorant adsorption efficacy was compared between single-stage adsorption and multi-stage adsorption in both NOM-free water and raw source water containing, highly competitive, NOM. Three PAC samples with varying pore size distribution were included to determine if the enhanced efficacy related to the match between NOM competitor size and PAC pore structure. Further mechanistic studies included NOM desorption tests and adsorptive odorant removal in PAC-pretreated water. The proposed easy-to-implement multi-stage PAC adsorption opens new possibilities for enhanced micropollutant removal in drinking water treatment by simply reshaping the loading pattern between target micropollutant adsorbates and NOM competitors.

2. Materials and methods

2.1. Odorous compounds

Seven odorous micropollutants were chosen for the study, including five cyclic acetals with solvent-like odorants and two terpene substances with musty odor (Sigma Aldrich, USA), as shown in Table 1. These selected odorants vary in hydrophobicity (logD between 1.64 and 3.17), molecular weight and molecular structure, and show substantial differences in adsorbability (i.e., adsorption affinity onto adsorbents). Among them, 2-methylisoborneol (MIB), geosmin, 2-ethyl-5,5-dimethyl-1,3-dioxane (EDD), and 2-butyl-5,5-dimethyl-1,3-dioxane (BMD) are frequently reported as odorants with low odor thresholds ($\leq$10 ng/L) [25]. The selected odorants were mixed and prepared as stock solutions in HPLC-grade methanol.

2.2. Water samples

A NOM-rich surface water sample was collected from a reservoir downstream of Lake Taihu, and its NOM characteristics are summarized in Table S1. Although this source represents a specific regional water body, the NOM composition and its competitive adsorption behavior toward odorants—particularly MIB—are broadly representative of NOM-rich surface waters commonly encountered in drinking water treatment. Notably, previous studies have shown that the NOM from this watershed exhibits stronger competitive effects than Suwannee River humic acids and several other Chinese natural waters [26]. Thus, the evaluation of odorant adsorption in Lake Taihu water should be representative for competitive NOM adsorption scenarios. To enhance water quality stability and remove suspended solids, the sample was filtered using 0.45 μm polyethersulfone filters (PALL, USA). For the "NOM-free" reference water, ultrapure water was adjusted to match the pH and major inorganic ion composition of the Taihu water sample, as detailed in Table S1. This ensured that the effects of NOM on odorant adsorption could be isolated, while maintaining comparable ionic conditions that may influence adsorption behavior and competition.

2.3. Activated carbon

Three commercially available PAC samples were employed in evaluating the performance of multi-stage adsorption. These PAC samples were produced of different raw materials (wood, coal, mixed) and have a varying pore structure (average pore size from 2.17 nm to 4.23 nm). More details are given in Table 2. The PAC samples were washed with ultrapure water to eliminate dissolved impurities, dried overnight at 105℃, and degassed in vacuum prior to preparing PAC suspensions at concentrations of 4 g/L and 20 g/L. Before PAC was dosed into the adsorption batches, homogenization was carried out by stirring magnetically for 0.5 h.

2.4. Single-/multi-stage adsorption

Fig. 1 gives the schemes of multi-stage (left) and single-stage (right) adsorption. The bottle point method (100 mL) was employed to perform adsorption isotherm tests. Minimal headspace was maintained, and bottles were securely sealed with Parafilm to minimize the risk of odorant volatilization. To ensure data reliability, quality assurance procedures included procedural blanks (PAC in NOM-free water, with all odorants remaining below detection limits) and matrix spikes (odorants spiked into NOM-rich water, showing < 5 % loss over 48 h due to effective sealing). Both single-stage and multi-stage adsorption experiments were carried out concurrently. Mixed odorants were initially spiked into both raw source water and NOM-free water supplemented

Table 1

Physicochemical properties of odorants in the current study. Predictions of LogD were made using Chemicalize (access date: 12 December 2024), https://chemicalize.com.
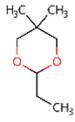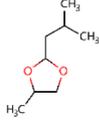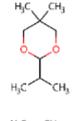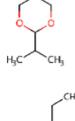
Category	Odorant	Abbreviation	Molecular weight	LogD (pH 7.4)	Structure	CAS No.
Solvent-like odor	2-Ethyl-5,5-dimethyl-1,3-dioxane	EDD	144.21	1.91		756–581
	2-Isobutyl-4-methyl-1,3-dioxolane	IMD	144.21	2.03		18433–93-7
	2-Butyl-5,5-dimethyl-1,3-dioxane	BDD	172.27	2.80		81367–55-7
	2-Isopropyl-5,5-dimethyl-1,3-dioxane	IPDD	158.24	2.39		7651–50-5
	2-Butyl-5,5-methyl-1,3-dioxolane	BMD	144.21	2.19		74094–60-3
Musty odor	2-Methylisoborneol	MIB	168.28	2.27		2371–42-8
	Geosmin	GSM	182.17	3.17		5173–70-6
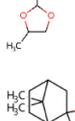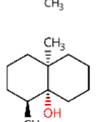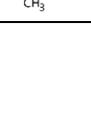
Table 2

Pore structure of PAC adsorbents in this study.

Designation	Raw material	BET surface area (m^2/g)	Total pore volume (cm^3/g)	Micropore volume (cm^3/g)	Mesopore volume (cm^3/g)	Average pore size (nm)
CK1000	Coal	986.0	0.51	0.34	0.17	2.17
SAE Super	Mixed (Bituminous/wood)	1487.0	1.00	0.29	0.71	2.94
WB900	Wood	926.0	0.77	0.23	0.54	4.23
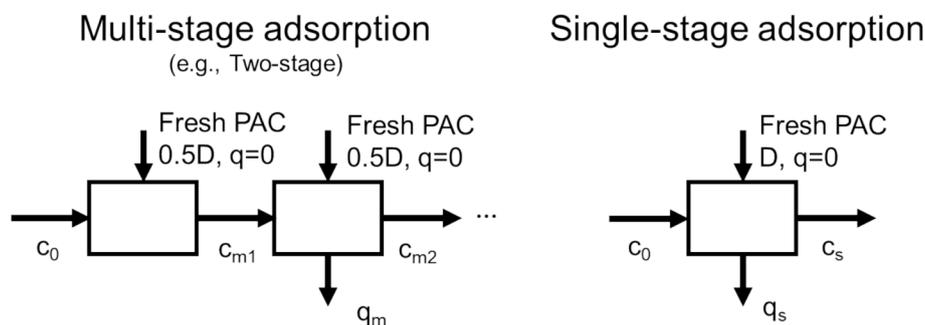
Fig. 1. Scheme for mass and volume fluxes in multi-stage (left) and single-stage (right) adsorption. Multi-stage adsorption involves sequential addition of fresh PAC in stages, with the total PAC dose (D) split evenly (e.g., 0.5D for two stages). In the first stage, the initial concentration (c_0) decreases to (c_{m1}) as adsorption occurs. In the second stage, fresh PAC is introduced, further reducing the concentration to (c_{m2}). Single-stage adsorption, in contrast, adds the entire PAC dose (D) at once, reducing the concentration from c_0 to c_s.

