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ВОССТАНОВЛЕНИЕ АКТИВИРОВАННОГО УГЛЯ ПОСЛЕ АДСОРБЦИИ КРАСИТЕЛЯ С ИСПОЛЬЗОВАНИЕМ ПЕРУКСУСНОЙ КИСЛОТЫ. ЧАСТЬ 1: ВЛИЯНИЕ МЕТАЛЛИЧЕСКИХ ИОНОВ, pH, ВРЕМЕНИ РЕГЕНЕРАЦИИ И КОНЦЕНТРАЦИИ КИСЛОТЫ

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Активированный уголь нашел широкое применение в различных отраслях промышленности для очистки сточных вод от загрязнений. Однако с течением времени его эффективность снижается, поскольку активные центры на поверхности угля постепенно насыщаются загрязняющими веществами, присутствующими в воде. Поэтому основная цель данного исследования заключается в поиске потенциального метода восстановления насыщенного метиленовым синим (МВ) активированного угля с использованием перуксусной кислоты (РАА) в качестве окислительного агента. Результаты исследования подтверждают эффективность РАА в разложении молекул МВ. Кроме того, было отмечено, что наличие ионов кобальта (Co^{2+}) действует в качестве катализатора, существенно усиливая процесс окисления. Такой подход позволяет полностью удалить МВ из активированного угля и восстановить его адсорбционную способность, не повреждая структуру угля и не образуя опасных побочных продуктов. Для определения оптимальных параметров процесса восстановления активированного угля исследователи тщательно изучали различные условия. Исследование показало, что наилучшие результаты достигаются при поддержании уровня pH в диапазоне от 6 до 7, использовании концентрации РАА 2,4 мМ, проведении реакции при комнатной температуре и продолжительности реакции 120 мин. При таких оптимальных условиях удалось достичь восстановительной эффективности впечатляющих 98%, что свидетельствует о высокой эффективности предложенного метода восстановления активированного угля, насыщенного метиленовым синим. Исходя из этих обнадеживающих результатов, можно сделать вывод о том, что перуксусная кислота (РАА) обладает огромным потенциалом в качестве окислительного агента для регенерации активированного угля.

Ключевые слова: адсорбция, регенерация, передовые окислительные процессы, РАА, активированный уголь

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REGENERATION OF ACTIVATED CARBON AFTER ADSORPTION OF DYE USING PERACETIC ACID. PART 1: EFFECTS OF METAL IONS, pH, REGENERATION TIME, AND ACID CONCENTRATION

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Activated carbon is commonly used in different industries to remove pollutants from wastewater. However, the efficiency of activated carbon decreases over time due to the saturation of the adsorption sites with the pollutant. This study explores a potential method for regenerating activated carbon that has been saturated with methylene blue (MB) using peracetic acid (PAA) as the oxidation agent. The results demonstrate that PAA can effectively degrade the dye molecules, while cobalt ions (Co^{2+}) can act as catalysts to enhance the oxidation process. The method can remove MB from the activated carbon and restore its adsorption capacity without causing any harm to the carbon structure or releasing hazardous byproducts. The study investigates different conditions to determine the optimal parameters for the activated carbon regeneration process. The optimal conditions for the regeneration process include a pH of 6-7, PAA concentration of 2.4 mM, room temperature, and a reaction time of 120 min. These conditions yielded a regeneration efficiency of 98%. The findings suggest that PAA is a promising oxidative agent for regenerating MB-saturated activated carbon.

Key words: adsorption, regeneration, advanced oxidative processes, PAA

INTRODUCTION

Activated carbon is an effective adsorbent material for organic compounds such as phenol, xylene, ethylene glycol, organic colorants, etc. in aqueous media. However, its reusability is limited by the difficulty of regeneration. Various regeneration methods have been studied, including thermal, steam, biological, and chemical methods [1-3]. The thermal method involves three stages: (1) drying at 105 °C, (2) pyrolysis in inert gas, and (3) gasification of the remaining organic compounds. Stages 2 and 3 are usually performed at around 800 °C, with an additional oxidizing agent (steam or CO gas) in stage 3 [4, 5]. The drawback of this method is that it consumes a lot of energy to evaporate water in stage 1 and to heat up the system in stages 2 and 3. Moreover, it causes some loss of activated carbon (5-10%) due to oxidation in stage 3. The steam regeneration method operates at 400-600 °C [6]. This method also requires high complexity and energy costs. The biological method uses microbes to degrade the organic compounds adsorbed on the carbon [7]. However, this method has a long regeneration time and can reduce the porosity of the carbon due to bacterial attachment [8, 9].

