

**РЕГЕНЕРАЦИЯ АКТИВИРОВАННОГО УГЛЯ ПОСЛЕ АДСОРБЦИИ КРАСИТЕЛЯ
С ИСПОЛЬЗОВАНИЕМ ПЕРУКСУСНОЙ КИСЛОТЫ.
ЧАСТЬ 2: РОЛЬ ПЕРУКСУСНОЙ КИСЛОТЫ В ОКИСЛИТЕЛЬНОМ ЭТАПЕ**

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Данное исследование посвящено изучению возможности регенерации активированного угля (удаления с его поверхности адсорбированного красителя метиленового синего с использованием надуксусной кислоты). В ходе исследований получены ценные сведения об изменении свойств поверхности активированного угля в процессе регенерации. При изучении адсорбционно-десорбционных равновесий было установлено, что примерно 4,5% изначально адсорбированного метиленового синего десорбируется при промывке, в то время как большая часть (95,5%) остается прочно связанной с активированным углем. Анализ методом инфракрасной спектроскопии с преобразованием Фурье позволил установить, что в процессе регенерации поверхность угля приобретает дополнительные функциональные группы – гидроксильные и карбоксильные. Анализ методом высокоэффективной жидкостной хроматографии подтвердил успешное удаление метиленового синего из жидкой фазы, указывая на высокую селективность системы надуксусных кислот/ Co^{2+} в отношении адсорбированных молекул метиленового синего. Кроме того, анализ методом Брунауэра-Эммета-Теллера показал, что восстановленный уголь сохраняет свои поверхностные характеристики и пористую структуру, с незначительным снижением удельной поверхности и общего объема пор. Эти результаты подчеркивают эффективность надуксусных кислот в процессе регенерации и его потенциал в повышении возможности повторного использования активированного угля в технологических процессах. Данное исследование предоставляет ценную информацию для дальнейших исследований механизма регенерации и оптимизации условий регенерации с использованием надуксусных кислот.

Ключевые слова: надуксусная кислота, активированный уголь, регенерация, окисление, адсорбция

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**REGENERATION OF ACTIVATED CARBON AFTER ADSORPTION
OF DYE USING PERACETIC ACID.
PART 2: ROLE OF PERACETIC ACID IN THE OXIDATION STEP**

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This study focuses on investigating how peracetic acids can be used to regenerate activated carbon and remove methylene blue. The research provides valuable insights into the process of regeneration and the effectiveness of peracetic acids in revitalizing the activated carbon surface. By conducting desorption experiments, we observed that approximately 4.5% of the initially adsorbed methylene blue was released into the washing solution, while the majority (95.5%) remained strongly attached to the activated carbon. Through Fourier-transform infrared spectroscopy analysis, it was determined that after undergoing oxidation by peracetic acids, the regenerated carbon surface exhibited the emergence of additional functional groups, such as hydroxyl and carboxyl groups. Liquid phase chromatography analysis confirmed the successful removal of methylene blue from the liquid phase, indicating the high selectivity of the peracetic acids/Co²⁺ system in treating the adsorbed methylene blue molecules. Furthermore, Brunauer - Emmett-Teller analysis demonstrated that the regenerated carbon retained its surface characteristics and porous structure, with only slight reductions in specific surface area and total pore volume. These findings emphasize the effectiveness of peracetic acids in the regeneration process and its potential to improve the reusability of activated carbon in industrial applications. The study provides valuable insights for further research on the regeneration mechanism and the optimization of regeneration conditions utilizing peracetic acids.

Key words: peracetic acids, activated carbon, regeneration, oxidation, adsorption

INTRODUCTION

Activated carbon (AC) is widely utilized as an adsorbent in various industrial processes to eliminate contaminants from liquid and gas streams. However, the adsorption capacity of activated carbon diminishes over time as adsorbed compounds accumulate on its surface. To address this issue and improve the reusability of activated carbon, regeneration techniques have been developed, with peracids proving effective for the regeneration process [1].