with inorganic salts, with each individual compound added at a target concentration of 500 ng/L. This resulted in a total odorant concentration of approximately 3.5 µg/L, a level at which internal competition among the odorants was considered negligible [10,27]. In the multi-stage adsorption experiment, the total PAC dose was equally divided into several portions (two to four). The total PAC dose ranged from 2 to 80 mg/L, covering the typical range for seasonal odor management, with adsorption conditions set at a temperature of 20 °C and under constant mixing. Given that 48 h is the equilibrium time for single-stage odorant adsorption, the same period was maintained for multi-stage adsorption to ensure a comparable evaluation of the performance [28]. For example, in two-stage adsorption, half of the total PAC was dosed initially, and the remaining half was added after 24 h, without removing the existing PAC adsorbents, followed by another 24 h of mixing/adsorption. Upon completion, all PAC adsorbents were removed by filtration through polyethersulfone syringe filters (0.45 µm, Anpel, China). The odorant concentration and NOM characteristics were then assessed using the filtrate.

The relative contribution of each PAC dosing stage in the two-stage adsorption process was determined by incorporating an intermediate sampling step. In addition to the complete two-stage test, a parallel control group was established in which MIB and NOM concentrations were measured after the first-stage PAC contact (half of total PAC dose, 24 h), but prior to the addition of the second-stage PAC. These intermediate measurements, when compared with final concentrations after the second-stage treatment, enabled calculation of stage-specific adsorption performance.

To assess the adsorption and desorption behavior of NOM under varying loading conditions, batch adsorption–desorption experiments were conducted using a range of PAC doses (5–80 mg/L), with replicate samples included to ensure reproducibility. NOM adsorption was first carried out using raw NOM-rich water over a 48-hour contact period in 50 mL polypropylene centrifuge tubes. Following adsorption, the suspensions were centrifuged at 3000 rpm, and the supernatant was carefully decanted for DOC analysis, leaving behind approximately 2 mL of PAC-containing slurry. Subsequently, 48 mL of NOM-free water was added to each tube for desorption testing. The tubes were incubated at 20 °C for an additional 48 h. After desorption, the supernatant was collected and filtered through 0.45 µm PES membrane filters. The desorption efficiency of NOM was calculated as the ratio of the DOC mass recovered in the desorption supernatant relative to the originally adsorbed DOC mass [16].

To investigate the mechanism of multi-stage adsorption and evaluate whether reduced NOM competition enhances odorant adsorption, we performed control tests in PAC-pretreated water (10 and 20 mg/L, 24 h contact) in the first stage. After removing the PAC by 0.45 µm membrane filtration, the odorant mixture was subsequently spiked into the treated water. Adsorption isotherm and kinetic experiments were then performed to assess the enhancement of MIB adsorption kinetics under reduced NOM competition [29]. The adsorption kinetics data were analyzed using modified pseudo-second-order models [30,31]. Unlike the traditional pseudo-second-order model (Eq. (1)), where the rate constant k_2 (with a unit of mg/(ng·h) here) depends on the concentration in the solid phase, the modified pseudo-second-order model introduces a rate constant k_2^* (with a unit of 1/h) (Eq. (2)). This modification facilitates a more consistent comparison of odorant adsorption kinetic rates in raw or PAC-pretreated source water. In addition, q represents the amount of odorant adsorbed onto PAC at a given time t, while q_e denotes the equilibrium adsorption capacity under the specific experimental conditions (here, raw source water or PAC-pretreated source water). Further information on the modified pseudo-second-order models can be found in the works of Ersan, Kaya, Ersan, Apul and Karanfil [30] and Pan and Xing [31].

$$\frac{t}{q} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \times t \tag{1}$$

$$k_2^* = k_2 \times q_e \tag{2}$$

To comprehensively compare the adsorbabilities for the various odorants in single-/multi-stage adsorption, we calculated the total PAC demand needed for the removal of 80 % of the respective odorants from the aqueous solution, termed PAC-Od-Rem80%. This value was linearly interpolated based on the established on single-/multi-stage adsorption isotherms [10,19]. While advanced modeling approaches such as the ideal adsorbed solution theory-equivalent background compound (IAST-EBC) model could be employed for theoretical estimation, their application requires prior fitting of compound-specific adsorption parameters (e.g., K_F, $1/n_F$) for each odorant–adsorbent pair. Additionally, EBC models assume consistent adsorption behavior across multi-component and staged systems, which may not hold regarding varying odorant–PAC combinations. Therefore, interpolation was adopted here as a more direct and assumption-free method to compare the odorant adsorbabilities across different odorants and PAC adsorbents.

2.5. Visualization of odorant adsorption with operating lines

To visualize the process difference of single-stage adsorption and two-stage adsorption in the figures of adsorption isotherms, operating lines were used to describe the odorant equilibrium adsorption. The adsorption process begins at the initial odorant concentration (c = c_0) and fresh PAC (q = 0) and ends at equilibrium with c = c_e and q = q_e (index "e" refers to equilibrium). The equation for deriving operating lines is given in Eq. (3) [6].

$$q_e = \frac{1}{D_{PAC}} c_0 - \frac{1}{D_{PAC}} c_e \tag{3}$$

The slope of the operating line is $-\frac{1}{D_{PAC}}$, the negative reciprocal value of the adsorbent dose, as derived from adsorption theory [6]. Specifically, the operating lines for second-stage odorant adsorption have the same slope as the first-stage adsorption due to identical doses between two stages, but end at different equilibrium capacities (i.e., different c_e and q_e) from single-stage adsorption isotherm due to the varied NOM competition.