Besides the three methods mentioned above, the chemical regeneration method has attracted much attention because of its fast and cost-effective features. This method uses chemical agents to desorb the pollutants and renew the surface of the activated carbon. Depending on the type of the pollutants, the agents can be acids, bases, or organic solvents [10]. Among the chemical methods, advanced oxidation processes (AOPs) have great potential for regenerating activated carbon due to the high reactivity of free radicals, especially hydroxyl radicals (OH•) [11, 12]. Compared to conventional oxidants such as H_2O_2, peracetic acid (PAA) has a weaker O-OH bond, which generates more powerful radicals to oxidize contaminants [13]. Moreover, PAA has advantages such as easy preparation and no toxic by-products [14-16]. Therefore, PAA is considered as a promising novel oxidant that can work well with H_2O_2 in AOPs. Furthermore, transition

metal ions can act as effective catalysts to activate PAA and produce active radicals that can remove persistent contaminants [17-19]. This study presents the regeneration of activated carbon adsorbed with methylene blue (MB) using peracetic acid (CH_3COOOH) as an oxidizing agent and cobalt as a catalyst.

EXPERIMENTS

Chemicals

In this study, activated carbon (AC) was obtained from Jacobi, Sweden, with a size of 0.63-1 mm, a specific surface area of 1157.1 m^2/g, and an average pore diameter ranging from 3.97 nm to 4.27 nm.

Adsorption capacity of AC for MB

The adsorption experiments were conducted as follows: 1.0 g of AC was added to 250 ml of MB solution with a known concentration and shaken at 25 °C for 72 h at a speed of 150 rpm. The residual concentration of MB in the solution after a certain time was measured by the UV-Vis method at 664 nm. The MB adsorption capacity of AC at equilibrium (q_e, mg/g) and at any time t (q_t, mg/g) was calculated by equations $q_e = \frac{C_0 - C_e}{m} \times V$ (Eq. 1) and $q_t = \frac{C_0 - C_t}{m} \times V$ (Eq. 2), where C_0 is the initial concentration of MB, C_t and C_e are the concentrations of MB at time *t* and at equilibrium (mg/L), V is the volume of the solution (L), and m is the mass of AC (g).

Regeneration of MB-saturated AC by PAA

To prepare the adsorbent, 1 g of granular activated carbon (AC) was dispersed in 250 mL of distilled water at 25 °C. The mixture was continuously stirred and then quickly added to the required amount of PAA/Co^{2+}. At specific time intervals, a small volume of the solution (1 mL) was taken out to determine the concentration of MB. The regeneration process took 120 min. The MB concentration was determined using molecular absorption spectroscopy (UV-VIS) on a Biochrom Libra S60 instrument.

RESULTS AND DISCUSSION

The study on the methylene blue adsorption capacity of activated carbon

The adsorption equilibrium of MB was studied over a range of initial concentrations from 150 mg/L to 2500 mg/L. The results of the equilibrium adsorption capacity (q_e, mg/g) are presented in Table 1.

The adsorption equilibrium was analyzed using the Langmuir and Freundlich adsorption models. The Langmuir model was represented by the equation: $\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{1}{q_m} C_e$ (Eq. 3), and the Freundlich model was represented by the equation: $\lg q_e = \lg K_F + \frac{1}{n} \lg C_e$ (Eq. 4).

The analysis results are shown in Fig. S1 and S2 in the **Supplementary**. The characteristic parameters of the Langmuir and Freundlich models are summarized in Table 2.

