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SORPTIVE AND CATALYTIC PROPERTIES OF ACTIVATED CARBON USED FOR THE REMOVAL OF CRYSTAL VIOLET FROM AN AQUEOUS SOLUTION IN THE PRESENCE OF HYDROGEN PEROXIDE

SORPCYJNO-KATALITYCZNA ROLA WĘGLA AKTYWNEGO W PROCESIE USUWANIA FIOLETU KRYSTALICZNEGO Z ROZTWORU WODNEGO W OBECNOŚCI NADTLENKU WODORU

Abstract: Activated carbons play an important role in the processes of purifying waters, wastewaters and gases. While analyzing the effectiveness of these processes mainly the sorptive properties of the activated carbons are considered, taking no account of their catalytic abilities. According to the data presented in the literature, activated carbons catalyze the decomposition of oxidants such as hydrogen peroxide or ozone creating the hydroxyl radical, which is the strongest oxidating factor. This reaction may be used to oxidate the organic impurities in the aqueous solutions. In the activated carbon – oxidant – organic impurities system most probably both the processes of sorption and catalytic oxidation of the organic compounds take place.

In this paper the effectiveness of removing the crystal violet from the aqueous solution in the presence od various activated carbons and hydrogen peroxide was examined ($C_d = 20 \text{ mg/dm}^3$, $C_{H_2O_2}(1) = 375 \text{ mg/dm}^3$, $C_{H_2O_2}(2) = 3750 \text{ mg/dm}^3$, $m_{ac} = 0.5 \text{ g}$, t = 160 min). In the research the commercial activated carbon WDex, activated carbon WDex oxidated with hydrogen peroxide and activated carbon WDex saturated with crystal violet and regenerated with Fe²⁺/H₂O₂ and Fe²⁺/Ox (sorption – oxidation of adsorbed compounds) were used.

It has been observed that in specified conditions the effectiveness of removing the dye in the presence of carbon and oxidant is greater (72 mg/g) than the sorptive abilities of the activated carbons (34 mg/g). It has been also concluded that the efficiency of the process depends on the type of the activated carbon used, the amount of the hydrogen peroxide and the method of carrying out the process.

It has been also show that the effectiveness of removing crystal violet from the aqueous solution is greater when the process is carried out in the activated carbon – crystal violet – hydrogen peroxide system than in case of removing the dye by the sorption on activated carbon – regeneration of the activated carbon – subsequent regeneration (56 mg/g). The results of the research confirm both the sorptive and catalytic properties of the activated carbons in the analyzed process.

Keywords: activated carbon, oxidation, sorption, crystal violet, hydrogen peroxide

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Activated carbon, characterized by a large surface area and specific chemical properties resulting from a highly porous structure, is commonly used for treating water, wastewater and gases [1]. Its sorptive capacity is reported to be particularly useful in processes involving the removal of impurities from a liquid or gaseous phase. The catalytic properties are considered to be of less importance; however, when combined with the sorptive capacity, their role in the removal process increases. Activated carbon (AC) can be used, for instance, to remove organic compounds by means of the AOP method, where organics are oxidized using hydroxyl radicals OH^{*} generated in the reaction system of hydrogen peroxide and/or ozone in the presence of UV irradiation as well as Fenton's reagent (ie H_2O_2 and Fe^{2+} ions) in the presence or absence of UV irradiation [2–7]. Hydroxyl radicals OH^{*} forming in the reaction system are characterized by a high oxidizing potential (2.7 mV), which makes them the most effective oxidants. As shown in [7] and [8], hydroxyl radicals also form in a reaction system containing hydrogen peroxide or ozone in the presence of activated carbon according to the following equations:

$$\begin{array}{l} AC - OH + H_2O_2 \longrightarrow AC - OOH + H_2O \\ AC - OOH + H_2O_2 \longrightarrow AC - OH + H_2O + O_2 \\ AC + H_2O_2 \longrightarrow AC^+ + OH^- + OH^* \\ AC^+ + H_2O_2 \longrightarrow AC + HO_2^* + H^+ \end{array}$$

The AOP process can be applied to oxidize the organic compounds present in the solution and those adsorbed on the surface of the activated carbon.