2.6. Analytical methods

All odorant samples were analyzed by headspace solid-phase microextraction (SPME) using an 85-mm Carboxen/PDMS fiber (57334-U, Supelco, USA) coupled to a gas chromatography tandem mass spectrometer (GCMS/MS-TQ8040, Shimadzu, Japan) equipped with a VF-624 MS column (Agilent, USA) (Nie et al., 2023). To ensure analytical reliability, method recovery for the target odorants was determined to be 92 ± 6.2 %, and the limits of detection (LOD) and quantification (LOQ) are summarized in Table S2.

A surface area and porosimetry system using the nitrogen adsorption/desorption test (ASAP 2460, Micromeritics) was used to evaluate the PAC pore structure. Specific surface areas, corresponding to micro/*meso*-pore volumes and pore size distributions, and average pore sizes were calculated using the Brunauer-Emmett-Teller (BET) equation and non-local density functional theory.

For NOM analysis, UV_{254} measurements of water samples were carried out using a UV–Vis spectrophotometer (UV-2600, Shimadzu, Japan). Dissolved organic carbon (DOC) was evaluated using a TOC analyzer (TOC-VCPH, Shimadzu, Japan).

3. Results and discussion

3.1. Performance of two-stage adsorption for odorant removal

The efficacy of the two-stage PAC dose strategy was evaluated through the examination of adsorption isotherms in Lake Taihu water, as

illustrated in Fig. 2. The results indicate a 34.5 % reduction in required PAC doses for 80 % MIB removal (i.e., PAC-Od-Rem80%, from 40.9 mg/L to 26.8 mg/L) in the two-stage process, compared to the single-stage process, suggesting that the two-stage PAC dosing strategy was effective in enhancing odorant adsorption in NOM-containing waters. The observed increase in adsorption capacity, resulting from the two-stage PAC adsorption process, may be attributed to a reduction in NOM competition during the second stage of the process, and/or a higher MIB loading onto the fraction of PAC during the first stage (lower PAC doses usually relate to higher loading of micropollutants). The results from NOM-free water (Fig. 2, right) demonstrate that the two-stage PAC dosing strategy did not outperform the single-stage PAC dosing strategy with regard to MIB adsorption in the absence of co-existing NOM. The MIB adsorption has been demonstrated to be highly reversible in the absence of background NOM [11]. Consequently, higher MIB loading onto the fraction of PAC during the first stage is less conceivable to enhance MIB adsorption from NOM-free water, as the desorption process may facilitate the transfer of adsorbed odorants to fresh adsorbents introduced at a later stage. Therefore, in raw, NOM-containing, source water, the alleviation of NOM competition may be the primary reason for the enhanced odorant removal by two-stage PAC dosing.

To elucidate the operational dynamics during a two-stage adsorption process, we assessed the individual contribution of each phase of PAC dosing on the removal of MIB and NOM constituents (Fig. 3 and Fig. 4). The individual contributions of the first and second adsorption stages were determined by incorporating an intermediate sampling point between the two PAC doses. By analyzing MIB and NOM concentrations following the first-stage PAC contact but prior to the second-stage addition, we were able to quantitatively distinguish the respective removal efficiencies of each stage. The relative contribution of the second-stage PAC dosing to MIB removal was 56 %-72 % at a first-/second-stage dose lower than 10 mg/L (total PAC dose of 20 mg/L). The low MIB loading on PAC observed at low adsorbent doses—corresponding to equilibrium concentrations above 350 ng/L—is primarily attributed to strong competition from highly adsorbable NOM fractions against trace-level micropollutants. This trend is consistent with previous studies, which have shown that at low PAC dosages, the limited number of high-energy adsorption sites is rapidly occupied by hydrophobic NOM fractions. As a result, micropollutants such as MIB are constrained to adsorb onto lower-affinity sites, thereby reducing the overall uptake capacity [28,32]. As the second-stage PAC dosing was carried out at a lower liquid-phase MIB concentration than the first stage, it can be concluded that the alleviated NOM competition, evoked by the first-step adsorption, had the greatest benefit for the second-step adsorption at this dose. Here, our comparison of MIB removal between the first and second stages is logical, as the removal efficiency of adsorptive pollutants/odorants at any given PAC dose in a raw source water (with a fixed NOM composition) is typically consistent—i.e.,

largely independent of the initial pollutant/odorant concentration [33,34]. This assumption holds when the equilibrium concentrations between the compared stages do not differ substantially.

Fig. 4 illustrates the relative contribution of enhanced NOM adsorption during the two-stage PAC process, with the corresponding isotherms presented in Fig. S5. At low PAC doses (< 10 mg PAC/L for each stage), the NOM loading on the activated carbon remained similar between first-stage and second-stage of PAC dosing (characterized as 40 %-60 % by UV_{254}). Considering the large difference between first- and second-stage in removing MIB, through their similar UV_{254} removal at bulk level, the competitiveness of the adsorbed NOM fractions could be different [29]. At high PAC dose range (≥ 25 mg/L for each stage), the decrease of relative NOM loading at the second-stage was less pronounced (from 26.7 % to 21.1 %) for the second-stage PAC dose, while the MIB loading contribution at the second-stage continued to decrease (from 30.6 % to 20.7 %, cf. Fig. 3). This indicates that the two-stage PAC dosing strategy at higher PAC doses could be less efficient.

3.2. Impact of odorant/PAC characteristics in multi-stage adsorption

Fig. 5 illustrates that for EDD and MIB, the most weakly adsorbable odorants among those examined, the reduction in PAC consumption, evaluated as PAC-Od-Rem80% was the largest (48.0 % and 34.5 %) in two-stage PAC dosing compared to a single-stage dosing process. When the PAC dosing stages increased from two to three or four, a slight reduction in the PAC consumption was still observed for the strongly adsorbable geosmin, BDD and BMD. However, the elevated PAC-Od-Rem80% for EDD/MIB adsorption even indicated a deteriorated benefit in EDD/MIB adsorption in the three- or four-stage adsorption process. In the early publication by Sontheimer et al. (1988), it has been proposed that multi-stage adsorption, which simulates the counter-current contact process, is associated with the selective adsorption of strongly adsorbable organic components from the bulk. The preferential adsorption of odorants over NOM components (DOC or UV/fluorescent surrogates) indicates that odorants should be recognized as medium-to-strong adsorbates in the multi-component continuum [35–37]. From our results, the further increase from two stages to three/four stages only enhanced the adsorptive extraction of strongly adsorbable odorants, while two-stage adsorption was optimal for the less strongly adsorbable odorants (EDD/MIB, medium adsorbates among all solutes). Further investigation is required to determine whether an unequal division of PAC dose can provide a greater increase in adsorptive extraction efficiency.