Table 1

The equilibrium adsorption capacity (q_e, mg/g) of activated carbon (AC)

Таблица 1. **Равновесная адсорбционная емкость (q_e, мг/г) активированного угля (АУ)**

C_o (mg/L)	C_e(mg/L)	q_e (mg/g)	C_e/q_e
150	0.85	37.29	0.02
300	15	71.25	0.21
500	60	110.00	0.55
1000	225	193.75	1.16
1500	510	247.50	2.06
2000	890	277.50	3.21
2500	1346	288.50	4.67

Table 2

MB adsorption isotherm parameters according to Langmuir and Freundlich adsorption isotherm

Таблица 2. **Параметры изотермы адсорбции МГ по изотерме адсорбции Ленгмюра и Фрейндлиха**

Langmuir adsorption isotherm				Freudlich adsorption isotherm		
q_m (mg/g)	K_L (L/mg)	R_L	R^2	K_F (mg/g)	n	R^2
303	0.0135	0.33	0.99	36	3.37	0.98

The high regression coefficients of the equations for both the Langmuir and Freundlich models indicate that they are well-suited to describe the MB adsorption onto activated carbon. According to the linear Langmuir adsorption isotherm equation, the maximum adsorption capacity of activated carbon for MB is calculated to be 303 mg/g, with an adsorption constant (K_L) of 0.0135 L/mg.

The basic characteristic of Langmuir adsorption can be expressed in terms of the separation coefficient R_L, which is dimensionless and used to predict whether the adsorption process is favorable or unfavorable. If $R_L > 1.0$, the adsorption is unfavorable, while R_L of 1.0 indicates linear isothermal adsorption. If $1 > R_L > 0$, the adsorption is favorable, and R_L of 0 means the process is unfavorable. In this study, the calculated R_L value ranges from 0 to 1, indicating favorable adsorption. Additionally, the value of n in the obtained Freundlich model ranges from 1 to 10, which is also a favorable range for adsorption. Both models suggest that activated carbon is a highly effective adsorbent for MB pigment.

Study on the regeneration of activated carbon adsorbed MB (AC-MB) by peracetic acid in the presence of metal ions

The role of metal ions in PAA activation

The strong oxidizing activity of PAA is attributed to the formation of acetyloxyl radicals ($CH_3COO•$) and acetyl(per)oxyl radicals ($CH_3COOO•$) in the presence of transition metal ions. One proposed mechanism for radical formation involving metal ions, particularly Co^{2+}, was discussed by Kim et al. [20]:

First, •OH and $CH_3COO•$ radicals are generated when CH_3COOOH reacts with Co^{2+} (which is reduced by Co^{2+}):

$$CH_3COOOH + Co^{2+} \rightarrow CH_3COO^- + Co^{3+} + {}^{\bullet}OH \quad (1)$$

$$CH_3COOOH + Co^{2+} \rightarrow CH_3COO^{\bullet} + Co^{3+} + OH^- \quad (2)$$

The Co^{3+} generated is then reduced by PAA, as shown in equation (3):

$$CH_3COOOH + Co^{3+} \rightarrow CH_3COOO^{\bullet} + Co^{2+} + H^+ \quad (3)$$

The generated radicals continue to undergo further transformations:

$$CH_3COO^{\bullet} \rightarrow CH_3^{\bullet} + CO_2 \quad (4)$$

$$CH_3COO^{\bullet} + CH_3COOOH \rightarrow CH_3COOO^{\bullet} + CH_3COOH \quad (5)$$

$$CH_3COOO^{\bullet} \rightarrow HO_2^{\bullet} + CH_2CO \quad (6)$$

$$CH_3COOO^{\bullet} + H_2O_2 \rightarrow HO_2^{\bullet} + CH_3COOH \quad (7)$$

According to this process, Co^{2+} acts as a catalyst, and the acetyloxyl ($CH_3COO•$) and acetyl(per)oxyl ($CH_3COOO•$) radicals are generated more readily than •OH radicals. The acetyloxyl radical decomposes rapidly to produce $CH_3•$ and CO_2. On the other hand, acetyl(per)oxyl reacts rapidly with double bonds and the benzene ring, leading to the destruction of the color carrier and auxiliaries. This mechanism also explains the pH-dependence of PAA activity: when the pH is increased, the reaction equilibrium (3) shifts to the right, leading to the production of more acetyl(per)oxyl radicals and increased activity. In general, higher concentrations of PAA result in more effective regeneration of activated carbon.

To study the effect of catalytic metal ions, AC-MB regeneration experiments were conducted using four metal ions: Co^{2+}, Cu^{2+}, Mn^{2+}, and Fe^{3+}. The results are presented in Fig. 1.

