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APPLYING THE COMBINED PROCESSES OF SORPTION AND OXIDATION TO REMOVE ORGANIC COMPOUNDS FROM AN AQUEOUS ENVIRONMENT USING THE EXAMPLE OF *p*-CHLOROPHENOL

ZASTOSOWANIE POŁĄCZONYCH PROCESÓW SORPCJI I UTLENIANIA DO USUWANIA ZWIĄZKÓW ORGANICZNYCH ZE ŚRODOWISKA WODNEGO NA PRZYKŁADZIE *p*-CHLOROFENOLU

Abstract: The efficiency of the removal of *p*-chlorophenol from an aqueous solution was tested to simulate the elimination of highly-hazardous soluble chloro-organic compounds from the environment. The methods selected to remove this pollutant were the consecutive and simultaneous processes of sorption and oxidation utilizing hydrogen peroxide and Fenton's reagent. Hydrogen peroxide was incapable of oxidizing *p*-chlorophenol, whereas in Fenton-driven oxidation the substance was decomposed immediately. Sorption on activated carbon proved to be an effective method for removing *p*-chlorophenol from an aqueous solution. Moreover, the *p*-chlorophenol-spent activated carbon was successfully regenerated by oxidation of the adsorbed substance applying hydrogen peroxide or Fenton's reagent and then reused as a sorbent. However, the regeneration process involved a considerable loss of mass of the activated carbon due to oxidation. A more effective method of removal of *p*-chlorophenol from an aqueous solution was oxidation with Fenton's reagent or hydrogen peroxide in the presence of activated carbon. Under such conditions, the processes of sorption and oxidation of the organic substance took place both in an aqueous solution and on the surface of the activated carbon, which contributed to the regeneration and reuse of the carbon.

Keywords: activated carbon, oxidation, sorption, hydrogen peroxide, *p*-chlorophenol

Pollution of the environment due to the release of organic compounds is still a serious problem despite the introduction of increasingly restrictive environmental

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standards. The main reasons for such concern are high toxicity of these substances and numerous sources of their emission [1, 2]. Organic pollutants include chloro-organic compounds, which can be found in industrial and municipal waste arising from the activities of the chemical, petrochemical, pharmaceutical, paper-making and other manufacturing industries, agriculture (*eg* crop protection chemicals) and households (*eg* household chemicals). Since most chloro-organic compounds are well-soluble in water, they can easily migrate in the environment, posing a considerable threat to it. It is thus necessary that their emission be limited. In this context, treatment of industrial wastewater containing chloro-organic compounds is particularly important.

Treating industrial wastewater effectively and efficiently requires applying various methods, sometimes combined, because of the highly complex chemical composition of its organic constituents [3]. One of the solutions that much attention has recently been paid to is to apply sorption using activated carbon as a sorbent [4–6] and oxidation, with the latter being frequently carried out using *Advanced Oxidation Processes* (AOPs), where the oxidants are ozone, hydrogen peroxide and Fenton's reagent and the process is performed in the presence or absence of UV irradiation. The common feature of these methods is the formation of hydroxyl radicals in the reaction environment, which are some of the strongest radicals, having an oxidizing potential of 2.7 V [1]. The processes of sorption and oxidation with the AOP methods are highly efficient and effective. However, there may be a problem with disposal of spent sorbents, considerable amounts of oxidants required and a consequent increase in the volume of wastewater to be treated. Combining the two processes into a simultaneous event is being considered as an alternative [7, 8]. In such a case, organic compounds are removed in a two- or one-stage process, *ie* consecutive sorption and oxidation, which involves regeneration of activated carbon, or simultaneous sorption and oxidation, with the latter being a more favourable solution. The literature [7–9] suggests that activated carbon not only acts as a sorbent but also catalyzes the decomposition of oxidants, which contributes to the formation of hydroxyl radicals. Despite numerous studies on the subject [7, 8, 10], it is still unclear how the process proceeds. Some researchers [7] assume that organic compounds are first adsorbed on activated carbon, and then oxidized. Others, however, *eg* [10] claim that the catalytic role of activated carbon is the most significant, as it contributes to the formation of hydroxyl radicals in the reaction environment.