To compare the efficacy of different PAC adsorbents in the two-stage dosing process, three typical PAC products from different raw materials and with varying pore structure (average pore size from 2.17 nm to 4.23 nm) were studied. Reduced PAC consumption was observed for all PAC samples studied in the two-stage adsorption process, compared to the single-stage process (Fig. 6). Rich mesoporous SAE Super and the wood-
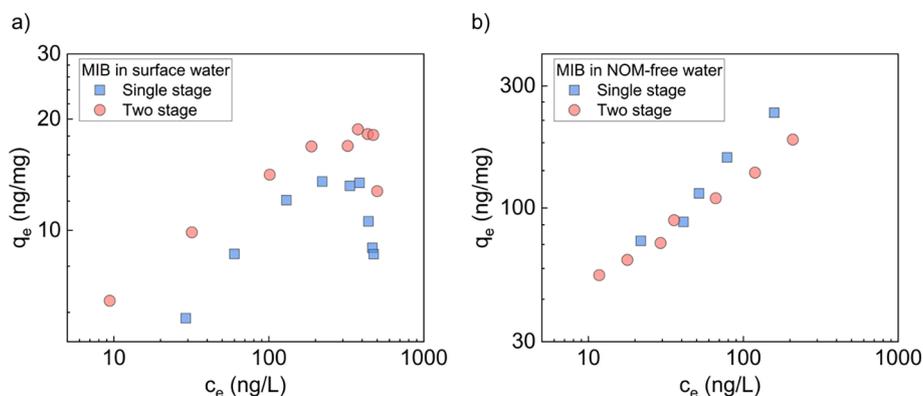

Fig. 2. MIB equilibrium adsorption isotherms for single-stage and two-stage PAC dosing in (a) NOM-rich source water and (b) NOM-free water; initial MIB concentration was 519 ± 22 ng/L; PAC: WB900.
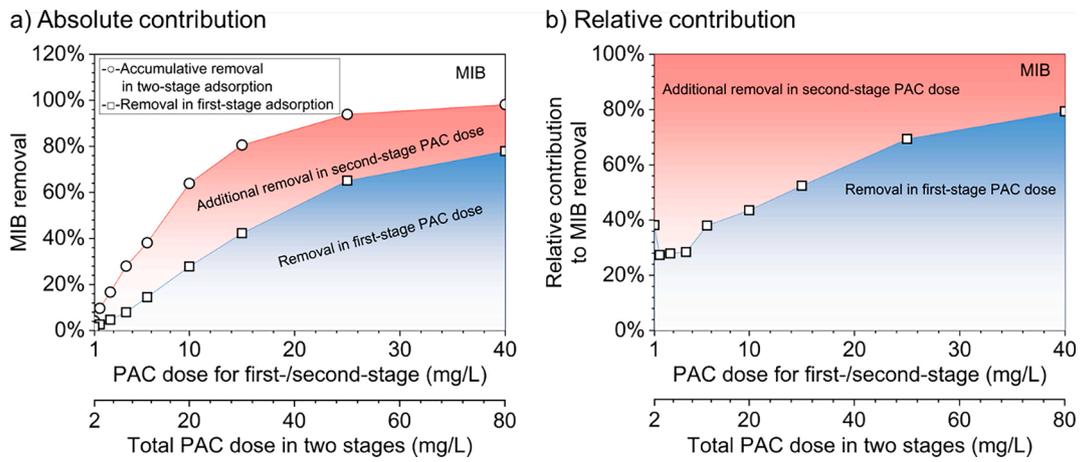
Fig. 3. The contribution of first/second-stage dose on MIB removal by PAC WB 900. a) absolute contribution; b) relative contribution.
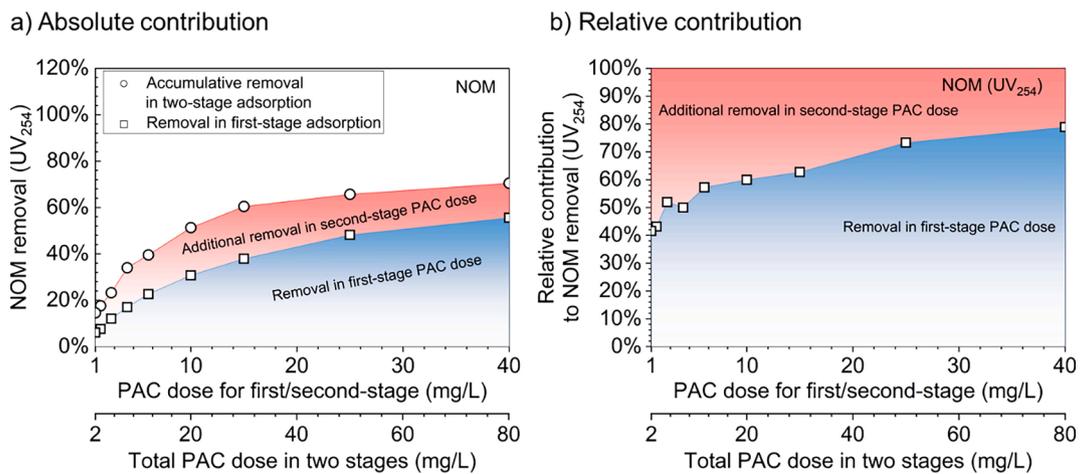
Fig. 4. The contribution of first/second-stage dose on NOM removal (evaluated using UV_{254}) by PAC WB900. a) absolute contribution; b) relative contribution.
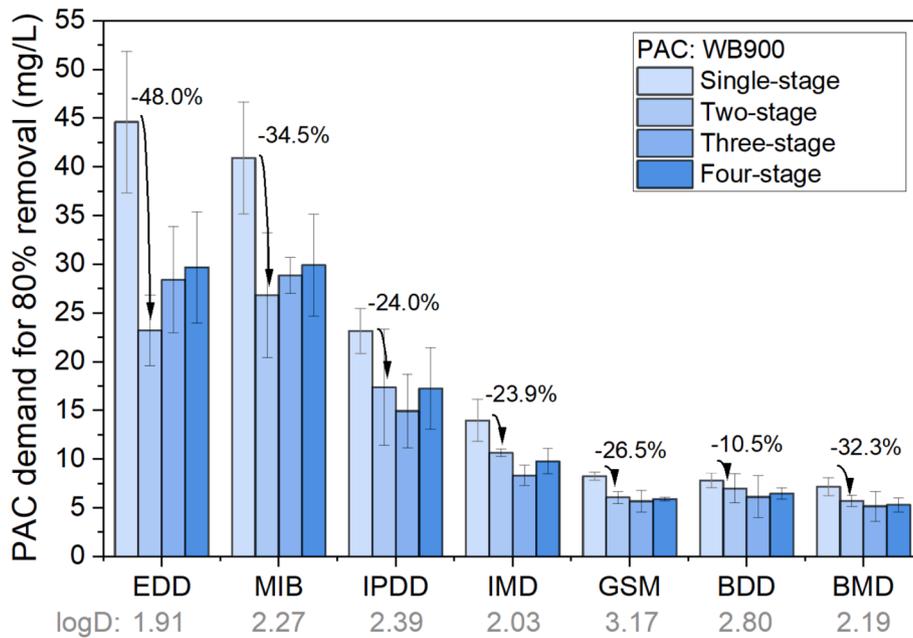
Fig. 5. PAC doses required for 80 % odorant removal (interpolated from data presented in adsorption isotherm data in Fig. S1) in 1-, 2-, 3-, and 4-stage PAC processes with equal PAC dose division among stages (e.g., 24 mg/L in the 1-stage process compare to 12 + 12 mg/L in the 2-stage, or 8 + 8 + 8 mg/L in the 3-stage, or 6 + 6 + 6 + 6 mg/L in the 4-stage process, respectively).