It is interesting to note that the effectiveness of different metal ions in promoting the regeneration of AC-MB by PAA varies. Co^{2+} shows the highest regeneration efficiency, which can be explained by the ability of Co^{2+} to form complexes that facilitate the formation of free radicals. The other metal ions, including Cu^{2+}, Mn^{2+}, and Fe^{3+}, have different levels of regeneration efficiency, with Fe^{3+} having the least effect. These differences may be due to the different properties of the metal ions, such as their electronic configurations and coordination geometries, which can affect their ability to form complexes with PAA and promote the generation of free radicals. Based on the experimental results obtained, Co^{2+} is used as a catalyst for further studies.

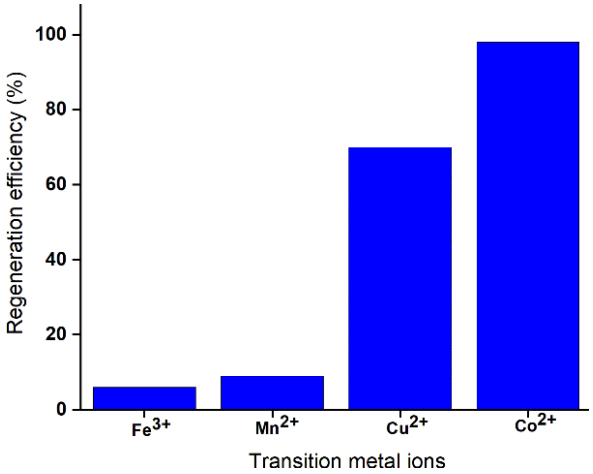

Fig. 1. Effect of a metal catalyst on the ability to regenerate activated carbon after MB adsorption

Рис. 1. Влияние металлического катализатора на способность к регенерации активированного угля после адсорбции МГ

Effect of pH on regeneration of AC-MB by PAA

In this study, the impact of pH on the regeneration of AC-MB by PAA was investigated by testing five pH values (4, 5, 6, 7, and 8). The experimental procedure involved adding the adsorbed AC, which was saturated with MB, to the regeneration solution at a rate of 4 g/L and shaking it at a speed of 150 rpm, 25 °C for 120 min. The pH of the solution was then adjusted using 0.1M NaOH to achieve the desired pH values from 4 to 8. Finally, the AC sample was evaluated for its adsorption capacity and efficiency of adsorption of MB in comparison with the original AC sample after regeneration.

The results presented in Fig. 2 and Fig. 3 demonstrate that the MB adsorption efficiency of the first regenerated AC sample remained consistently high, reaching approximately 98%, when regenerated at a pH range of 6 to 7. However, there was a slight decrease in MB adsorption efficiency when the regenerated pH was increased to 8. Conversely, when the pH was less than 6, the MB adsorption efficiency of the regenerated AC sample decreased rapidly. These findings suggest that near-neutral pH values (6 or 7) yielded better regeneration efficiency compared to low pH (4 or 5) and high pH (8) values. This assertion is corroborated by the adsorption capacity and MB adsorption efficiency of AC after three regeneration cycles (Fig. 5 and 6). Specifically, regenerating the AC at a pH of 6 or 7 resulted in a MB adsorption efficiency of 98% for the regenerated AC, significantly higher

than the original AC, with a maximum adsorption capacity of 299 mg/g.

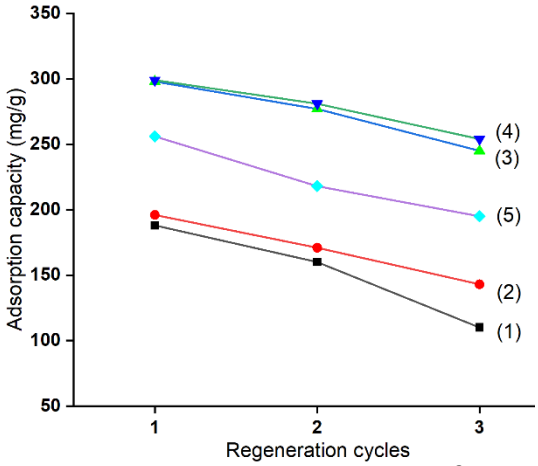

Fig. 2. Effect of pH on AC regeneration by PAA/Co^{2+}: (1): pH=4; (2): pH = 5; (3): pH=6; (4): pH=7; (5): pH=8

Рис. 2. Влияние pH на регенерацию АУ ПАК/Co^{2+}: (1): pH=4; (2): pH = 5; (3): pH=6; (4): pH=7; (5): pH=8