This paper provides a comparative analysis of the course and efficiency of the process of removal of soluble chloro-organic compounds from aqueous solutions, through sorption and oxidation, using the example of *p*-chlorophenol. Activated carbon, WD extra, was used as a sorbent. The process of oxidation was conducted using hydrogen peroxide or Fenton's reagent. The testing involved sorption of *p*-chlorophenol on activated carbon, followed by regeneration of the spent sorbent by means of hydrogen peroxide or Fenton's reagent so that it could be used again. The results were compared with those obtained for *p*-chlorophenol removed from an aqueous solution through simultaneous sorption and oxidation. The process was performed twice, using the same amount of activated carbon.

Materials and methods

The materials used for the analysis were:

– commercially available *granular activated carbon* (GAC), WD extra, produced by Gryfskand, whose physical and chemical parameters, and pore structure are shown in Tables 1 and 2,

– aqueous solution of *p*-chlorophenol with a concentration of 1 g/dm^3.

Table 1

Physical and chemical properties of the WDextra activated carbon [7]

Parameter	Value
Surface area [m^2/g]	1050
Bulk density [g/dm^3]	415
Water adsorption capacity [cm^3/g]	0.80
Mechanical strength [%]	96
Abrasion resistance [%]	0.5
Ash content [%]	21.8
Methylene blue index (MBI)	36
Iodine value [mg/g]	980

Table 2

Capillary pore distribution in WD extra carbon [7]

WD extra	Capillary pore radius [nm]					$\Sigma \Delta V$
	< 1.5	1.5–15	15–150	150–1500	1500–7500	
	Capillary pore volume, ΔV					
[cm^3/g]	0.3048	0.1727	0.1230	0.2716	0.0872	0.9530
[%]	32.0	18.1	12.9	28.5	8.5	100

1. Determining the parameters of the porous structure

The porous structure of the material was assessed by performing low-temperature adsorption of nitrogen (at 77 K). The adsorption and desorption isotherm was established with a volumetric method, using a Fisons Sorptomatic 1900 and a Micromeritics ASAP 2010. The dechlorination efficiency, the iodine value and the methylene blue index were estimated according to DIN 19603, PN-83/C-97555.04, and PN-82/C-97555.03, respectively.

2. Sorption isotherm

The isotherm of sorption of *p*-chlorophenol on activated carbon, WD extra, was determined for a system of 0.5 g of activated carbon and 100 cm^3 of *p*-chlorophenol solution with concentrations ranging from 200 to 1000 mg/dm^3. The time of contact required to achieve the adsorption equilibrium in the *p*-chlorophenol – WD extra system was 4 hours. The sorption was conducted at room temperature.

3. Removing *p*-chlorophenol from a solution

a) Sorption. 1 g samples of activated carbon, WD extra, were placed in conical flasks and treated with 300 cm^3 of an aqueous solution of *p*-chlorophenol with a concentration of 0.8 g/dm^3. The contents were stirred intensively. After 10, 30, 60 and 120 minutes, the sorption was stopped and the amount of *p*-chlorophenol remaining in the solution was determined.

b) Oxidation

– *H_2O_2*. 300 cm^3 of *p*-chlorophenol solution with a concentration of 0.8 g/dm^3 was treated with NaOH to obtain a pH of 8. Then, H_2O_2 was added, with the amount ensuring stoichiometric oxidation of *p*-chlorophenol. The contents were stirred intensively. After 10, 30, 60 and 120 minutes, a sample of the oxidizing mixture was taken and analyzed for the content of *p*-chlorophenol.