Fig. 6. Estimated one/two-stage PAC dosing required for 80% odorant removal (Od-Rem-PAC80%, interpolated from adsorption isotherm data in Fig. S1–S3) for PAC products with different pore structure.

based WB900 were associated with higher percentages of PAC demand reduction than the more microporous CK1000 suggesting that the microporous PAC itself may be in a lower capacity to mitigate NOM competition during two-step adsorption. A previous study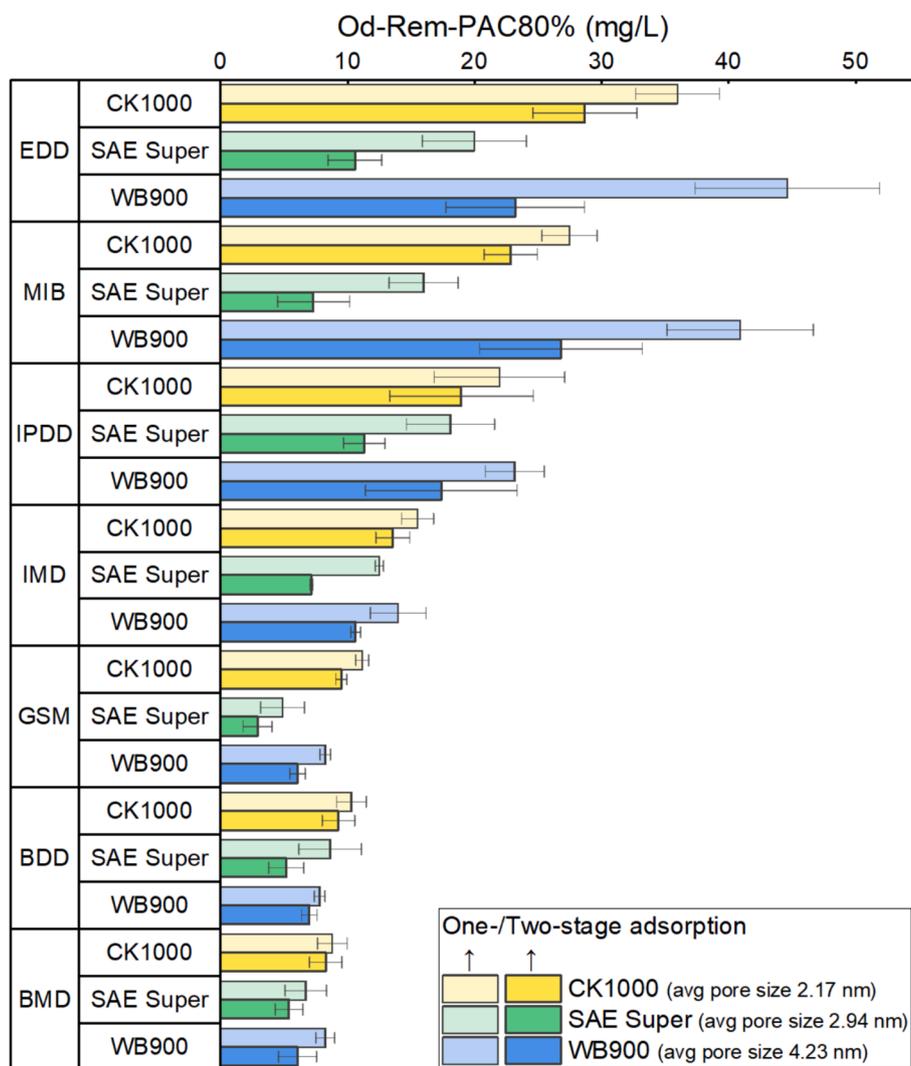 have also illustrated that the most competitive NOM components, whose molecular weights are mainly concentrated in the range of 150–450 Da [12], mainly adsorb in the narrow mesopores with larger sizes compared to odorants (116–182 Da), preferring the micropores [38]. In addition to the aforementioned reasons, the faster adsorption kinetics of mesoporous adsorbents further supports that PAC products with a higher proportion of mesopores may serve as more effective candidates for multi-stage adsorption inequilibrium processes at full-scale.

In Table 3 various scenarios and performances of literature-reported multi-stage adsorption processes for aqueous pollutant removal are summarized and compared. Prior multi-stage adsorption research, conducted earlier than 2015, focused on the adsorption of biologically refractory organics in the context of tertiary treatment of domestic wastewater and industrial wastewater dye treatment. Two-stage adsorption systems were found to save 20 %-60 % PAC consumption for the bulk organics removal [20,21]. In contrast to the sequential dosing of pristine adsorbents, Zietzschmann et al. (2015) proposed a multi-stage adsorption process using recirculation of used PAC for pollutant removal from WWTP effluent. Their findings indicated that one additional recirculation stage could increase the micropollutant

adsorption capacity of PAC up to 14 %. The comprehensive effect of PAC recirculation, resulting from this efficiency enhancement via multi-stage PAC reuse, yielded > 40 % higher micropollutant removals in comparison with a one-stage setup (Meinel et al., 2016).