The mechanism of regeneration using peracids involves a series of steps that facilitate the removal of

adsorbed compounds and the rejuvenation of the activated carbon surface. The regeneration mechanism using peracids involves key steps for rejuvenating activated carbon. Peracids, such as peracetic acids (PAA) or hydrogen peroxide (H₂O₂), act as potent oxidizing agents, generating reactive oxygen species (ROS) including hydroxyl radicals ($\cdot\text{OH}$). In contrast to conventional oxidants like H₂O₂, PAA possesses a less robust O–OH bond, thereby yielding more potent radicals for the purpose of targeting pollutants [2, 3]. Beyond this, PAA boasts additional benefits, including straightforward preparation and the absence of any toxic by-products during usage. Consequently, PAA emerges as a

compelling and optimal alternative to H_2O_2 as an innovative oxidant within advanced oxidation processes (AOPs). These ROS then react with adsorbed compounds, initiating their oxidation through hydrogen abstraction and electron transfer. As a result, complex organic compounds are broken down into smaller fragments, facilitating their removal from the carbon surface. The oxidation process also weakens the interaction between the adsorbed compounds and the carbon surface, promoting their desorption. By removing the adsorbed compounds and treating the activated carbon surface, the regeneration process rejuvenates the adsorption sites, restoring the carbon's adsorption capacity for future cycles [4, 5]. Numerous research studies have investigated the mechanism of regeneration using peracids. For instance, explored the impact of thermal regeneration conditions and inorganic compounds on the characteristics of activated carbon used in power plants conducted a preliminary evaluation of alternative regenerant solutions for the chemical regeneration of granular activated carbon focused on the advanced oxidation of aromatic organic compounds using cobalt/peracetic acid systems [6, 7].

In Part 1, we conducted a comprehensive study on the impact of different factors, including the nature of metal ions, pH levels, regeneration time, and PAA concentration, on the efficacy of regenerating activated carbon following the adsorption of methylene blue (MB) [8]. In this Part 2, we will delve into the role of PAA in both the oxidation of MB in solution and the oxidation of the adsorbed MB molecules on the surface of AC. The information obtained will serve as a valuable source for further research and elucidation of the mechanism involved in the regeneration process of activated carbon using PAA.

EXPERIMENTS

The adsorption of MB and regeneration experiments on activated carbon were conducted in a manner similar to Part 1 of our work.

To investigate the role of PAA in the oxidation of MB and its impact on the activated carbon surface, the following procedures were performed: Initially, 1 g of fully saturated activated carbon (AC-s) was immersed and continuously stirred in 250 mL of distilled water at a temperature of 25 °C. Subsequently, the activated carbon was separated from the liquid phase after washing (AC-w). The concentration of MB in the liquid phase was monitored using UV-VIS measurement techniques. The structural characteristics of the activated carbon before and after adsorption, as well as after washing (AC-w), were analyzed using FT-IR spectroscopy and the adsorption-desorption isotherm determined by nitrogen (BET) at a temperature of 77 K.

To assess the degradation products of MB by the PAA/ Co^{2+} system, high-performance liquid chromatography (HPLC) was employed. The composition of the PAA/ Co^{2+} solution following the regeneration of activated carbon was determined using HPLC coupled with UV-Vis spectroscopy analysis on a Shimadzu PDA detector, utilizing a C18 reverse-phase column. The mobile phase contains CH_3OH and a buffer mixture of acetate (0.01 M CH_3COOH , 0.01 M CH_3COONa) in a volume ratio of 60:40. The measurement conditions included a flow rate of 1.0 mL/min, a pressure of 120 bar, a column temperature of 40 °C, and measurement wavelengths of 658 nm and 280 nm. The 658 nm wavelength is indicative of colored compounds, while the 280 nm wavelength characterizes aromatic compounds containing functional groups.

RESULTS AND DISCUSSIONS

The dependence of MB concentration in the AC-s washing water on the washing time is presented in Fig. 1.