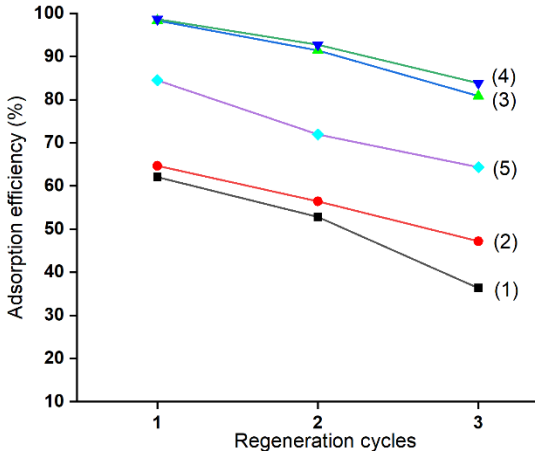

Fig. 3. Effect of pH on MB adsorption efficiency of AC material after 3 regenerations with PAA/Co^{2+} at different pH values: (1): pH=4; (2): pH = 5; (3): pH=6; (4): pH=7; (5): pH=8

Рис. 3. Влияние pH на эффективность адсорбции МГ материалом АУ после 3-х регенераций ПАК/Co^{2+} при различных значениях pH: (1): pH=4; (2): pH = 5; (3): pH=6; (4): pH=7; (5): pH=8

This efficiency only slightly decreased after the third regeneration cycle. However, when the regeneration was conducted at pH 6, the adsorption efficiency was below 70% and dropped significantly after the third regeneration cycle.

Effect of regeneration time

The influence of contact time between saturated AC samples and PAA/Co^{2+} on the regeneration efficiency was also investigated, and the corresponding results are presented in Fig. 4. The findings demonstrated the regeneration rate of the activated carbon following exposure for varying durations of time (30, 60, 90, 120, 150, and 180 min).

As depicted in Fig. 4, the reaction time played a crucial role in the initial stages of regeneration. Within the first 120 min, the regeneration rate escalated from 25% to approximately 98%, reaching its peak at 98% after 120 min. Subsequently, these results exhibited a slight decline, remaining relatively unchanged. This can be attributed to the fact that, after 120 min, all Co^{2+} ions had been reduced to Co^{3+} as per reaction (3), thereby constraining the rate of hydroxyl radical and acetyl (per)oxyl radical ($CH_3COOO•$). Based on this observation, all coal regeneration experiments were conducted for a suitable duration of 120 min.

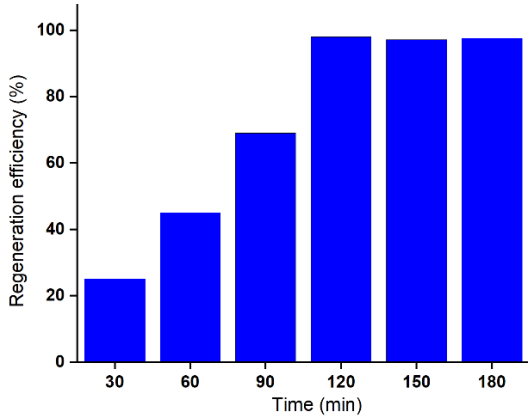

Fig. 4. Effect of regeneration time on regeneration efficiency with pH of 6.6 and a temperature of 25 °C

Рис. 4. Влияние времени регенерации на эффективность регенерации при pH 6,6 и температуре 25 °C

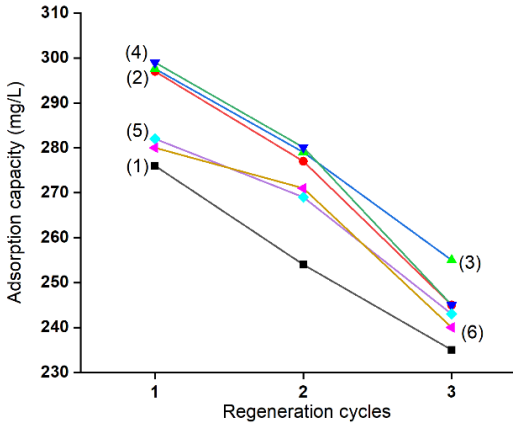

Fig. 5. Effect of PAA concentration in the regeneration process of AC-MB on the adsorption capacity of activated carbon after regeneration: (1): 1.2 mM; (2): 2.4 mM; (3): 3.6 mM; (4): 7.2 mM; (5): 9.6 nM; (6): 14.4 mM