In this study, we evaluated a multi-stage adsorption approach designed to be operationally streamlined and potentially applicable to source water treatment. It is worth noting that 80 % removal was used as the benchmark for evaluating PAC consumption savings. By contrast, previous studies often assessed savings based on higher, idealized removal efficiencies (90 %–99 %), which may not translate directly to practical settings. Therefore, the PAC savings achieved in our study—ranging from 10.5 % to 48 %—remain outstanding when compared against existing research. These PAC demand calculations are based on equilibrium conditions, which are unlikely to occur in full-scale water treatment plants where contact times are limited and dynamic. Nevertheless, equilibrium-based assessments serve as a useful benchmark for evaluating relative adsorbability across different compounds and informing the design and optimization of PAC dosing strategies.

3.3. Mitigated NOM competition in multi-stage adsorption

As shown in Fig. S4, improved adsorptive selectivity for NOM (inferred via UV$_{254}$) was found in the second-stage PAC adsorption compared to the first-stage PAC adsorption, achieving a higher MIB

Table 3
Applied scenarios and performance of the reported multi-stage adsorption process for aqueous pollutant removal.

Target adsorbate	Applied system	Concentration of target adsorbate	Mode for multi-stage adsorption	Performance	Optimal stages	Traits	Reference
Specific NOM/EfOM fractions	Water/ wastewater treatment	mg/L	Two-stage PAC dose; equal/ unequal division of PAC dose	25–60 % lower carbon dose (80 % target removal, computational results)	N/A	Selective separation of strongly adsorbed NOM/ EfOM components through adsorption analysis.	[21]
Bulk organics (COD)	RO-PAC in industrial wastewater treatment	mg/L	Two-stage PAC dose	42 % lower carbon dose	N/A	Coupled with membrane system.	[20]
Dyes (acid Blue 74, basic brown 1, methylene blue, etc.)	Not specified	mg/L	Two-/three-stage system with individual contractor	41–66 % PAC dose saving (99 % target removal)	One-stage (1/ n < 0.05) Two-stage (1/n < 0.06–0.2) Three-stage (1/n < 0.3–0.8)	Designed for single-solute system, theoretical calculation-based.	[22]
Pharmaceutical micropollutants (carbamazepine, diclofenac, sulfamethoxazole)	PAC process in tertiary wastewater treatment	ng/L − µg/L	Recirculation of used PAC (1–7 reuse stage)	~14 % additional removal per stage	The more the better, if practice feasible	Micropollutant cannot obtain favorable adsorption sites on the surface of the recirculated PAC adsorbent.	[19]
Pharmaceuticals, industrial chemicals (carbamazepine, benzotriazole, 4-formylaminoantipyrine)	PAC + coagulation, WWTP effluent	ng/L − µg/L	Recirculation of used PAC + addition of fresh PAC (14 stages)	~30–50 % additional removal (in total)	The more, the better	Coagulation has no effect (other than increased DOC removal)	[18]
Odorants	NOM-rich source water treatment	ng/L	Two-stage PAC dose, purge-flow	10.5–48.0 % PAC saving (80 % target removal, experimental results)	Two-stage for weak adsorbates; Three-/four-for strong adsorbates	Micropollutant can obtain favorable adsorption sites on the surface of fresh adsorbent in the subsequent stage PAC dose.	This study

adsorption with similar NOM loading. Therefore, to unravel the mechanism for the improved odorant selectivity, NOM adsorption/desorption was studied for the aforementioned PAC products. It is important to note that the desorption of adsorbed organics is noticeably stronger in NOM-free water than in NOM-containing waters, due to larger desorption concentration gradient [13]. With increasing PAC dose, and thus decreasing NOM loading, the NOM desorption decreased linearly from ~ 100 % at 5 mg/L to ~ 20 % at 80 mg/L PAC dose for all three examined PAC adsorbents (Fig. 7), confirming the partially irreversible NOM adsorption onto activated carbon. LeBoeuf and Weber [39] found that the desorption hysteresis (irreversible adsorption) is prominent for hydrophobic organic matter that are usually more effective competitors.
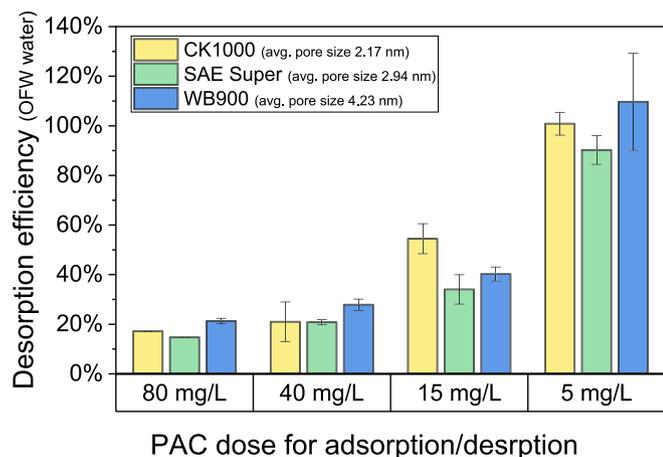

Fig. 7. Efficiency of NOM desorption at variable PAC doses for three different PAC products. Note that desorption evaluated in NOM-free water, with stronger desorption driving forces than NOM-containing water.

This also means that the NOM competitors effectively accumulated on a small fraction of the activated carbon adsorbents, allowing for the improvement of adsorptive selectivity of odorants in the two-stage adsorption as shown in Fig. S4. However, the adsorptive behavior inferred here is based primarily on bulk NOM indicators (e.g., UV_{254}), which do not differentiate among specific NOM components. To further elucidate the molecular basis of this partial irreversibility and competitive selectivity, future work should incorporate advanced NOM characterization techniques, such as size-exclusion chromatography, fluorescence spectroscopy or high-resolution mass spectrometry. These approaches will help identify the composition, adsorbability, and reversibility of key NOM fractions, and clarify their roles in competitive adsorption during staged PAC treatment.