An example application of the sorptive and catalytic properties of activated carbon is the removal of organic impurities from textile dye effluents, which contain not only dyes but also hydrogen peroxide used for bleaching [9–11]. Such industrial wastewater can be treated by sorption on activated carbon as well as by oxidation using hydroxyl radicals.

In this analysis, the sorptive and catalytic properties of activated carbon were used for removing crystal violet from an aqueous solution by sorption and oxidation with hydrogen peroxide in the presence of virgin and regenerated WD-extra carbons. The regeneration of activated carbon saturated with crystal violet was conducted using classic Fenton's reagent, Fe^{2+}/H_2O_2 , or modified Fenton's reagent, Fe^{2+}/Ox , with the latter containing an oxidant which becomes an alternative source of hydrogen peroxide. Once introduced into a reaction system, the reagent ensures gradual formation and a more effective use of hydroxyl radicals. The aim of the study was to compare the effectiveness of sorption with that of oxidation in removing a dye from a solution. It was also essential to assess the effectiveness of the analyzed method of carbon regeneration and the possibility of carbon reuse.

Methodology

The materials used for the analysis were:

- commercial activated carbon produced by Gryfskand, Hajnowka, PL (WD-extra), commonly used as a sorbent,

– commercial activated carbon subjected to preliminary oxidation (WD-extra $/H_2O_2$),

– commercial activated carbon saturated with crystal violet, which was then regenerated with classic Fenton's reagent, (WD-extra/Fe²⁺/H₂O₂),

– commercial activated carbon saturated with crystal violet, which was then regenerated with modified Fenton's reagent, Ox, (WD-extra/Fe²⁺/Ox).

Sorption of crystal violet on activated carbons

0.1 g, 0.2 g, 0.5 g, 1.0 g and 1.5 g samples of different activated carbons, WD-extra, WD-extra/Fe²⁺/H₂O₂, and WD-extra/Fe²⁺/Ox, were placed in 300 cm³ conical flasks to be treated with an aqueous solution of crystal violet, the concentration of which was $C_{\rm fk} = 20 \text{ mg/dm}^3$. The flasks were shaken for 12 hours, the time being determined on the basis of the sorption kinetics analysis. The sorption isotherms for crystal violet are shown in Fig. 1.

Oxidation of virgin activated carbon with hydrogen peroxide

Accurately weighed 0.5 g samples of maiden activated carbon, WD-extra, were placed in 300 cm³ conical flasks and treated with distilled water and an H_2O_2 solution in the amount necessary to obtain a concentration of 3750 mg H_2O_2/dm^3 . After being stirred vigorously for 160 minutes, the solution over the activated carbon was decanted. The oxidation procedure was repeated twice. Finally, the activated carbon was rinsed with distilled water (4 × 200 cm³). The activated carbon prepared in this way is represented by the symbol WD-extra/ H_2O_2 .

Regeneration of WD-extra activated carbon saturated with crystal violet

After sorption, the WD-extra carbon saturated with crystal violet was separated from the solution. No drying time was selected. The carbon was placed in a beaker of distilled water. 300 mg/dm³ of H_2O_2 or Ox was then introduced at pH = 3–4. Subsequently, iron(II) sulphate(VI) (FeSO₄ · 7H₂O) was added, the weight ratio being Fe²⁺/H₂O₂ or Ox = 1:4. The reactor content was stirred for 20 min at room temperature. The carbon was rinsed with distilled water (5 × 100 cm³). The activated carbons regenerated in this way are represented by the symbols WD-extra/Fe²⁺/H₂O₂ and WD-extra/Fe²⁺/Ox, respectively.

