

Solid Waste Valorization-Driven Synthesis of *Pinus yunnanensis* Bark-Derived Adsorbent for Efficient Styrene Removal

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Abstract

Forestry waste valorization is critical for the circular economy. This work converts *Pinus yunnanensis* bark into styrene adsorbents via hydrothermal carbonization, with H₂PO₄/KOH modification to enhance performance. Acid-modified carbon showed a 41% higher specific surface area than the unmodified sample. It exhibited excellent thermal stability at 142–268°C. Styrene adsorption on modified carbons fitted the pseudo-second-order model (chemisorption), while the unmodified sample followed the pseudo-first-order model (physical adsorption). This study provides a sustainable route to upcycle forestry waste into efficient VOC adsorbents, aligning with green development goals.

Introduction

In 1989, the World Health Organization (WHO) formally defined volatile organic compounds (VOCs) as a category of organic compounds that exist in the atmospheric environment in a vapor phase. The defining physicochemical properties of VOCs, as specified in this definition, include: the saturated vapor pressure of over 133.332 Pascals (Pa) under ambient temperature conditions; and the boiling point range of 323 Kelvin (K) to 533 Kelvin (K) at standard atmospheric pressure (101.325 kilopascals, kPa)^[1–3]. The primary sources of volatile organic compounds (VOCs) in the atmosphere encompass two major categories^[4]: anthropogenic sources, which include human production activities and daily life processes, and natural sources, such as plant metabolism, forest fires, and volcanic eruptions. Indoors, VOC emissions mainly originate from daily office supplies, printers, and other similar electrical or mechanical equipment^[5]. During industrial production processes, the VOCs generated are chemically diverse, with the main classes including alkanes, aromatic hydrocarbons, halogenated hydrocarbons, and sulfur/nitrogen-containing compounds^[6]. Most volatile organic compounds (VOCs) exhibit high toxicity, carcinogenicity, and hazardous properties, thereby posing substantial threats to human health and the ecological environment. From the perspective of human health, prolonged exposure of healthy individuals to VOCs at concentrations above a certain threshold can induce irritative damage to the eyes, nasal cavity, and pharyngeal mucosa; in severe cases, it may further trigger systemic symptoms such as headaches, as well as impairments in memory and visual function, and in extreme instances, even result in death^[7, 8]. Certain specific volatile organic compounds (VOCs), such as styrene and ethylbenzene, further exhibit a certain degree of carcinogenicity^[9]. Alkanes, aromatic compounds, and alkenes contribute to photochemical smog formation, while halogenated hydrocarbons like CFCs deplete the ozone layer^{10–12]}. Photochemical smog, characterized by high ozone concentrations, arises from photochemical reactions involving VOCs and nitrogen oxides under specific climatic conditions^[13, 14]. The ozone layer, vital for absorbing harmful UV radiation, faces depletion due to halogenated compounds^[11, 12].

Volatile Organic Compounds (VOCs) serve not only as precursors to PM_{2.5} but also exert direct adverse effects on human health^[15-17]. Styrene, as a typical volatile organic compound (VOCs), indeed poses significant threats to the atmospheric environment and human health. It not only has an unpleasant foul odor and irritating smell but also participates in photochemical reactions in the atmosphere, forming secondary pollutants such as ozone and PM_{2.5}, which exacerbates air pollution^[18-20].

In the field of volatile organic compounds (VOCs) abatement, adsorption technology has emerged as a crucial treatment technique, attributed to its characteristics of high efficiency, low energy consumption, high adsorption capacity, recyclability, and environmental friendliness^[21-25]. The adsorption method utilizes the pore structure and surface properties of adsorbents (such as activated carbon, zeolites, etc.) to capture and immobilize volatile organic compound (VOCs) molecules. Compared with other VOCs abatement technologies, the adsorption method exhibits significant advantages when treating waste gases containing low-concentration VOCs^[26-30]. The global planting area of pine trees is approximately 120 million hectares, mainly distributed in Asia, Europe, and North America^[31]. China is one of the countries with the richest pine tree resources in the world, with a planting area of over 80 million hectares, accounting for more than 65% of the global total^[31]. In Liangshan Prefecture, Sichuan Province, China, pine forests cover a vast area; however, the region also faces frequent forest fires and the problem of forestry waste. If such waste, such as pine bark, is not utilized, it tends to accumulate and trigger fires, posing a threat to the ecological environment^[32]. Converting forestry waste into high-value products, such as biomass activated carbon, is an important approach to achieve sustainable development. This not only reduces waste accumulation and lowers fire risks but also provides useful materials for other fields^[33].

Liangshan Prefecture is rich in pine resources, particularly *Pinus yunnanensis* and *Pinus armandii*^[34], and using local pine bark to prepare biomass activated carbon presents multiple potential advantages: it leverages the region's abundant pine resources to secure sufficient raw materials for activated carbon production^[34]; as pine bark is a type of forestry waste with low acquisition costs, it helps reduce the production cost of activated carbon; converting pine bark into activated carbon can not only reduce waste accumulation and lower fire risks but also provide a low-cost solution for VOCs abatement^[35]; additionally, this approach benefits the ecological protection and sustainable development of regional pine forests^[36] while realizing the resource utilization of forestry waste to increase local residents' income and promote local economic development^[36].

Methods

Materials

Phosphoric acid (H_3PO_4), potassium hydroxide (KOH), and potassium permanganate (KMnO_4) were of analytical grade and purchased from Chengdu Kelong Chemical Reagent Co., Ltd. Styrene (C_8H_8) was of analytical grade and obtained from Shanghai Macklin Biochemical Technology Co., Ltd. Yunnan pine barks were collected from the mountains within Xichang Forest Park, Xichang City, Sichuan Province, China.