Although the two-stage PAC dosing was developed in accordance with the principle of countercurrent adsorption, previous studies on micropollutant adsorption in NOM-free water have revealed that the desorption of adsorbed micropollutants is relatively easy to occur for trace levels of micropollutants (ng/L − µg/L) [13]. Consequently, step-by-step PAC injection is not an effective method for purifying the trace-level adsorbates in the absence of background NOM (as indicated in Fig. 2b). In contrast, the effectiveness of the two-stage PAC adsorption in raw source water is related to the alleviation of NOM competitive adsorption. This probably occurs when a small amount of PAC can enrich strongly competitive NOM at a higher loading in a more irreversible way. It has also been found that the adsorbed NOM not only has low desorption on its own, it also it has a limiting effect on the desorption of micropollutants [11,13,40]. Moreover, emerging evidence suggests that odorants may form hydrophobic associations or weak complexes with NOM constituents—particularly those with high aromaticity or high molecular weight—which may not only reduce their free-state availability for direct adsorption onto PAC, but could also shift the interaction from a competitive to a potentially cooperative regime

via co-adsorption or indirect stabilization effects [41]. Although such NOM-odorant association mechanisms are beyond the scope of the present study, they merit further investigation to fully elucidate the respective roles of competitive and cooperative interactions under varying NOM conditions.

To evaluate whether the subsequent PAC dose enhances odorant adsorption as a result of reduced NOM competition during the initial adsorption stage, isotherm and kinetic experiments were conducted using PAC-pretreated water. In this approach, raw source water was first treated with PAC (10 or 20 mg/L) for 24 h to partially remove strongly adsorbable NOM. The PAC was then removed by filtration, and odorants were spiked into the pretreated water to examine their adsorption behavior in the absence of competing NOM fractions (more details in Section 2.4). Fig. 8 illustrates that MIB adsorption capacity was enhanced from untreated source water to PAC-pretreated waters. The large discrepancy in isotherms observed in the experiments underscores the necessity for an operating diagram to elucidate the two-stage adsorption process in a multicomponent adsorption system. This contrasts with the multi-stage adsorption in a single-component system, as previously proposed by researchers [6,21]. The operational line for the second-stage adsorption should not align with the isotherm for first-stage adsorption (Fig. 8c). The second-stage adsorption isotherm occupies a higher position in the diagram due to the partially alleviated NOM competition in the second-stage adsorption [29].

A kinetic examination was also conducted to study the dynamic odorant adsorption in pretreated water, with an evaluation of kinetics using a modified second-order kinetic model (Fig. 8b and Table S3). This model allowed for a rational comparison of adsorption kinetics at different equilibrium loading conditions [30,31]. The results demonstrated that the kinetics of odorant adsorption was enhanced due to the

PAC pretreatment, with the modified second-order kinetic constant (k_2^*) increasing from $3.12\,\text{h}^{-1}$ to $3.42\,\text{h}^{-1}$ in PAC$_{10\text{mg/L}}$-treated water (i. e., surface water pretreated with 10 mg/L PAC) and $3.68\,\text{h}^{-1}$ in PAC$_{20\text{mg/L}}$-treated water. This indicates that the alleviation of NOM competition may not only result in an elevated equilibrium adsorption capacity but also in reinforced adsorption kinetics in PAC-pretreated waters. Although the period for the second-stage PAC adsorption was shorter due to the segmentation of the adsorption process, the enhanced adsorption kinetics ensured effective adsorption. The collective enhancement of adsorption capacity and kinetics implies a reduction in both site competition and pore blockage from background NOM, clarifying how NOM competition alleviated after early-stage PAC adsorption. In practical operation, the first-stage PAC dose can be introduced at the water intake, while the second-stage PAC could be added concurrently with coagulants (e.g., Fe^{3+} or Al^{3+}) in the treatment process to facilitate flocculation and sedimentation. While the current study isolated adsorption process only to explore mechanistic effects, the integration with coagulation may alter PAC dispersion dynamics and site accessibility. In particular, the addition of coagulants may restrict PAC mobility or induce premature agglomeration, thereby limiting its effective contact with micropollutants [42,43]. This underscores the strategic importance of the first-stage PAC dose in staged dosing designs, as it allows for sufficient interaction between PAC and odorants before coagulation takes place—potentially enhancing subsequent adsorption kinetics and overall removal efficiency. Further studies are warranted to evaluate this staged configuration under flow-through and coagulation-coupled scenarios representative of full-scale applications.

The enhancement effect of the two-stage adsorption was thus potentially achieved by three collective mechanisms (Fig. 9): the accumulation of competitive NOM during the first-stage PAC adsorption and
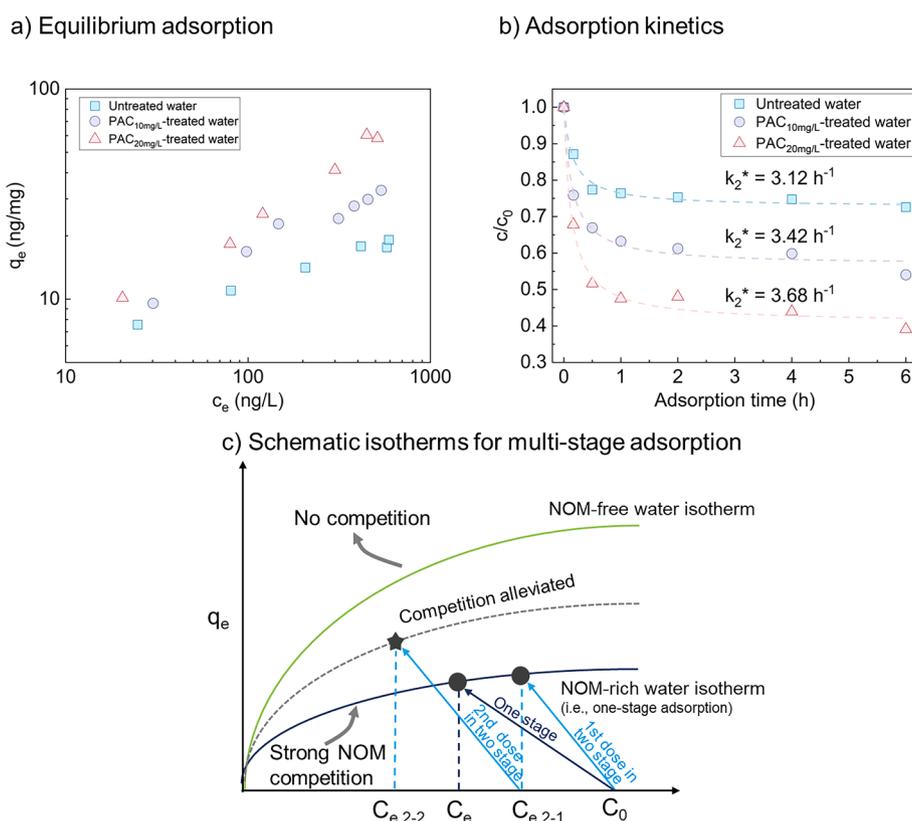