Removal of crystal violet from an aqueous solution by sorption and oxidation

0.5 g samples of different activated carbons, ie WD-extra, WD-extra/H₂O₂, WD-extra/Fe²⁺/H₂O₂, and WD-extra/Fe²⁺/Ox, were placed in 300 cm³ conical flasks and treated with 200 cm³ of the dye solution with a concentration of about 20 mg/dm³ and an H₂O₂.

solution until the concentration 3750 mg H_2O_2/dm^3 was achieved. The samples were stirred vigorously for 160 minutes. The dye concentration in the reaction mixture was determined at time intervals. After 160 minutes, the solution over the activated carbon was decanted. The carbon was then treated with a subsequent portion of the crystal violet solution with a concentration of about 20 mg/dm³ and the hydrogen peroxide solution. The procedure was repeated many times until the decolourization efficiency declined considerably.

Analysing the changes in the concentration of crystal violet

The concentration of crystal violet in the aqueous solution was determined using a Marcel Media UV/VIS spectrophotometer with wavelength $\lambda = 590$ nm.

Measuring the pH of the water extract of the activated carbons

1.0 g of carbon was treated with 20 cm^3 of distilled water, shaken for one hour, and stored at room temperature for 24 hours until used to measure the pH of the solution in contact with activated carbon.

Discussion of results

The first stage of the analysis focused on assessing the sorptive efficiency of WD-extra activated carbon used for removing crystal violet from an aqueous solution. As can be seen from Fig. 1, the dye sorption exhibited Langmuir behaviour up to



Fig. 1. Sorption isotherms of crystal violet on maiden and regenerated activated carbons, WD-extra, WD-extra/F e^{2+}/H_2O_2 and WD-extra/F e^{2+}/Ox

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12 mg/g; then it increased and reached a maximum, which was 32 mg/g. The sorptive efficiency did not rise even if the sorption time was increased up to 24 hours.

Activated carbon saturated with a dye becomes waste, which needs to be disposed of properly. One of the methods of disposal is regeneration by chemical oxidation of the adsorbed substances. In this analysis, WD-extra activated carbon saturated with crystal violet was subjected to chemical regeneration using classic or modified Fenton's reagent, Fe^{2+}/H_2O_2 and Fe^{2+}/Ox , respectively. When classic Fenton's reagent was used, the adsorbed substances (ie crystal violet) decomposed in the presence of hydrogen peroxide and Fe^{2+} ions introduced into the system in an acidic environment. On the other hand, during the regeneration with modified Fenton's reagent, hydrogen peroxide formed gradually and directly in the reaction system. In either case, the forming hydroxyl radicals were responsible for the decomposition of the organic substances. However, the decomposition process was more effective when modified Fenton's reagent was applied.

The effectiveness of the regeneration process was determined by studying the sorptive capacities of the regenerated activated carbons, WD-extra/Fe²⁺/H₂O₂ and WD-extra/Fe²⁺/Ox. The static sorption isotherms in Fig. 1 show that the sorptive capacity and the mechanism of sorption on maiden activated carbon, WD-extra, were similar to those obtained for the activated carbon regenerated with Fenton's reagent. The sorption of crystal violet on the regenerated activated carbons exhibited Langmuir behaviour up to 10–12 mg/g, then it increased significantly to 30 mg/g. The isotherms suggest that there was a change in the spatial orientation of the sorbed particles after they had covered a certain surface area of both the maiden and the regenerated activated carbon (using specific active centres) and/or the presence of another type of interactions between the activated carbon and the dye.

The static sorption on the maiden activated carbon, WD-extra, and the regenerated activated carbons, WD-extra/Fe²⁺/H₂O₂ and WD-extra/Fe²⁺/Ox, shows that sorption is an effective method for removing crystal violet from an aqueous solution. The results indicate that the procedure applied to regenerate activated carbon saturated with crystal violet was effective and that it allowed recovery and reuse of the sorbent. It should be noted, however, that although the sorption on activated carbons is a suitable method for removing impurities from an aqueous solution, this process requires long contact time of the sorbent with the sorbate. It was thus essential to analyze the behaviour of the crystal violet – activated carbon system after hydrogen peroxide was introduced. Sanchez-Polo et al have pointed out [3] that activated carbon catalyzes the decomposition of hydrogen peroxide to form hydroxyl radicals. Thus, under desired conditions, the solution should decolourize more rapidly.