– *in Fenton's reaction Fe^{2+}/ H_2O_2*. 300 cm^3 of *p*-chlorophenol solution with a concentration of 0.8 g/dm^3 was treated with H_2SO_4 to obtain a pH of about 4. Subsequently, H_2O_2 and iron(II) sulphate(VI) (FeSO$_4$ · 7H$_2$O) were added, the former in the amount ensuring stoichiometric oxidation of *p*-chlorophenol). The Fe^{2+}/ H_2O_2 ratio by weight (corresponding to the quantity of Fe^{2+}) was 1:5. The contents were stirred intensively. After 10, 30, 60 and 120 minutes, a sample of the oxidizing mixture was taken and the amount of the remaining *p*-chlorophenol was determined.

c) Sorption + oxidation

– *WD extra + H_2O_2*. 300 cm^3 samples of *p*-chlorophenol solution with a concentration of 0.8 g/dm^3 were weighed into conical flasks and treated with NaOH to obtain a pH of 8. Then, 1 g of activated carbon, WD extra, and H_2O_2 were added. The amount of the latter had to be adjusted to ensure stoichiometric *p*-chlorophenol oxidation. The contents of the flasks were shaken intensively. After 10, 30, 60 and 120 minutes, the oxidation was stopped and the amount of *p*-chlorophenol remaining in the reaction mixture was determined.

– *WD extra + Fe^{2+}/ H_2O_2*. 300 cm^3 samples of *p*-chlorophenol solution with a concentration of 0.8 g/dm^3 were placed in conical flasks and treated with H_2SO_4 to obtain a pH of about 4. Subsequently, activated carbon (WD extra), H_2O_2 and iron(II) sulphate(VI) (FeSO$_4$ · 7H$_2$O) were introduced into the solution, the first in the amount of 1 g, the second in the amount ensuring stoichiometric oxidation of *p*-chlorophenol and the third in the Fe^{2+}/H$_2$O$_2$ ratio by weight (corresponding to the quantity of Fe^{2+}) of 1:5. The flasks were then shaken intensively. After 10, 30, 60 and 120 minutes,

oxidation was stopped and the amount of *p*-chlorophenol remaining in the reaction mixture was determined.

4. Regenerating *p*-chlorophenol-spent activated carbon

– *H_2O_2*. 5 g samples of activated carbon, WD extra, saturated with *p*-chlorophenol, were treated with 500 cm^3 of water. NaOH was added to achieve a pH of 8. Then, the solution was treated with H_2O_2, in the amount ensuring stoichiometric oxidation of *p*-chlorophenol. The contents were stirred for 20 minutes at room temperature and then the carbon was rinsed five times using 100 cm^3 of distilled water. The activated carbon regenerated with hydrogen peroxide was represented by the symbol WD extra/H_2O_2.

– *Fe^{2+}/H_2O_2*. 5 g samples of activated carbon, WD extra, saturated with *p*-chlorophenol, were treated with 500 cm^3 of water. The pH-value was reduced to about 4 by adding H_2SO_4. Then, H_2O_2 and iron(II) sulphate(VI) (FeSO$_4$ · 7H$_2$O) were introduced into the reaction system, the former in the amount ensuring stoichiometric oxidation of *p*-chlorophenol. The Fe^{2+}/H_2O_2 ratio by weight (corresponding to the quantity of Fe^{2+}) was 1:5. The contents were stirred intensively for 20 minutes, and then the carbon was rinsed five times using 100 cm^3 of distilled water. The activated carbon regenerated using a Fenton-driven mechanism was represented by the symbol WD extra/Fe^{2+}/H_2O_2.

5. Determining the concentration of *p*-chlorophenol and chloride ions

The concentration of *p*-chlorophenol was determined using a chromatographic method by means of a Thermo Scientific Focus GC gas chromatograph. Prior to that, liquid to liquid extraction of the analyte was conducted using xylene according to the procedure developed by the authors in order to extract *p*-chlorophenol and other by-products of the oxidation taking place in the solution. The analyte recovered from the aqueous solution amounted to 92 %. The quantity of chloride ions, Cl$^-$, was determined by volumetric titration with a voltometer being part of a Behr CL10 AOX analyzer [11, 12].