Fig. 8. a) MIB adsorption isotherms, b) MIB adsorption kinetics (PAC dose 10 mg/L), evaluated with modified second-order kinetic constant (k_2^*), in untreated source water and PAC-pretreated water by PAC WB900; c) Schematic illustration of multi-stage PAC adsorption based on isotherm and operation lines. C_0 represents the initial concentration of the target compound before any PAC addition. C_e denotes the final equilibrium concentration after a one-stage PAC adsorption at the total dose. In the two-stage dosing scenario, $C_{e,2-1}$ refers to the intermediate concentration after the first-stage PAC contact, and $C_{e,2-2}$ represents the final equilibrium concentration after the second-stage PAC addition.
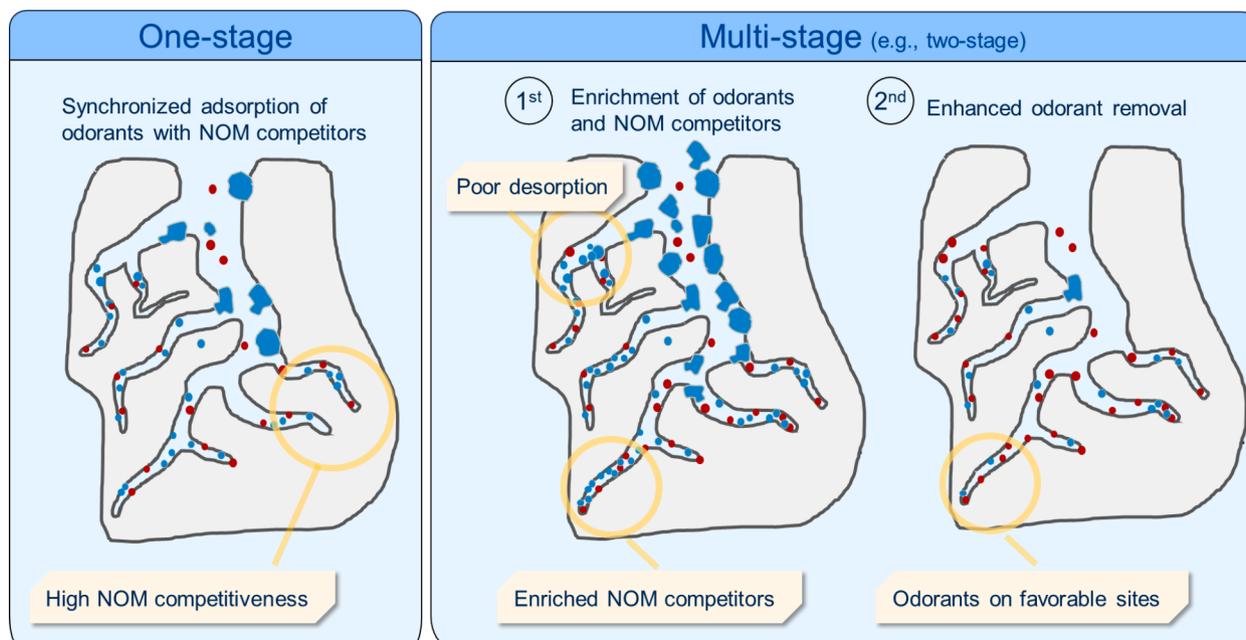
Fig. 9. Schematic diagram of the principle of multi-stage PAC adsorption in mitigating NOM competition against odorants.

less desorption associated with poor NOM adsorption reversibility; the potential desorption inhibition of the adsorbed odorants by high NOM loading on first-stage PAC; and the strengthening of the odorant adsorption kinetics during second-stage PAC dose.

4. Conclusion and Outlook

- This study confirmed that multi-stage PAC adsorption is a practical and effective strategy for improving odorant removal by mitigating NOM competition. Specifically, two-stage adsorption was effective in the removal of trace-level odorants in NOM-rich water, while not helpful in NOM-free water.
- PAC adsorbents with a higher percentage of narrow mesopores adapted two-stage adsorption better with a stronger reduction of PAC consumption. Odorants with weak PAC adsorbability, such as MIB and EDD, benefited most from the two-stage PAC dose than three-stage or four stage doses. A more than two-stage PAC dose did not notably increase the odorant adsorption. Further research on combining different PAC products, unequal division of PAC dosage, and studies under non-equilibrium conditions is highly recommended to achieve higher adsorption efficiency in waterworks.
- Mechanistic studies revealed that the enhancement effect of the two-stage adsorption was potentially achieved by three collective mechanisms: the irreversible accumulation of competitive NOM during the first-stage PAC adsorption; the desorption inhibition of the adsorbed odorants by high NOM loading on first-stage PAC; and the strengthening of the odorant adsorption kinetics during the second-stage dose of fresh PAC. In summary, our findings provide practical, cost-effective strategies to mitigate NOM competition, improve odorant adsorption, and reduce PAC usage in drinking water treatment practices.
- While this study provides valuable insights into the staged PAC adsorption process, several aspects warrant further investigation to enhance its practical applicability. Future research should explore the use of multiple PAC products, non-uniform PAC dose allocation strategies, and adsorption performance under non-equilibrium conditions representative of full-scale waterworks. Additionally, given that NOM characterization in this study was limited to bulk parameters (e.g., DOC, UV_{254}), the application of advanced analytical tools—such as size-exclusion chromatography, fluorescence

spectroscopy or high-resolution mass spectrometry—would be beneficial for unraveling specific NOM fractions most responsible for competitive adsorption, and for identifying potential threshold conditions—whether in NOM concentration or composition—above which the two-stage PAC adsorption offers a clear advantage over one-stage configurations. These efforts would contribute to the development of more efficient, selective, and site-adapted PAC dosing strategies.

CRediT authorship contribution statement

Qi Wang: Writing – original draft, Visualization, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Shaozhen Nie:** Writing – original draft, Validation, Investigation, Formal analysis, Data curation. **Frederik Zietzschmann:** Writing – review & editing, Supervision. **Luuk C. Rietveld:** Writing – review & editing, Supervision. **Fang Liu:** Writing – review & editing, Validation, Investigation. **Min Yang:** Writing – review & editing, Resources, Project administration. **Jianwei Yu:** Writing – review & editing, Supervision, Project administration, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.seppur.2025.134270.

Data availability

Data will be made available on request.

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