For this reason, the solution of crystal violet was treated with hydrogen peroxide in the amount sufficient to achieve a concentration of 3750 mg/dm³. The measurement results in Figure 2 show that despite its oxidizing properties, hydrogen peroxide did not cause any changes in the dye concentration. This means that there was no oxidation reaction in the crystal violet – hydrogen peroxide system. When hydrogen peroxide and WD-extra activated carbon ($C_{\rm fk} = 20 \text{ mg/dm}^3$, $C_{\rm H_{2}O_2} = 3750 \text{ mg H}_2O_2/dm^3$, $m_{\rm WD-extra} = 0.5$ g) were introduced into the dye solution, the decolourization efficiency



Fig. 2. Rate of change in the concentration of the subsequent portions of crystal violet in the presence of hydrogen peroxide ($C_{H_2O_2} = 3750 \text{ mg } H_2O_2/\text{dm}^3$) and WD-extra activated carbon

was much higher than during sorption (Fig. 2). Under such conditions, the dye concentration was reduced to 80 % as early as after 20 minutes; the total decolourization of the solution was achieved about 120 minutes after the reaction was initiated. When the same sample of activated carbon was treated with a new portion of crystal violet and hydrogen peroxide twice, the decolourization of the solution was equally effective. This suggests that some oxidizing agent responsible for the decomposition of crystal violet formed in the reaction system. The agent was most probably hydroxyl radicals forming during the reaction between the activated carbon and hydrogen peroxide, and the activated carbon is the reaction catalyst. When the same sample of activated carbon was treated with subsequent portions of the dye and the oxidant, the oxidation process slowed down slightly. Under such conditions, a 50 % reduction in the dye concentration was observed after approximately 80 minutes. Even though there was a decrease in the rate of decolourization of the crystal violet solution, 32 mg/g of crystal violet per 1 g of WD-extra activated carbon was removed by sorption. In the presence of WD-extra carbon and hydrogen peroxide, the amount of the dye removed was as much as 72 mg/g (Table 1). It is important, however, that the process involved a 3.6 % loss of mass of the activated carbon.

As it was necessary to explain the changes in the decolourization rate for the subsequent portions of the crystal violet solution in the presence of activated carbon and hydrogen peroxide, first the carbon was subjected to preliminary oxidation, and then it was used to remove the dye from the solution under the same conditions as in the previous experiments. The results in Fig. 3 show that the changes in the concentration of crystal violet in the subsequent portions of the solution were similar to those obtained for maiden activated carbon, WD-extra, starting with the fourth portion of the solution

Table 1

Methodology	Activated carbon			
	WD-extra	WD-extra/H ₂ O ₂	WD-extra/Fe ²⁺ /Ox	WD-extra/Fe ²⁺ /H ₂ O ₂
Sorption [mg/g]	32		30	30
Amount of dye removed in the presence of H_2O_2 [mg/g]	72	48	56	24
Loss of mass of activated carbon [%] [Δ_m]	3.6	5.3	5.3	10

Comparing the efficiency of activated carbons used to remove crystal violet from an aqueous solution by sorption and upon oxidation with H_2O_2 ($C_{H_2O_2}$ = 3750 mg H_2O_2/dm^3)

being decolourized (see Fig. 2). The results indicate that the preliminary oxidation of the surface of the WD-extra activated carbon by using hydrogen peroxide was not favourable for the dye decomposition. This justifies the high treatment efficiency of virgin activated carbon (WD-extra) in the activated carbon – crystal violet – hydrogen peroxide system as well as the loss of the sorptive and catalytic properties of the carbon because of its contact with hydrogen peroxide. It should be noted that the total amount of the dye removed from the solution was 48 mg per 1 g of the activated carbon and that it was higher than that obtained by sorption on the maiden activated carbon, WD-extra (Table 1). Of importance is also the fact that the oxidation of the dye was accompanied by the oxidation of the carbon matrix, the evidence of which was a 5.3 % loss of mass of the activated carbon.