Discussion of results

The analysis was conducted for activated carbon, WD extra, characterized by a large surface area of 1050 m^2/g, a pore volume of 0.95 cm^3/g, a micropore volume of 0.30 cm^3/g, an iodine value of 990 mg/g, a methylene blue index of 36 cm^3, and a detergent value of 21 mg/g (Tables 1 and 2). These parameters indicate that the selected activated carbon possesses high sorptive capacity. With regard to *p*-chlorophenol, it was 150 mg/g (Fig. 1). The carbon had a low dechlorination index of 2 cm, which was an evidence of good catalytic capacity with regard to the decomposition and reduction of chlorates to chloride ions, and, indirectly, also the decomposition of hydrogen peroxide, which contributes to the formation of hydroxyl radicals.

Activated carbon, WD extra, saturated with *p*-chlorophenol, was regenerated by oxidizing the adsorbed substance using hydrogen peroxide or Fenton's reagent. Prior to
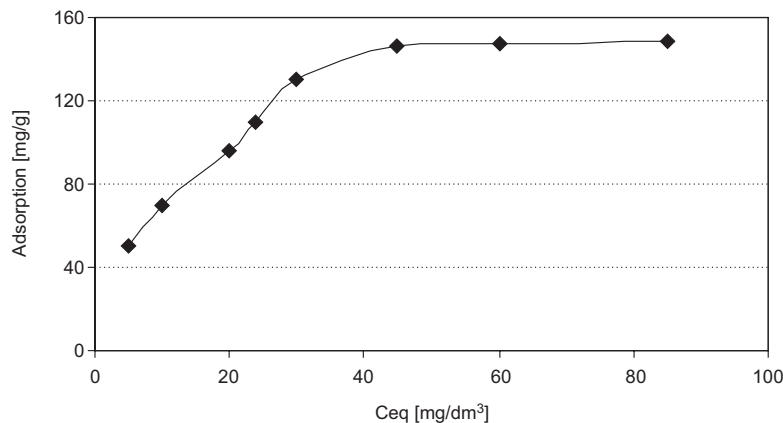
Fig. 1. Isotherm of sorption of *p*-chlorophenol from an aqueous solution on activated carbon, WD extra

that, it was essential to check the oxidation capacity of *p*-chlorophenol using H_2O_2 and Fenton's reagent. As shown in Fig. 2, the Fenton's reaction in which the hydroxyl radicals acted as the oxidizing agent led to immediate decomposition of this substance.
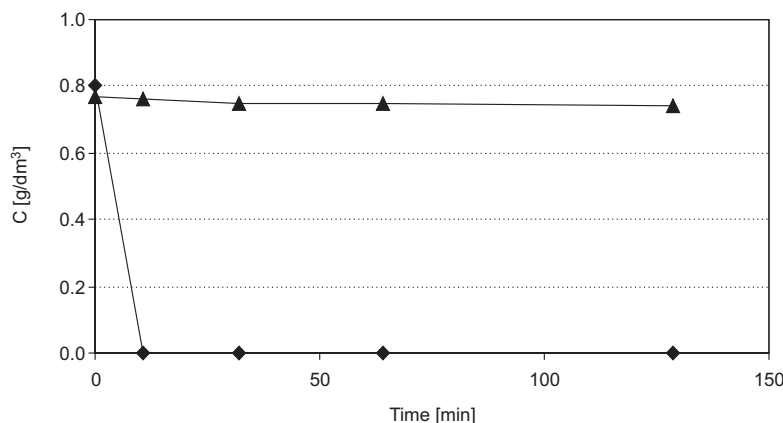

Fig. 2. Changes in the concentration of *p*-chlorophenol for both oxidants in the function of oxidation time