Description of Reaction System

During the operation of the experimental apparatus, first purge the pipeline with nitrogen for 30 minutes. After stabilization, control the flow rate at 500 mL/min and adjust the thermostatic water bath temperature to 25 °C. Use a 1-mL syringe to draw 0.5 mL of liquid styrene and inject it into the styrene generator. Weigh 1 g of adsorbent and place it into the adsorption tube. Simultaneously prepare a 5% potassium permanganate solution for tail gas treatment. Pump air into the system using an air pump to drive the styrene gas from the generator through the gas buffer bottle, adsorption tube, and 500-mL gas sampling bag, then pass it through the tail gas purification device before discharge. Adsorption saturation is deemed achieved when the inlet styrene concentration at both ends of the adsorption tube reaches 95% of the outlet concentration.

Preparation of Biomass-Based Adsorbent Materials

Yunnan pine barks were soaked in deionized water for 6 hours, followed by three rounds of washing to remove surface impurities such as dust. They were then dried in an oven at 105°C for 12 hours, crushed, and sieved through a 100-mesh screen. The processed barks were mixed with deionized water at a mass-to-volume ratio (g:mL) of 1:10 in a polytetrafluoroethylene (PTFE) liner, stirred uniformly, and allowed to stand for 1 hour. The mixture was then sealed in a hydrothermal reactor and reacted at a constant temperature of 240°C for 4 hours. After the reactor cooled down, the product was filtered using a Buchner funnel, dried in an oven at 105°C for 12 hours, and named pine bark-based adsorbent materials (PBAM), Fig. 1.

Figure 1

Three grams of PBAM was added to a 150 mL conical flask, followed by 60 mL of 1 mol/L H_3PO_4 solution. The mixture was oscillated at 150 rpm for 6 h at room temperature (23°C), then statically soaked for another 8 h. Subsequently, the product was filtered, rinsed with distilled water until neutral, and finally dried in an oven at 105°C for 12 h to obtain the acid-modified pine bark-based adsorbent material (AMPBAM).

Three grams of PBAM was introduced into a 150 mL conical flask, with 60 mL of 1 mol/L KOH solution subsequently added. The mixture was agitated at 150 rpm for 6 h at ambient temperature (23°C), followed by static immersion for an additional 8 h. Afterward, the resultant product was filtered, rinsed with distilled water until neutrality was achieved, and finally oven-dried at 105°C for 12 h to yield the alkali-modified pine bark-based adsorbent material(AlkPBAM).

Analytical Methods

The surface morphology was observed via a TESCAN MIRA LMS scanning electron microscope (Czech Republic). The contents of elements including carbon, hydrogen, oxygen, nitrogen, and sulfur were determined using an Elementar Unicube organic elemental analyzer (Germany). A Fourier transform infrared spectrometer (FT-IR-650) was employed to analyze the types and relative contents of oxygen-containing functional groups. Thermal stability was evaluated with a TA Discovery TGA 550 thermogravimetric analyzer (USA). The adsorption properties were characterized using a Micromeritics ASAP 2460 specific surface area and porosity analyzer.

Results and discussion

Microscopic morphological characteristics

Scanning electron microscopy (SEM) characterization was performed on the three prepared biochars, namely PBAM, AMPBAM, and AlkPBAM. From the perspective of microscopic morphology, the differences in the materials during the preparation process as well as the changes after acid modification and alkali modification were investigated.

Figure 2

As can be seen from Fig. 2 (a, b), PBAM exhibits the highest quantity of relatively small pores. These pores serve as abundant adsorption sites for styrene molecules, thereby endowing PBAM with excellent adsorption potential for styrene. It can be observed from Fig. 2 (c, d) that the surface pores of AMPBAM are arranged in a relatively regular pattern and possess the highest porosity. This structural feature is conducive to enhancing the molecular diffusion rate to a certain extent, facilitating faster mass transfer of adsorbate molecules from the exterior to the interior of the biochar particles. As shown in Fig. 2 (e, f), AlkPBAM is characterized by larger surface pores, accompanied by partial pore stacking. Such a pore structure exerts a dual effect on the adsorption of styrene molecules—both promotional and inhibitory. Specifically, larger pores may reduce mass transfer resistance (promotional), while pore stacking might block some effective adsorption sites

(inhibitory). Combined with the results of adsorption experiments, it is confirmed that the promotional effect of AlkPBAM on styrene adsorption outweighs the inhibitory effect.

Thermal stability is another crucial factor for evaluating the adsorption performance of activated carbon. Excellent thermal stability allows activated carbon to maintain favorable adsorption performance across a range of environmental conditions, which is essential for its practical application. In the present study, thermogravimetric analysis (TGA) was utilized to investigate the thermal stability of PBAM, and the resulting TGA curves are illustrated in Fig. 3 below.

Figure 3

As presented in Fig. 3, the weight loss behavior of PBAM can be categorized into four distinct stages. First stage (30–299°C): This stage corresponds to the water evaporation process. The biomass char exhibited minimal weight variation, with a total weight loss of 9.618%. Second stage (299–358°C): A sharp weight reduction of the biomass char was observed in this temperature range, accounting for a weight loss of 16.011%. Concurrently, the corresponding derivative thermogravimetric (DTG) curve displayed a prominent peak at 328°C, which indicates the maximum weight loss rate at this specific temperature. Third stage (358–603°C): Weight loss in this stage was attributed to the oxidative degradation of hydroxyl groups, during which gaseous products (i.e., H₂O, CO₂, and CO) were generated and released. The cumulative weight loss reached 39.157% in this stage. Fourth stage (603–800°C): This stage is defined as the ash combustion period. At the end of this stage, the residual mass of the biomass activated carbon was only 13.57%.

As deduced from the TGA curve, the activated carbon maintained high thermal stability within the temperature range of 142–268°C (note: "below" was adjusted to "within" to align with the subsequent "142–268°C interval" and logical consistency, as "below a range" is grammatically imprecise; the intended meaning is stability inside this specific temperature span), with a weight loss of merely 3.84%. This result confirms that the biomass activated carbon synthesized in this study possesses favorable adsorption stability within the temperature interval of 142–268°C.

The adsorption capacity of activated carbon is closely related to its specific surface area and pore structure, which are also the key focuses in the analysis of activated carbon's adsorption performance. The specific surface area, porosity, adsorption curve types, and their variation patterns of PBAM and AMPBAM are presented in Fig. 4.