Fig. 3. Rate of change in the concentration of the subsequent portions of crystal violet in the presence of hydrogen peroxide ($C_{H_2O_2} = 3750 \text{ mg } H_2O_2/\text{dm}^3$) and activated carbon subjected to preliminary oxidation, WD-extra/ $H_2O_2^2$

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The regenerated carbons, WD-extra/Fe²⁺/H₂O₂ and WD-extra/Fe²⁺/Ox, were used to determine how the properties of activated carbon affected the efficiency of removal of crystal violet from an aqueous solution. Figure 1 shows that the sorptive capacities of these carbons were similar to that of virgin carbon, WD-extra. As can be seen from Figs. 4 and 5, the treatment efficiency of the chemically regenerated carbons introduced into the crystal violet $-H_2O_2$ system was similar to that of maiden activated carbon subjected to preliminary oxidation, WD-extra/H2O2, despite the fact that the solution decolourized as a result of the dye decomposition. It should be noted that under predetermined conditions, the total amount of the dye removed in the presence of the WD-extra/Fe²⁺/Ox carbon was 56 mg/g and it was higher than that obtained during sorption. The overall efficiency of the dye oxidation process in the presence of activated carbon regenerated with Fenton's reagent, WD-extra/Fe²⁺/H₂O₂, was considerably lower - only 24 mg/g. The results confirm the previous conclusion that the dye was removed from the solution in the crystal violet $-H_2O_2$ – activated carbon system not by sorption but by oxidation, and the reaction catalyst was the activated carbon. The efficiency of the analyzed process was lower than that achieved in the presence of maiden carbon, WD-extra. It should be noted that the change in the chemical character of the carbon surface due to the contact with the oxidant did not affect its sorptive capacity for crystal violet; however, it reduced its catalytic capacity for the decomposition of hydrogen peroxide and the formation of hydroxyl radicals. Because of the presence of ash in the analyzed carbons, the functional groups, and accordingly, the chemical character of the carbon surface could not be determined. It was possible to establish only the pH of the water extracts of the activated carbons. Thus, the pH of the maiden activated carbon, WD-extra, was basic (8.60), the pH of the water extract of



Fig. 4. Rate of change in the concentration of the subsequent portions of crystal violet in the presence of hydrogen peroxide ($C_{H_2O_2} = 3750 \text{ mg } H_2O_2/\text{dm}^3$) and WD-extra activated carbon saturated with a crystal violet solution, and then regenerated with classic Fenton's reagent

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Fig. 5. Rate of change in the concentration of the subsequent portions of crystal violet in the presence of hydrogen peroxide ($C_{H_2O_2} = 3750 \text{ mg } H_2O_2/\text{dm}^3$) and WD-extra activated carbon saturated with a crystal violet solution, and then regenerated with modified Fenton's reagent

the activated carbon after regeneration with classic Fenton's reagent was acidic (4.0), and, finally, the pH of the water extract of the activated carbon regenerated with modified Fenton's reagent was almost neutral (6.5), despite the fact that the regeneration was performed in a solution with pH 3.0. This suggests that the catalytic removal of crystal violet from an aqueous solution was more effective in the basic environment, whose source can be both the surface of the activated carbon as well as the ash and other substances introduced during the regeneration with modified Fenton's reagent.

It should be noted that the oxidation of crystal violet in the solution was accompanied by a mass loss of the activated carbon. The loss, however, was varied. The smallest loss of mass, ie 3.6 %, was reported for the maiden activated carbon, WD-extra, at the highest efficiency of dye oxidation. The mass losses for WD-extra/H₂O₂ and WD-extra/ Fe²⁺/Ox resulting from the oxidation of crystal violet were comparable (5.3 %). Since the mass loss of the activated carbon regenerated with Fenton's reagent, WD-extra/Fe²⁺/ H₂O₂, reached as much as 10 %, it was impossible to reuse the carbon. The results suggest that the hydroxyl radicals forming in the dye – oxidant – activated carbon system reacted not only with the organic substance present in the solution but also with the carbon matrix, which caused a change in the chemical character of the activated carbon surface and a complete oxidation of the carbon material.