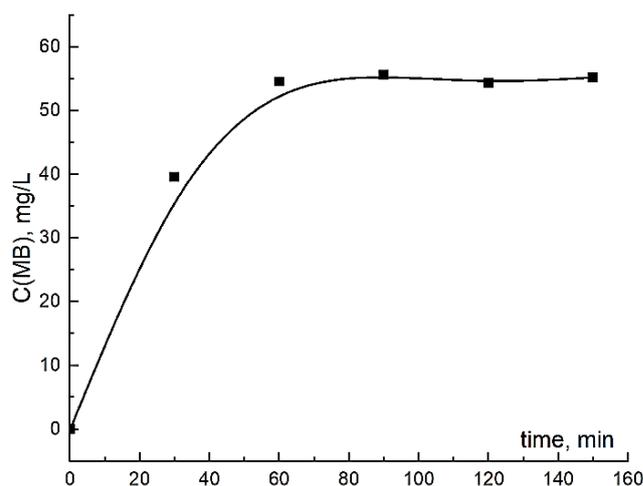


Fig. 1. MB concentration in AC-s washing solution over time
Рис. 1. Концентрация MB в растворе при промывке AC-s со временем

Fig. 1 illustrates that MB was detached from the surface of the activated carbon. After approximately 60 min, the MB concentration in the rinse solution reached equilibrium at approximately 54.5 mg/g. The adsorption capacity of the activated carbon was determined to be approximately 303 mg/g. Consequently, the percentage of MB mass (wt%) released into the washing water solution, compared to the amount of MB adsorbed on the activated carbon, was calculated as 4.5%. This indicates that roughly 4.5% of the physically adsorbed and weakly bound MB on the activated carbon surface was separated into the water, while the remaining 95.5% of MB still remained on the activated carbon.

The findings depicted in Fig. 1 provide valuable insights into the desorption behavior of MB from the surface of the AC-s. The observed trend indicates that, over time, MB molecules are gradually released from the AC-s and enter the washing solution. After approximately 60 min, the desorption process reaches equilibrium, as evidenced by the steady MB concentration in the washing solution, which stabilizes at around 54.5 mg/L. To put this desorption phenomenon into perspective, it is essential to consider the adsorption capacity of the activated carbon, which was previously determined in Part 1 of our study as 303 mg/g. This value represents the maximum amount of MB that the activated carbon can adsorb and retain on its surface under optimal conditions. By calculating the percentage of MB desorbed from AC-s into the washing solution relative to the initial adsorbed MB on the activated carbon, using the equation: $\frac{54.5 \text{ mg/L} \times 0.25 \text{ L}}{303 \text{ mg}}$, we find

that a small fraction of the MB molecules, roughly 4.5%, which were initially physically adsorbed and weakly bound to the surface of the activated carbon, have now detached from the AC surface and entered the washing solution. Meanwhile, the majority of the MB, approximately 95.5%, remains firmly retained on the activated carbon, maintaining its strong adsorption affinity.

To get insights into the chemical composition and surface characteristics of the activated carbon before and after the washing process, as well as the changes in the functional groups and surface properties of the activated carbon after the regeneration processes, FTIR analysis was performed. Fig. 2a presents the results of the FTIR spectra for AC-s and AC-w and Fig. 2b illustrates the FTIR spectra of three samples: the initial activated carbon (AC), activated carbon regenerated by PAA (AC-rPAA), and activated carbon regenerated by PAA/Co²⁺ (AC-rPAA/Co).

The results presented in Fig. 2b reveal that the utilization of strong oxidizing agents like PAA leads to the emergence of additional functional groups on the surface of the regenerated carbon. By referring to the research conducted by Bansal et al. [9], it becomes possible to identify specific functional groups present on the carbon surface through characteristic infrared absorption peaks associated with each surface group.

The peak intensification observed around the wavenumber of 3448 cm⁻¹ can be attributed to the presence of O-H bonds within hydroxyl groups. At the wavenumber of 2931 cm⁻¹, the appearance of a peak signifies the existence of C-H bonds within aromatic rings. However, following the oxidation by PAA, a new absorption peak appears at 1727 cm⁻¹, indicating

the presence of the C=O double bond in carboxyl groups [10,11]. Additionally, at the wavenumber of 1627 cm⁻¹, the presence of C=C bonds within aromatic rings and C=O bonds within carboxyl, ketone, lactone, and aldehyde groups is observed. The peak at 1380 cm⁻¹ corresponds to C-O bonds present in phenol and alcohol groups [12].