Figure 4

As shown in Fig. 4, the N₂ adsorption-desorption isotherms of PBAM partially overlap. According to the classification criteria of the International Union of Pure and Applied Chemistry (IUPAC), these isotherms exhibit characteristics consistent with Type IV isotherms.

When the relative pressure (P/P_0) ranges from 0 to 0.46, the adsorption curve and desorption curve completely overlap, indicating that PBAM is dominated by micropore adsorption in this range. In the P/P_0 range of 0.46–1, an H4-type hysteresis loop is observed, which suggests that PBAM possesses both microporous (pore diameter < 2 nm) and mesoporous (pore diameter between 2 nm and 50 nm) structures. When $P/P_0 = 1$, the adsorption curve tends to stabilize, with a maximum adsorption capacity of approximately 40 cm³/g.

Meanwhile, the pore size distribution plots reveal that both PBAM and AMPBAM exhibit distinct peaks in the 5–7 nm region. This indicates that both materials are dominated by mesopores while also containing a portion of micropores. The unique characteristics of this pore structure endow PBAM-based materials with excellent adsorption performance.

The specific surface area, total pore volume, and average pore diameter of PBAM and AMPBAM, along with their specific values, are presented in Table 1.

Table 1
EA analysis was conducted to determine the elemental composition of the samples

Sample names	$S_{\text{BET}}(\text{m}^2 \text{ g}^{-1})$	$V_{\text{total}}(\text{cm}^3 \text{ g}^{-1})$	average pore diameter (nm)
PBAM	23.8724	0.062370	7.4276
AMPBAM	33.8404	0.091726	7.8518

Table 1

As shown in Table 1, after acid modification, both the specific surface area and pore volume increased significantly. Combined with the N₂ adsorption-desorption isotherms, it can be observed that the biomass activated carbon after acid modification possesses a more abundant pore structure.

EA analysis was conducted to determine the elemental composition of the samples

The adsorption capacity of activated carbon is closely associated with its elemental composition and carbon content, which also constitutes one of the key indicators for

assessing the superiority of activated carbon's adsorption performance. Organic elemental analysis was applied to conduct a comparative investigation on the elemental compositions between the raw activated carbon and the prepared biomass-based activated carbon, with the results summarized in Table 2.

Table 2

EA analysis was conducted to determine the elemental composition of the samples

Sample names	C(%)	H(%)	O(%)	N(%)
Pine bark powder	49.26	0.63	44.87	5.24
PBAM	57.52	0.64	37.11	4.73
AMPBAM	61.03	0.66	33.59	4.72
AlkPBAM	52.28	0.56	42.82	4.34

Table 2

Table 2 shows the raw material pine bark powder is mainly composed of carbon (C) and oxygen (O), with their contents being 49.26% and 44.87%, respectively. After the synthesis of PBAM via the hydrothermal method, the C content increased significantly: the C contents of PBAM, AMPBAM, and AlkPBAM increased by 8.26%, 11.77%, and 3.02%, respectively, while the contents of O and nitrogen (N) both decreased to a certain extent. This phenomenon can be attributed to the raw material undergoing a series of chemical reactions, such as hydrolysis, decarboxylation, and polymerization, which form and retain hydrochar, accompanied by the generation of some irrelevant by-products.

When $H_2PO_4^-$ is used as a modifier, it may undergo chemical reactions with certain components in the biomass char during the modification process to form new carbon-containing compounds; these compounds may then lead to a certain increase in the total carbon content of the biomass char. In contrast, KOH modification provides a large number of OH^- ions, which promote the formation of abundant oxygen-containing groups (e.g., $COOH$ and OH). As a result, the oxygen content of AlkPBAM is increased to a certain degree.

Experimental conditions of styrene adsorption and removal

Experiment on dynamic adsorption curves for styrene adsorption and removal

To investigate the styrene adsorption performance of PBAM, AMPBAM, and AlkPBAM, their adsorption performance was evaluated by determining and calculating the saturated

adsorption capacity and the time required to reach the saturated state, as well as plotting dynamic adsorption curves. Meanwhile, a comparative analysis was conducted on their styrene adsorption behaviors under aerobic and anaerobic environments. The dynamic adsorption curves of PBAM, AMPBAM, and AlkPBAM for styrene are shown in Fig. 5 below.

Figure 5

As shown in Fig. 5, the adsorption capacities of the three different adsorbents (PBAM, AMPBAM, and AlkPBAM) under aerobic conditions are all higher than those under anaerobic conditions, indicating that oxygen has a promoting effect on styrene adsorption.

The maximum adsorption capacity of AMPBAM is 0.015 mg/g. Compared with PBAM, the maximum adsorption capacity of AlkPBAM for styrene is increased by 22%, reaching 0.011 mg/g; meanwhile, the maximum adsorption capacity of AMPBAM for styrene is increased by 66% compared with PBAM, reaching 0.015 mg/g. When the adsorption time reaches 180 min, the adsorption of styrene by PBAM basically reaches saturation, and PBAM loses its adsorption capacity.

Adsorption Breakthrough Curves of PBAM, AMPBAM, and AlkPBAM for Styrene

A dynamic evaluation apparatus was used to evaluate the adsorption performance of three adsorbents, namely PBAM, AMPBAM, and AlkPBAM. Figure 6 shows the styrene breakthrough curves of these three adsorbents at a concentration of 50 ppb. Adsorption breakthrough was considered to be achieved when the outlet styrene concentration reached 5% of the inlet styrene concentration ($C_t/C_0 = 0.05$); adsorption saturation was deemed to be reached when the outlet styrene concentration reached 95% of the inlet concentration ($C_t/C_0 = 0.95$).

Figure 6

As shown in Fig. 6, the adsorption breakthrough time of PBAM is only 15 min, while the adsorption breakthrough times of AMPBAM and AlkPBAM are mainly concentrated at 30 min. This is because the activation with KOH and H_2PO_4 can dehydrate and remove the tar and volatile substances generated during the synthesis of biomass-activated carbon; meanwhile, it also increases the specific surface area and porosity to a greater extent, thereby improving the adsorption performance of AMPBAM and AlkPBAM.