Conclusions

The experimental results show that WD-extra activated carbons are suitable for the sorptive removal of crystal violet from an aqueous solution. Moreover, this sorbent can

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be effectively regenerated using classic or modified Fenton's reagent. The sorptive capacities of the regenerated activated carbons, WD-extra/Fe²⁺/H₂O₂ and WD-extra/Fe²⁺/Ox, were comparable to those of the maiden carbon, WD-extra. It was found that the removal of crystal violet from an aqueous solution using WD-extra activated carbon was more effective in the presence of hydrogen peroxide (72 mg/g) than in its absence (32 mg/g). This indicates that the reaction system contained an oxidizing agent, other than hydrogen peroxide, which was responsible for the decomposition of crystal violet. The hydroxyl radicals formed by the reaction of activated carbon was the reaction catalyst. Another finding was that the oxidation of the dye present in the solution was accompanied by the oxidation of the carbon surface, the result of which was a change in its chemical character. The oxidation of the carbon surface was not favourable, because it caused a reduction in the catalytic capacity of the activated carbon for the decomposition of hydrogen peroxide and the formation of hydroxyl radicals, and also a significant loss of the carbon mass.

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SORPCYJNO-KATALITYCZNA ROLA WĘGLA AKTYWNEGO W PROCESIE USUWANIA FIOLETU KRYSTALICZNEGO Z ROZTWORU WODNEGO W OBECNOŚCI NADTLENKU WODORU

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Abstrakt: Węgle aktywne odgrywają ważną rolę w procesach oczyszczania wód, ścieków oraz gazów. Analizując efektywność tych procesów, bierze się pod uwagę głównie właściwości sorpcyjne węgli aktywnych, pomijając ich zdolności katalityczne. Dane literaturowe wskazują, że węgle aktywne katalizują reakcję rozkładu utleniaczy, takich jak nadtlenek wodoru czy ozon z utworzeniem najsilniejszego czynnika utleniającego jakim jest rodnik hydroksylowy. Reakcja ta z powodzeniem może być wykorzystana do utleniania zanieczyszczeń organicznych w roztworach wodnych. W układzie węgiel aktywny – utleniacz – zanieczyszczenia organiczne, najprawdopodobniej mają miejsce zarówno procesy sorpcji, jak i katalitycznego utleniania substancji organicznych.

W pracy podjęto badania nad efektywnością usuwania fioletu krystalicznego z roztworu wodnego w obecności różnych węgli aktywnych i nadtlenku wodoru ($C_d = 20 \text{ mg/dm}^3$, $C_{H_2O_2}(1) = 375 \text{ mg/dm}^3$, $C_{H_2O_2}(2) = 3750 \text{ mg/dm}^3$, $m_{ac} = 0.5 \text{ g}$, t = 160 min). W badaniach wykorzystano handlowy węgiel aktywny WDex, węgiel aktywny WDex utleniany nadtlenkiem wodoru oraz węgiel aktywny WDex nasycony fioletem krystalicznym i poddany regeneracji za pomocą Fe²⁺/H₂O₂ i Fe²⁺/Ox (sorpcja – utlenianie zaadsorbowanych substancji).

Zaobserwowano, że w zadanych warunkach skuteczność usuwania barwnika w obecności węgla i utleniacza jest większa (72 mg/g) w stosunku do sorpcyjnych zdolności węgli aktywnych (34 mg/g). Stwierdzono również, że wydajność tego procesu zależy od rodzaju zastosowanego węgla aktywnego, jak i od ilości nadtlenku wodoru oraz sposobu realizacji procesu.

Wykazano, że efektywność usuwania fioletu krystalicznego z roztworu wodnego jest większa w przypadku realizacji tego procesu w układzie węgiel aktywny – fiolet krystaliczny – nadtlenek wodoru, w porównaniu do usuwania barwnika poprzez sorpcję na węglu aktywnym – regeneracja węgla aktywnego – ponowna regeneracja (56 mg/g). Uzyskane wyniki badań potwierdzają zarówno sorpcyjne, jak i katalityczne właściwości węgli aktywnych w analizowanym procesie.

Słowa kluczowe: węgiel aktywny, utlenianie, sorpcja, fiolet krystaliczny, nadtlenek wodoru