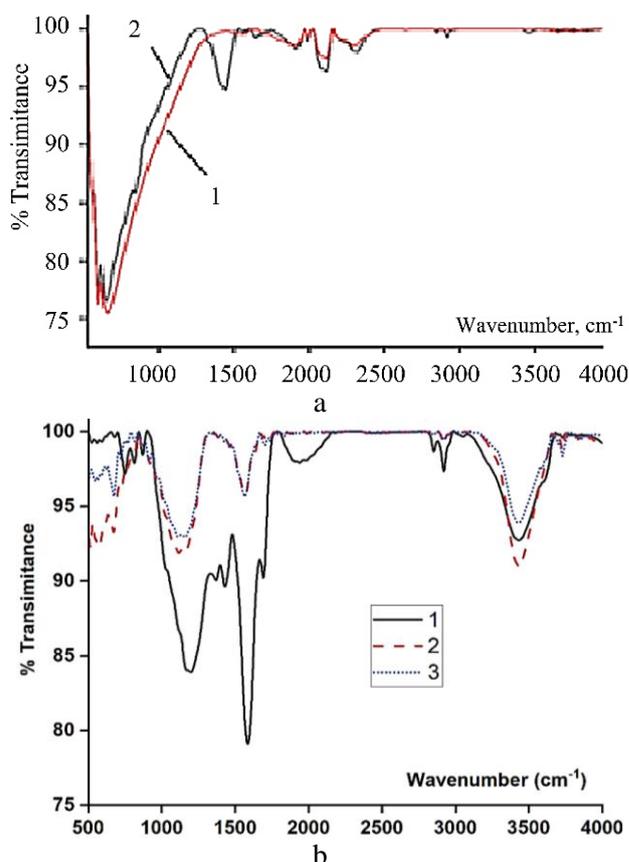


Fig. 2a. FT-IR spectra of saturated activated carbon with MB (AC-s) and AC-s after washing (AC-w)

(1): (AC-s); (2): (AC-w)

Рис. 2а. Фурье-ИК спектры активированного угля насыщенного с МВ (АС-с) и АС-с после промывки (АС-в); (1): (АС-с); (2): (АС-в)

Fig. 2b. FT-IR spectra of original activated carbon (AC), AC regenerated by PAA (AC-rPAA), and AC regenerated by PAA/Co²⁺ (AC-rPAA/Co)

Рис. 2б. Фурье-ИК исходного активированного угля (АС), активированного угля, регенерированного с использованием PAA (АС-рPAA), и активированного угля, регенерированного с использованием PAA/Co²⁺ (АС-рPAA/Co) (1): АС; (2): АС-рPAA; (3): АС-рPAA/Co

Moreover, when subjected to oxidation by PAA/Co²⁺, further peaks become apparent in the infrared spectrum. The range around the wavenumber of 831 cm⁻¹ corresponds to CO₃²⁻ vibrations, while the band at 962 cm⁻¹ describes the δ(CoOH) bonding, and the range around 518 cm⁻¹ represents the vibration of ρ(CoOH) [13].

To evaluate the role of PAA in oxidizing MB in aqueous solution, an HPLC analysis was conducted. Fig. 3 displays an HPLC chromatogram of the MB stock solution, which has a concentration of 8.5 mg/L. The chromatogram was measured at 658 nm, revealing a retention time of 1.724 min and a peak area of 133448.

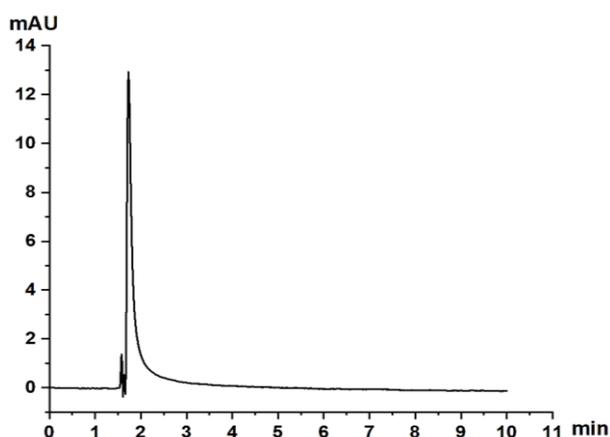


Fig. 3. HPLC chromatogram of the stock MB solution at a wavelength of 658 nm

Рис. 3. Хроматограмма ВЭЖХ исходного раствора метиленового синего при длине волны 658 нм