Optimization of Current Density

To investigate the styrene adsorption mechanisms of PBAM, AMPBAM, and AlkPBAM, curve fitting analysis of the adsorption process was performed using pseudo-first-order

and pseudo-second-order kinetic models, respectively, as shown in Fig. 7 and Table 3.

Table 3
Kinetic Fitting Parameters for Styrene Adsorption and Removal

Sample names	pseudo-first-order kinetic model			pseudo-second-order kinetic model		
	Q_e	K_1	R^2	Q_e	K_2	R^2
	(mg/g)			(mg/g)		
PBAM	0.01016	0.01665	0.9650	0.01335	1.12328	0.9504
AMPBAM	0.01768	0.01108	0.9755	0.02518	0.34444	0.9990
AlkPBAM	0.01245	0.01478	0.9678	0.01670	0.77687	0.9976

Figure 7

Table 3

As shown in Fig. 7, the styrene adsorption process of PBAM tends to conform to the pseudo-first-order kinetic model ($0.9650 > 0.9504$). However, when examining the styrene adsorption performance of AMPBAM and AlkPBAM, it is evident that their degree of fitting with the pseudo-second-order kinetic model is higher, with values of $0.9990 > 0.9755$ and $0.9976 > 0.9678$, respectively.

This result reveals that the adsorption mechanism of AMPBAM and AlkPBAM is not limited to physical diffusion; chemical interactions also play a crucial role. This can be attributed to the higher content of oxygen-containing functional groups on the surfaces of AMPBAM and AlkPBAM, which is consistent with the results of the FT-IR analysis. In contrast, PBAM has a lower content of surface functional groups, and its adsorption process is mainly dominated by physical diffusion.

Conclusions

In summary, biomass-activated carbon was synthesized from pine bark via hydrothermal carbonization. The specific surface area of the acid-modified biomass-activated carbon was $33.8404\text{ m}^2/\text{g}$, which was 41% higher than that of the unmodified one. The pine bark-based biomass-activated carbon exhibited good adsorption stability in the temperature range of $142\text{--}268\text{ }^\circ\text{C}$, with a weight loss of only 3.84%. The maximum adsorption capacities for styrene were 0.009 mg/g for the unmodified biomass-activated carbon, 0.011 mg/g for the alkali-modified one, and 0.015 mg/g for the acid-modified counterpart. The adsorption of styrene by the acid-modified and alkali-modified biomass-activated carbons was better fitted to the pseudo-second-order kinetic model, while the adsorption by the unmodified biomass-activated carbon was more consistent with the pseudo-first-order kinetic model.

Declarations

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Author contributions

L.N. design, experiment, analysis, drafting. H.Z. design, analysis. H.D. design, drafting. Y.Q. analysis, investigation. L.M. analysis, investigation. H.L. investigation.

Competing interests

The authors declare no competing interests.

Data Availability

Data is available under reasonable request to the corresponding author.