It was found that *p*-chlorophenol was not oxidized in the presence of hydrogen peroxide even though optimal reaction conditions were satisfied. From the literature [8, 11] it is clear, however, that the activated carbon catalyzes the decomposition of H_2O_2, which contributes to the formation of $OH^{\bullet}$ radicals. However, *p*-chlorophenol adsorbed on activated carbon was expected to be decomposed in the presence of Fenton's reagent or hydrogen peroxide, the result of which would be the regeneration of activated carbon. The regeneration efficiency was assessed by analyzing the changes in the concentration of chloride ions in an aqueous solution, being a product of the decomposition of

p-chlorophenol, as well as the sorptive capacity of the regenerated activated carbon (Figs. 3 and 4). The contact of the carbon surface with the adsorbed *p*-chlorophenol resulted in gradual decomposition of *p*-chlorophenol in the oxidant solution, the evidence of which was an increase in the amount of chloride ions. After 10 minutes of contact, the chloride ions released into the solution amounted to 25.5 % of the adsorbed *p*-chlorophenol. After 30 minutes, the decomposition increased to 44 %. Extending the reaction time fourfold gave rise to a slight increase in the degree of decomposition of up to about 48 %, in the case of Fenton's reagent. It should be noticed that the decomposition efficiency of the *p*-chlorophenol adsorbed on carbon was comparable for both oxidants. However, the reaction in a solution was different in nature. The OH$^{\bullet}$ radicals responsible for the oxidation of organic compounds in a WD extra/*p*-chlorophenol/H_2O_2 system were generated in the same way as in the Fenton's reaction. Neither of the oxidants caused complete decomposition of the adsorbed *p*-chlorophenol. The decomposition efficiency could not be improved either by increasing the amount of the oxidant or extending the contact time. A side effect of the oxidation was a considerable loss of the carbon mass (approx. 20 %).
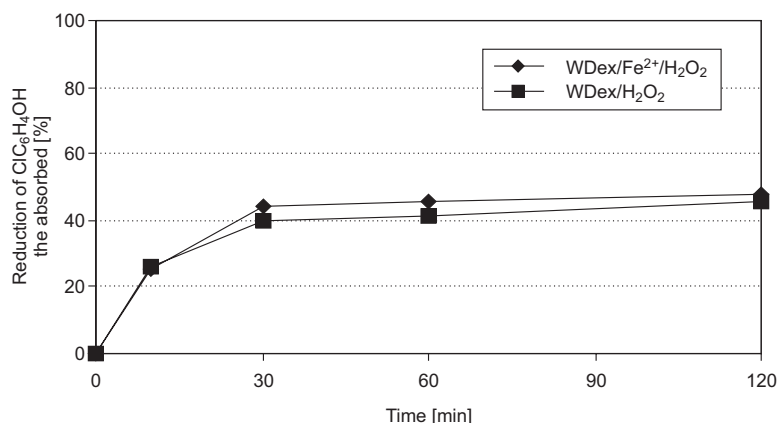

Fig. 3. Efficiency of the reduction of the adsorbed *p*-chlorophenol according to the oxidizing agent