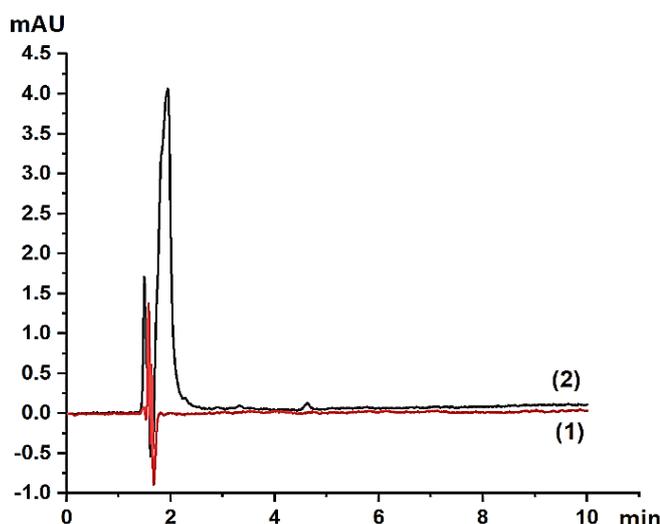


Fig. 4. HPLC chromatograms at wavelengths of 280 nm and 658 nm of the regenerated carbon immersion solution (1): 658nm; (2): 280nm

Рис. 4. Хроматограммы ВЭЖХ при длинах волн 280 нм и 658 нм раствора, полученного в результате иммерсии регенерированного угля

The HPLC chromatograms of the regenerated activated carbon immersed in PAA/Co²⁺ solution at wavelengths of 280 nm and 658 nm are presented in Fig. 4. The analysis of the chromatogram at a wavelength of 658 nm did not observe the characteristic peak

of the MB solution at a retention time of 1.724 min, nor did it observe any additional peaks within the retention time range from 3.0 to 9.0 min. A small peak appearing in the retention time range of 2.2 to 2.8 min is a noise signal resulting from the solvent change between the mobile phase and the sample solution (referred to as the dead volume peak of the column) [14,15]. This indicates that the processed product no longer contains chromophoric groups, specifically, the MB has been nearly completely treated.

Fig. 3 and 4 provide evidence that the MB present in the liquid phase has been effectively eliminated, suggesting that the success of the regeneration process is not solely dependent on the reactivity of the adsorbent and the diffusion of •OH radicals within the pores of the activated carbon [16, 17]. The oxidation or decomposition of MB in the liquid phase also plays a significant role. The reduction reaction between the saturated activated carbon and PAA/Co²⁺ occurs simultaneously in both the liquid phase and on the surface of the activated carbon.

The oxidizing radicals react with MB molecules in the liquid phase and simultaneously penetrate the solid phase, where they interact with MB adsorbed on the surface of the activated carbon [18]. Remarkably, the HPLC chromatogram of the solution after regeneration exhibits only two peaks corresponding to MB, indicating the absence of intermediate products such as quinol, catechol, and hydroquinol, which are typically associated with PAA reactions [19, 20]. The reduction reaction takes place extensively, leading to the complete release of all adsorbed MB molecules. This process generates CO₂ and H₂O without forming any intermediate compounds, all while maintaining the integrity of the activated carbon structure. This observation confirms the high selectivity of the reaction between PAA/Co²⁺ and MB adsorbed on activated carbon, making it a convenient and environmentally friendly approach.

The BET results of the original and third-regeneration samples of activated carbon are presented in Fig. 5 and Table, revealing that the initial activated carbon possesses a well-developed specific surface area, with a BET surface area of 1157 m²/g. The majority of the surface area is occupied by small-sized mesopores, accounting for 96% of the total surface area, while the small-sized mesopore volume represents 91% of the total pore volume. Upon adsorption of the dye molecule MB, the BET surface area of the carbon decreases to 657.7 m²/g (approximately 56.8% of the surface area prior to adsorption), and the mesopore volume reduces to 0.238 cm³/g (approximately 0.65% of the original mesopore volume).