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Figures

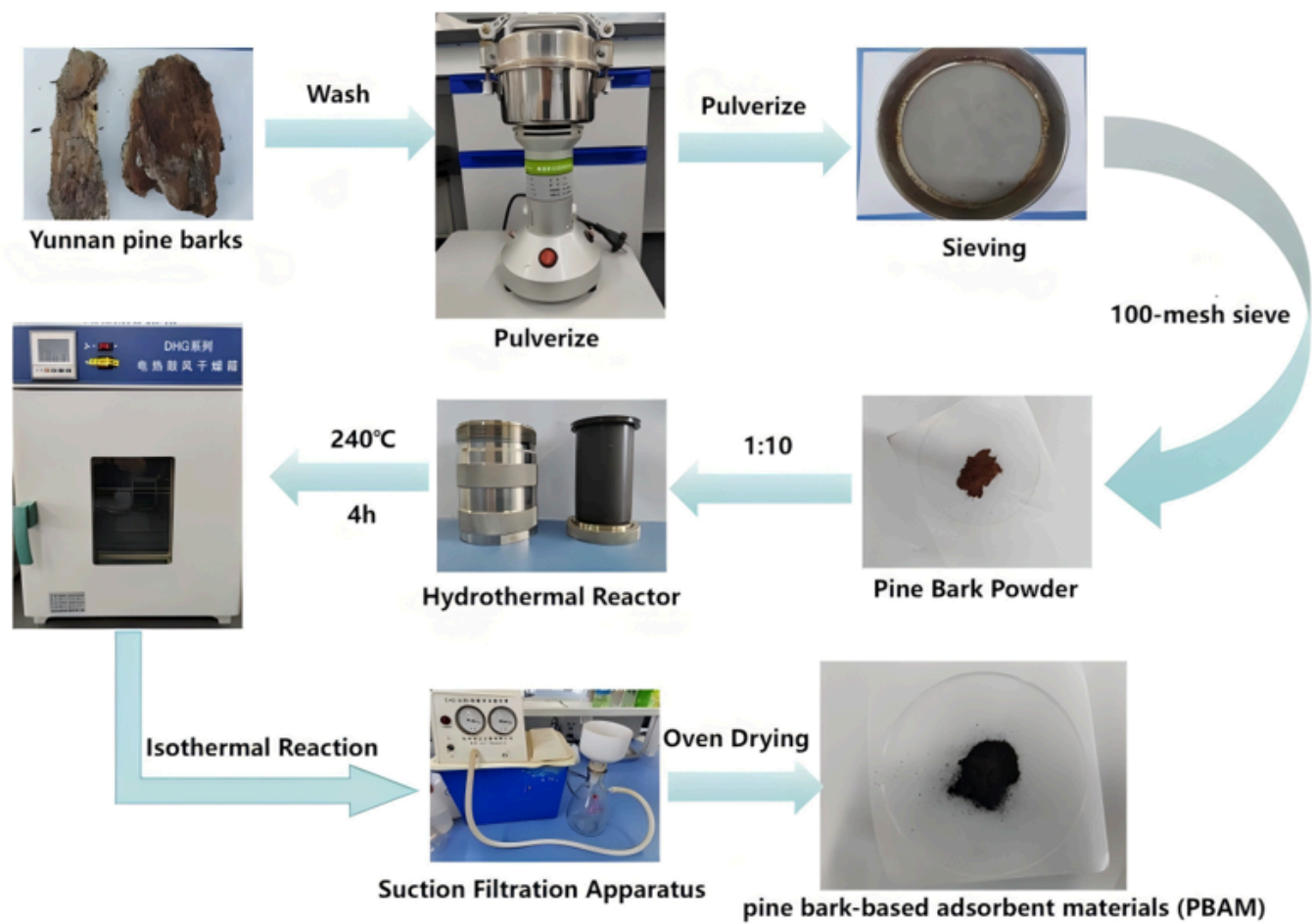


Figure 1

The Preparation Process of PBAM

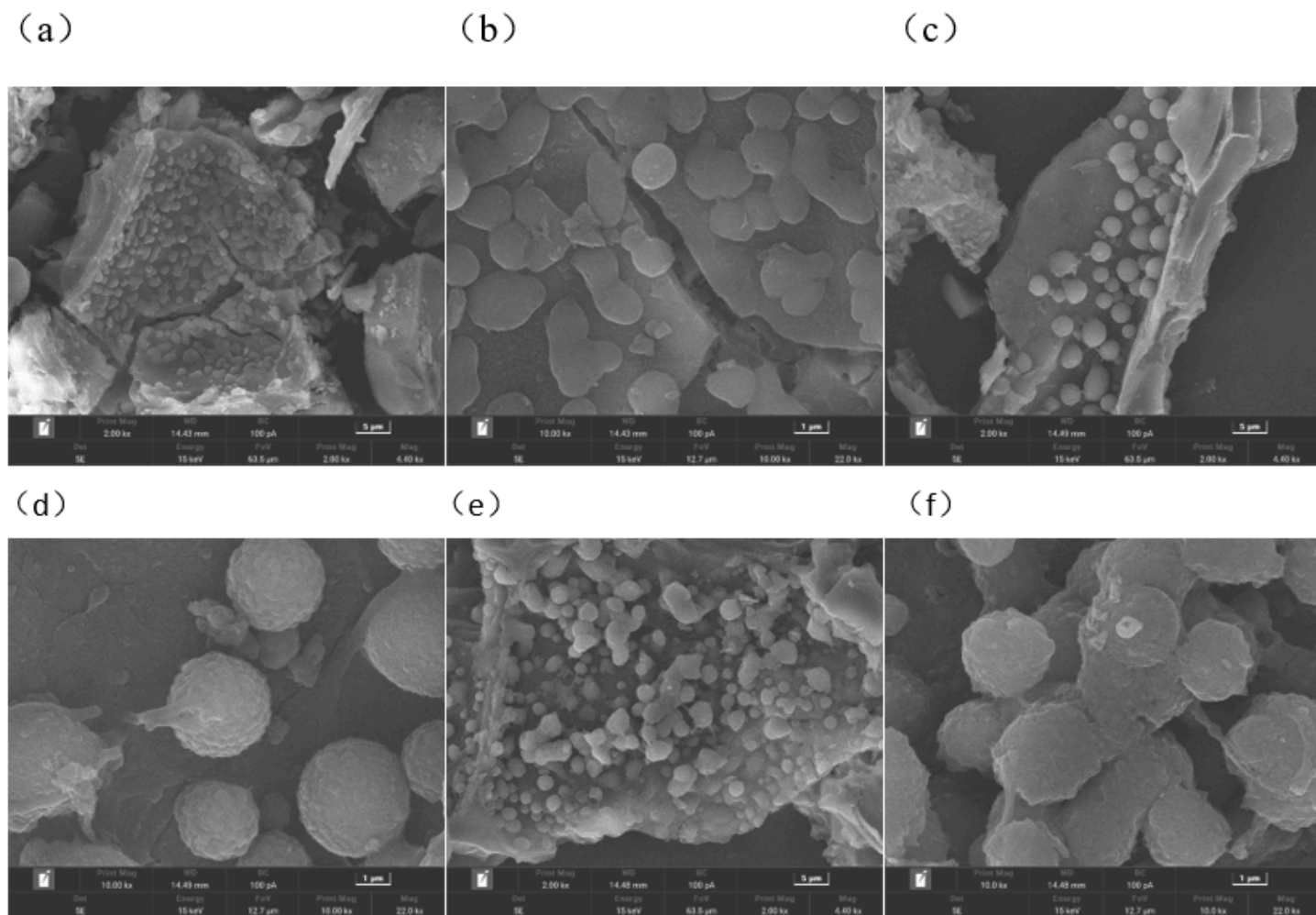


Figure 2

Scanning electron micrographs of PBAM, AMPBAM and AlkPBAM (a, b) PBAM; (c, d) AMPBAM; (e, f) AlkPBAM