Рис. 5. Влияние концентрации ПАК в процессе регенерации АУ-МГ на адсорбционную способность активированного угля после регенерации: (1) – 1,2 мМ; (2): 2,4 мМ; (3): 3,6 мМ; (4): 7,2 мМ; (5): 9,6 нМ; (6): 14,4 мМ

Effect of concentration of PAA

The impact of peracetic acid concentration was investigated under the specified conditions: MB-saturated AC was introduced into the regeneration solution

at a dosage of 4 g/L, with a shaking speed of 150 rpm and a temperature of 25 °C, pH = 6.6 and the time required to reach adsorption equilibrium was determined. The peracetic acid concentration ranged from 1.2 mM to 14.4 mM, while maintaining a constant pH level in the solution. The obtained results are presented in Fig. 5 and Table 3.

Table 3

Effect of PAA concentration (CPAA, mM) on MB adsorption efficiency of activated carbon after three regeneration cycles

Таблица 3. **Влияние концентрации ПАК (КПАА, мМ) на эффективность адсорбции МГ активированным углем после трех циклов регенерации**

C_{PAA}, mM / Adsorption efficiency, %	1.2	2.4	3.6	7.2	9.6	14.4
1st cycle	91.08	98.01	98.21	98.67	93.06	92.4
2nd cycle	83.82	91.41	92.07	92.44	88.77	89.43
3rd cycle	77.55	80.85	84.15	80.85	80.19	79.2

The results presented in Table 3 demonstrated the influence of PAA concentration on the regeneration ability of activated carbon materials, consequently affecting their adsorption capacity after regeneration. Upon investigating the regeneration process with a PAA concentration of 1.2 mM, the regeneration efficiency was observed to be the lowest. The MB adsorption capacities of the activated carbon were 276 mg/g, 254 mg/g, and 235 mg/g for regeneration 1, regeneration 2, and regeneration 3, respectively. As the PAA concentration for regeneration increased from 1.2 mM to 7.2 mM, the MB adsorption capacity of activated carbon after regeneration increased.

However, when the PAA concentration continued to rise within the range of 9.6 mM to 14.4 mM, the MB adsorption capacity of activated carbon after regeneration decreased. For the first regeneration cycle, the regeneration of activated carbon exhibited remarkably high efficiency, nearly approaching the original efficiency. The adsorption efficiency of the regenerated material was approximately 98% with PAA concentrations of 2.4 mM, 3.6 mM, and 7.2 mM. In the second and third regeneration cycles, the adsorption efficiency declined compared to the first regeneration. Based on the obtained results, it is evident that the regeneration process of AC-MB using PAA concentrations ranging from 2.4 mM to 7.2 mM achieves excellent performance, with very high MB adsorption efficiency. However, further increasing the PAA concentration did not yield any additional improvement in adsorption efficiency. Thus, a PAA concentration of 2.4 mM is the most suitable choice for the regeneration experiments.

CONCLUSION

In conclusion, this research focused on the adsorption capacity of activated carbon for MB and the regeneration of activated carbon adsorbed with MB using PAA in the presence of metal ions. The results showed that activated carbon exhibited a high adsorption capacity for MB, with a maximum adsorption capacity of 303 mg/g. The Langmuir and Freundlich adsorption models were found to accurately describe the adsorption equilibrium of MB onto activated carbon. The study also investigated the role of metal ions in the activation of PAA and found that Co^{2+} showed the highest regeneration efficiency. The effectiveness of different metal ions varied, which could be attributed to their different properties and ability to form complexes with PAA. The pH of the regeneration solution played a crucial role in the regeneration process, with near-neutral pH values (6-7) yielding better regeneration efficiency. The concentration of PAA had a significant impact on the regeneration process, with a concentration range of 2.4 mM to 7.2 mM resulting in excellent regeneration performance and high MB adsorption efficiency. Increasing the PAA concentration beyond 7.2 mM did not further improve the adsorption efficiency. Overall, this research provides valuable insights into the adsorption capacity of activated carbon for MB and the regeneration process using PAA in the presence of metal ions. These findings contribute to the development of efficient and sustainable methods for wastewater treatment and pollutant removal. Further studies could explore the application of this regeneration process on a larger scale and investigate the regeneration of other adsorbents for different pollutants.

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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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