The regenerated activated carbons were reused as sorbents to remove *p*-chlorophenol from an aqueous solution (contact time – 120 min). The results in Fig. 4 show that the removal efficiency of regenerated carbons, WD extra/H_2O_2-I and WD extra/Fe^{2+}/H_2O_2-I, with regard to *p*-chlorophenol removed from a solution was only slightly lower than that of the virgin carbon, despite incomplete decomposition of the originally adsorbed *p*-chlorophenol. The sorptive capacity of the carbon regenerated with H_2O_2 or Fenton's reagent was approx. 105 mg/g, whereas the capacity of the virgin carbon, WD extra, amounted to about 132 mg/g. Since the sorption of *p*-chlorophenol was still high and satisfactory, the carbons were regenerated again under the same conditions, to be reused as sorbents of *p*-chlorophenol. The removal efficiency of the twice-regenerated carbons, WD extra/H_2O_2-II or WD extra/Fe^{2+}/H_2O_2-II, with regard to *p*-chlorophenol,
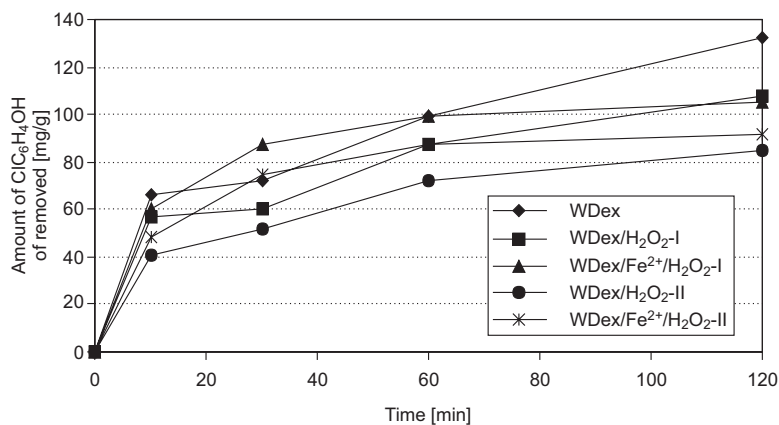
Fig. 4. Efficiency of the removal of *p*-chlorophenol from a solution for virgin carbon, WD extra, and regenerated carbons, WD extra/H$_2$O$_2$ and WD extra/Fe^{2+}/H$_2$O$_2$

was approx. 15% lower than that obtained for the once-regenerated carbon, but still high, reaching approx. 90 mg/g after the same contact time. The results indicate that the consecutive cyclic processes of sorption of *p*-chlorophenol and regeneration of activated carbon are effective and might be considered satisfactory except for the fact that every subsequent regeneration involves a loss of carbon mass. Applying many sorption/regeneration cycles is rather difficult. It should be emphasized that the problem has not been discussed in the literature on the subject.

The simultaneous processes of sorption and oxidation aiming at the removal of organic compounds from aqueous solutions are an alternative to the cyclic consecutive sorption/regeneration method. They allow regeneration of spent carbons and their reuse as sorbents. The analysis was performed using *p*-chlorophenol with a concentration identical to that observed during sorption, activated carbon, WD extra, and H$_2$O$_2$ or Fenton's reagent, the concentration of which was the same as for carbon regeneration. The carbon efficiency to remove *p*-chlorophenol from a solution was assessed by analyzing the changes in the concentration of *p*-chlorophenol and the amount of Cl$^-$ ions, whose presence indicated oxidation of the substance (Table 3 and Fig. 5). In the presence of activated carbon, Fenton's reagent caused almost immediate reduction in the concentration of *p*-chlorophenol in the solution, accompanied by the formation of chloride ions, the amount of which ensured stoichiometric, *ie* total oxidation of *p*-chlorophenol (Table 3). The analysis performed under the same conditions for H$_2$O$_2$ (WD extra+H$_2$O$_2$-1) showed a 31 % loss of *p*-chlorophenol (72 mg/g) after 10 minutes of the reaction. 83 % of this was oxidized (which corresponded to the amount of chloride ions occurring in the WD extra+H$_2$O$_2$/Cl-1 solution), whereas the other 17 % of the removed *p*-chlorophenol was adsorbed on activated carbon. The oxidation to sorption ratio was assessed to be 83/17. Extending the reaction time up to 30 minutes resulted in the removal of 91 mg/g of *p*-chlorophenol, 76 % of which was oxidized and the rest was adsorbed on carbon. The proportion of sorption to oxidation increased in time and after 120 minutes it was 41 %.