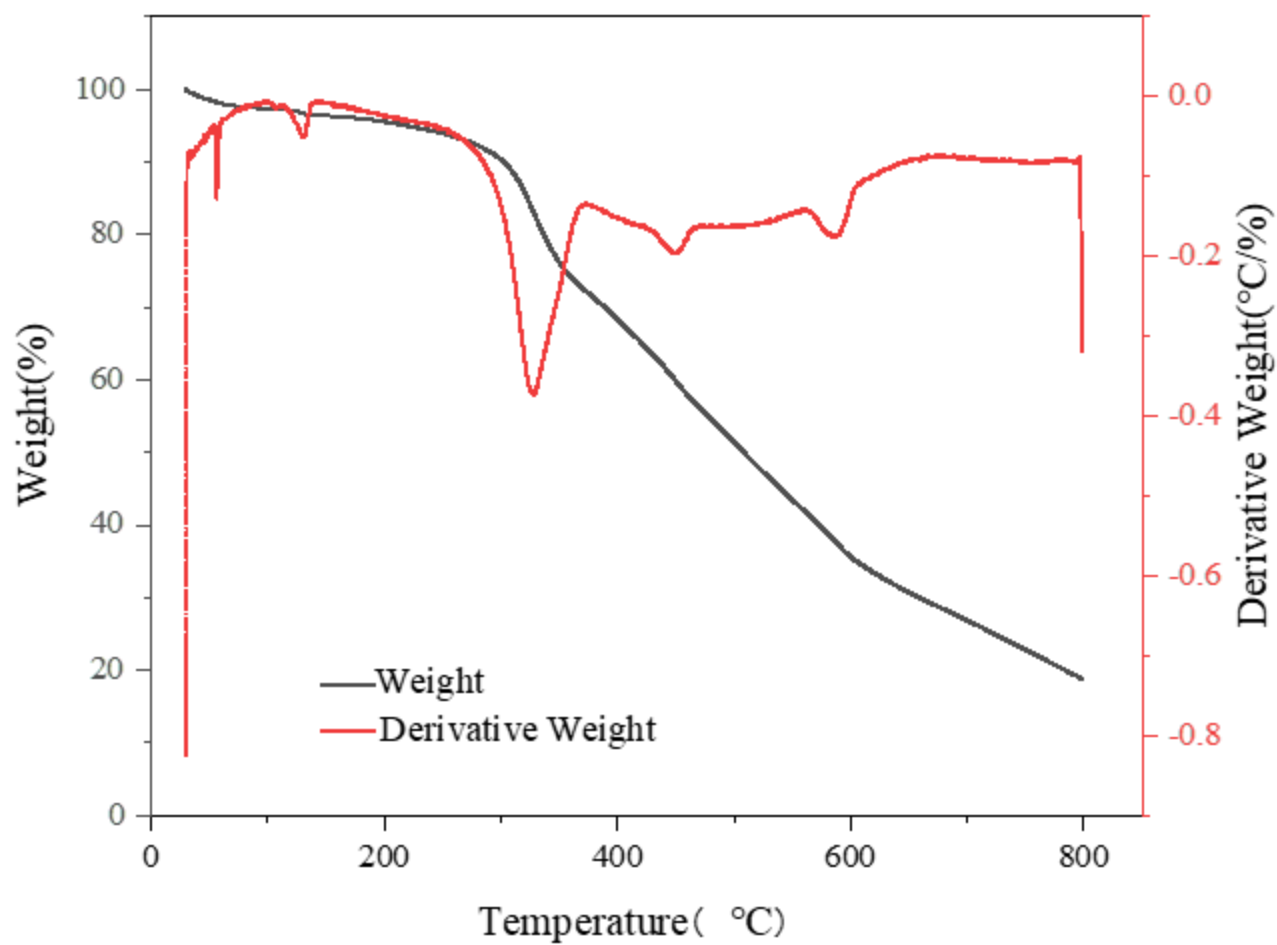


Figure 3

Thermogravimetric curve (TGA curve) of PBAM

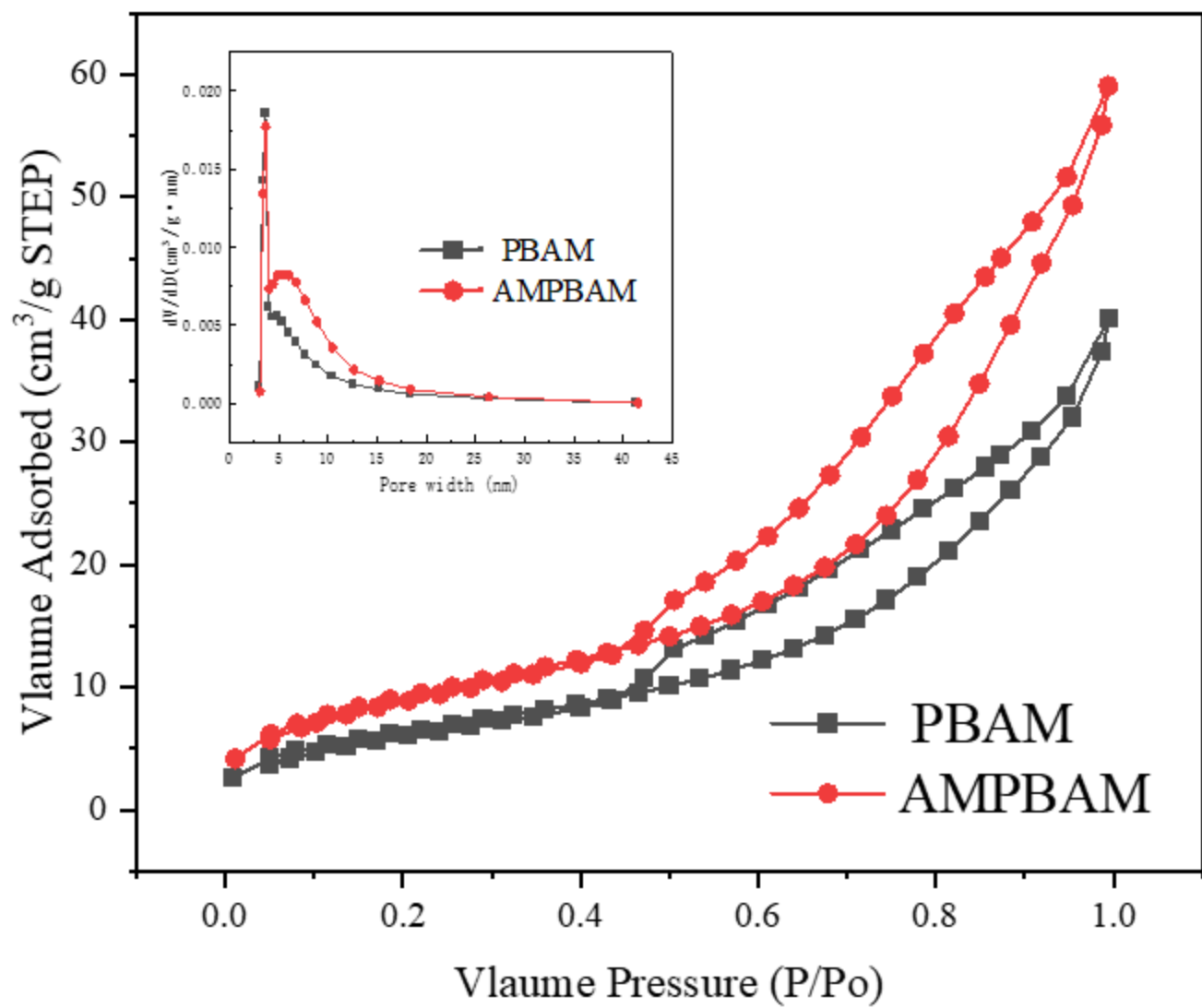


Figure 4

N₂ Adsorption-Desorption Isotherms and Pore Size Distribution Curves of PBAM and AMPBAM