Table

Properties of the original activated carbon (AC), saturated activated carbon with MB (AC-s) and activated carbon after the third regeneration by PAA/Co²⁺ (AC-r3)

Таблица. Свойства исходного активированного угля (AC), насыщенного активированного угля метиленовым синим (AC-s) и активированного угля после третьей регенерации с использованием PAA/Co²⁺ (AC-r3)

	BET Surface Area (m ² /g)	Plot Micropore Area (m ² /g)	Plot External Surface Area	Pore Volume (cm ³ /g)	Pore Size (nm)	
					Adsorption average pore width (cm ³ /g)	Desorption average pore width (cm ³ /g)
AC	1157.1	779.516	377.584	0.36408	4.2696	3.9695
AC-s	675.7	476.417	199.333	0.23856	4.1226	3.8768
AC-r3	1024.12	719.217	304.9	0.3368	4.1993	3.8929

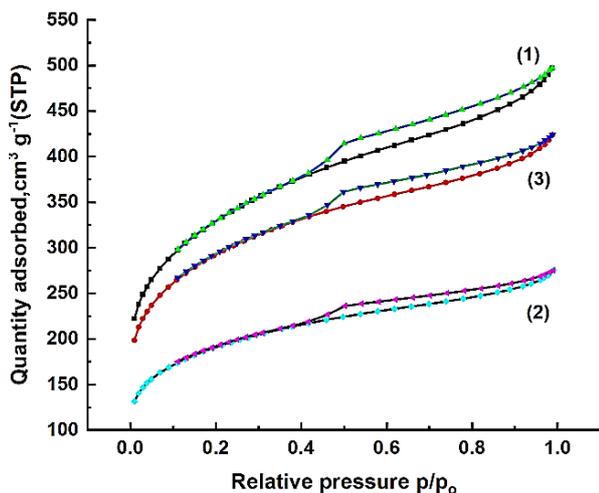


Fig. 5. Adsorption-desorption isotherms of nitrogen on AC, AC-s, and AC-r3. (1): AC; (2): AC-s; (3): AC-r3

Рис. 5. Изотермы адсорбции-десорбции азота на AC, AC-s и AC-r3

Furthermore, the results demonstrate that after oxidation, the specific surface area (S_{BET}) and total pore volume of the material samples decrease, albeit insignificantly, to 1024 m²/g (89% compared to the initial value). In comparison to the original activated carbon sample, the third-regeneration sample exhibits an 11.2% reduction in specific surface area and a 10.1% decrease in total pore volume. The decrease in specific surface area after regeneration could be attributed to the presence of residual MB that has not been completely decomposed. Meanwhile, the average pore size width remains almost unchanged, with only a slight decrease of approximately 0.07 nm. This indicates that the porous structure of the activated carbon largely retains its surface physicochemical properties after oxidation, and the regeneration process of activated carbon does not significantly affect the porous nature of the material.

CONCLUSIONS

In this study, the regeneration of activated carbon using peracetic acids (PAA) was investigated for

the removal of methylene blue (MB). The results revealed important insights into the regeneration mechanism and the effectiveness of PAA in rejuvenating the activated carbon surface. The desorption behavior of MB from the carbon surface was observed, with approximately 4.5% of the initially adsorbed MB being released into the washing solution, while the majority (95.5%) remained firmly retained on the activated carbon. FT-IR spectroscopy analysis showed the emergence of additional functional groups, such as hydroxyl groups and carboxyl groups, on the regenerated carbon surface after oxidation by PAA. HPLC analysis confirmed the successful elimination of MB from the liquid phase, indicating the high selectivity of the PAA/Co²⁺ system in treating adsorbed MB molecules. The BET analysis demonstrated that the regenerated carbon retained its surface physicochemical properties and porous nature, with only slight reductions in specific surface area and total pore volume. These findings highlight the effectiveness of PAA in the regeneration process and its potential for improving the reusability of activated carbon in industrial applications. The study provides valuable information for further research on the regeneration mechanism and the optimization of regeneration conditions using peracetic acids.

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CONFLICT OF INTEREST

The authors declare the absence a conflict of interest warranting disclosure in this article. Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

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