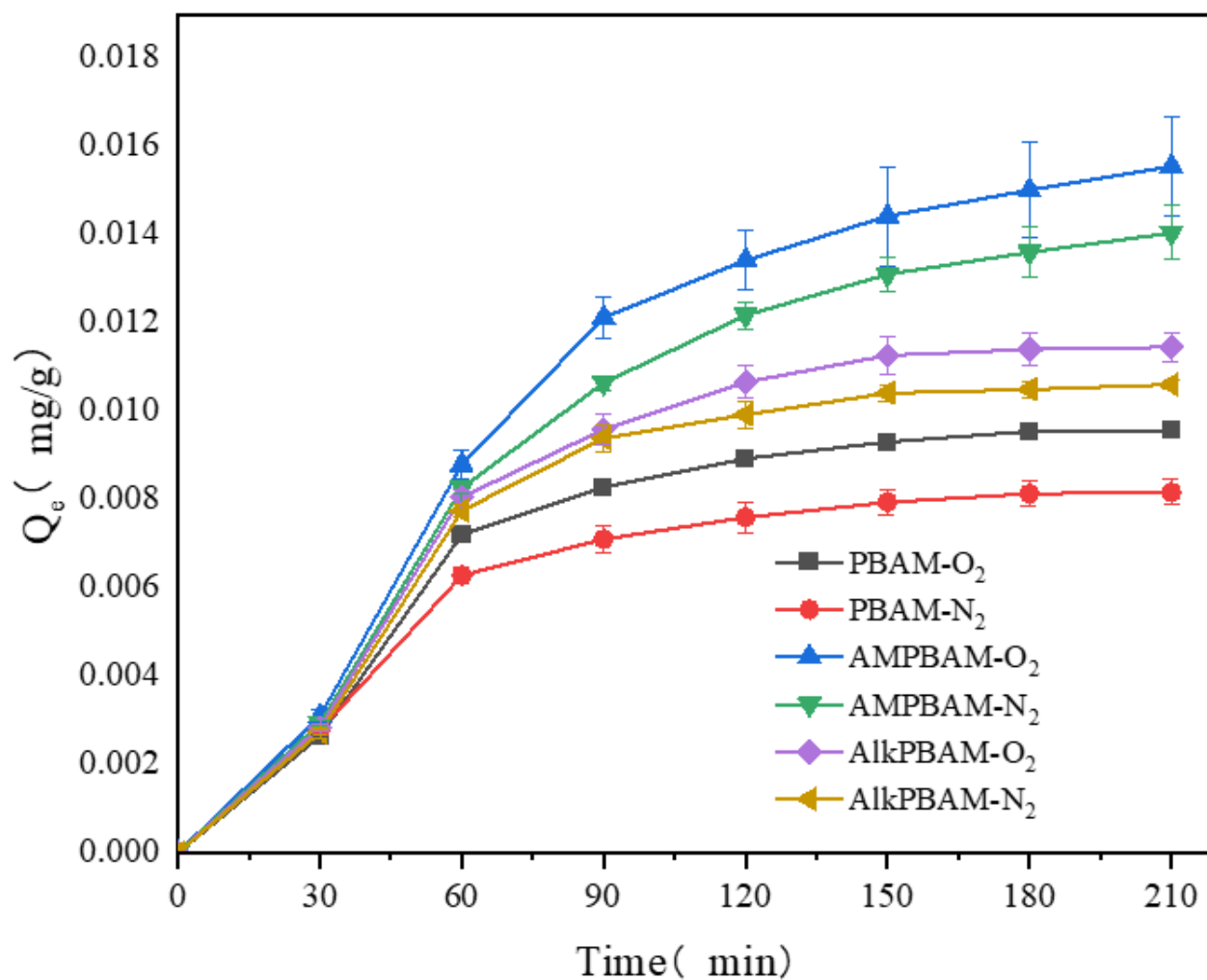


Figure 5

Adsorption Curves of PBAM, AMPBAM, and AlkPBAM for Styrene

AlkPBAM at a Concentration of 50 ppb

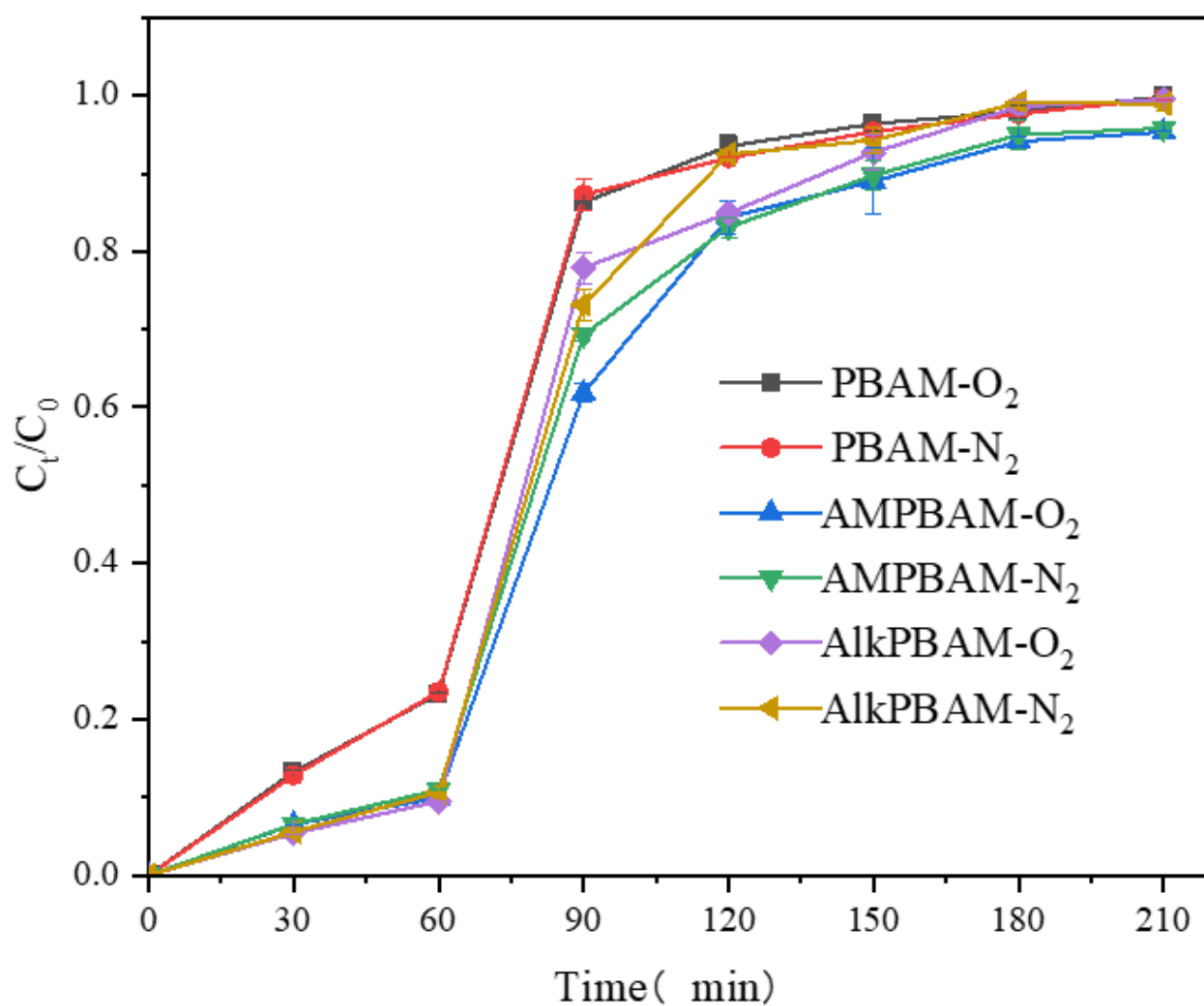


Figure 6

Styrene Adsorption Breakthrough Curve Diagram of PBAM, AMPBAM, and AlkPBAM at a Concentration of 50 ppb

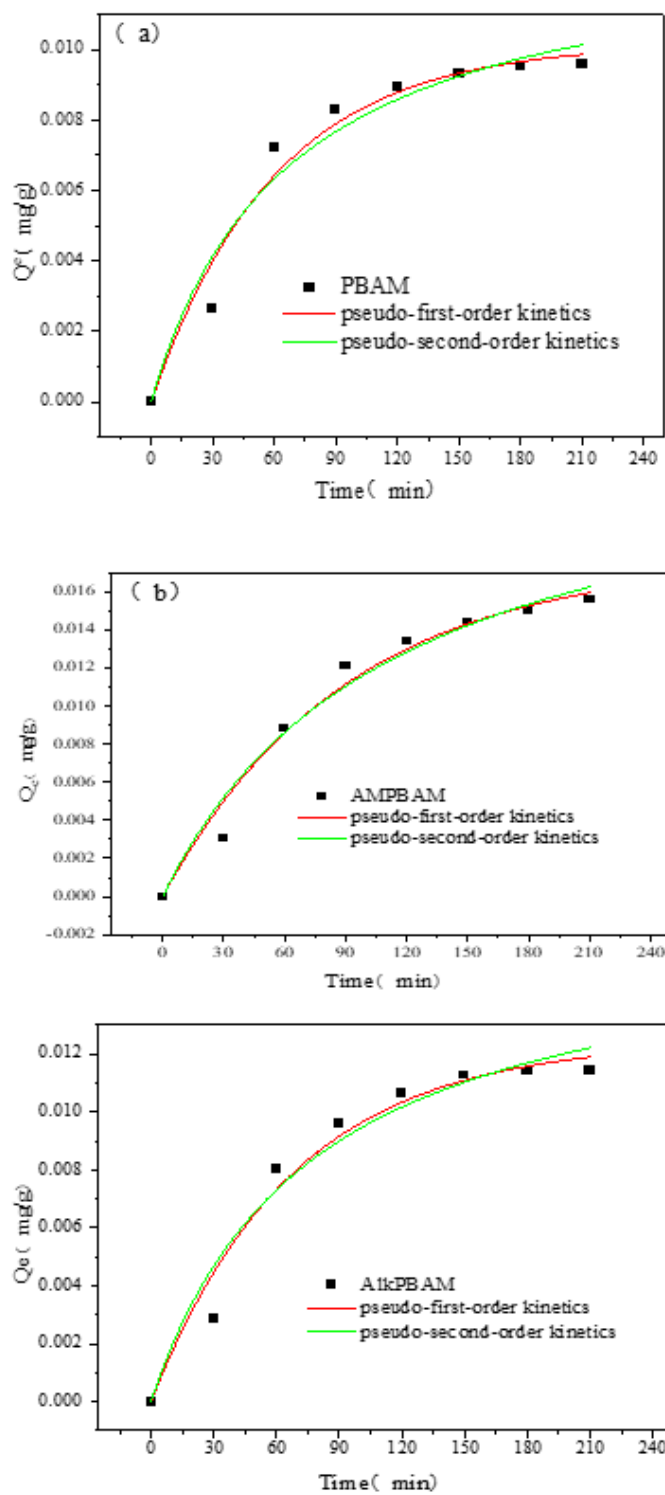


Figure 7

Kinetic Fitting for Styrene Adsorption and Removal—**a**—PBAM, **b**—AMPBAM, **c**—AlkPBAM