Table 3

Efficiency of the removal of *p*-chlorophenol per 1 g WD extra in simultaneous oxidation/sorption in two consecutive cycles

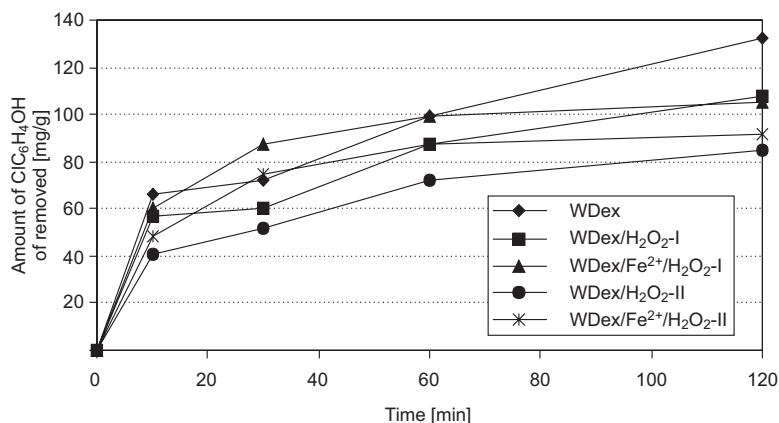| Reaction time [min]	WD extra + Fe^{+2}/H$_2$O$_2$		WD extra + H$_2$O$_2$ – cycle I			WD extra + H$_2$O$_2$ – cycle II		
	ClC$_6$H$_4$OH removed [mg]	ClC$_6$H$_4$OH oxidized [%]	ClC$_6$H$_4$OH removed [mg]	ClC$_6$H$_4$OH oxidized [%]	ClC$_6$H$_4$OH remaining on WD extra (adsorbed) [%]	ClC$_6$H$_4$OH removed [mg]	ClC$_6$H$_4$OH oxidized [%]	ClC$_6$H$_4$OH remaining on WD extra (adsorbed) [%]
10	240	100	72	83	17	59	54	46
30	—	—	91	76	23	65	60	40
60	—	—	105	68	32	90	46	54
120	—	—	138	60	40	99	43	57

Fig. 5. Comparison of the amount of *p*-chlorophenol removed from a solution on 1 g of activated carbon, WD extra, in the presence of hydrogen peroxide in two successive cycles, WD extra/H_2O_2-1 and WD extra/H_2O_2-2, with the amount of oxidized *p*-chlorophenol estimated based on the concentration of Cl^- ions in the WD extra/H_2O_2/Cl-1 and WD extra/H_2O_2/Cl-2 solutions

As shown in Fig. 5, the activated carbon regenerated by simultaneous removal of *p*-chlorophenol using H_2O_2 was reused for another *p*-chlorophenol removal process (WD extra+H_2O_2-2 and WD extra+H_2O_2/Cl-2). The results indicate that the removal efficiency was about 35 % lower, which can be explained by the partial immobilization of the non-oxidized *p*-chlorophenol adsorbed at the carbon surface. The analysis of the reaction course shows that the removal efficiency with regard to *p*-chlorophenol in the subsequent simultaneous sorption/oxidation was increasingly high; after 120 minutes, it was 57 %.

The total amount of *p*-chlorophenol removed from a solution by double sorption and regeneration of activated carbon with hydrogen peroxide or Fenton's reagent is comparable with the amount removed by simultaneous sorption, oxidation and regeneration in a solution. In the latter case, however, there was no considerable mass loss of activated carbon (with a total loss of carbon of up to 10 %), as was the case during sorption and regeneration, where the loss was 20 % per cycle. The process thus contributes to an increase in the useful life of activated carbon.

Conclusion

The analysis shows that the selected activated carbon, WD extra, possesses high sorptive efficiency with regard to *p*-chlorophenol – 150 mg/g. The decomposition of *p*-chlorophenol by means of hydrogen peroxide under the pre-determined conditions resulted in no oxidation of this substance. Using a Fenton reaction was a more effective method of removing *p*-chlorophenol. Its drawbacks, however, included the formation of considerable amounts of iron compounds in the reaction system and a decrease in the pH-value. An alternative solution was to combine the processes of sorption and oxidation involving regeneration of activated carbon consecutively or simultaneously.

The sorptive capacity of the activated carbon regenerated with H_2O_2 or Fenton's reagent was found to be similar to that of virgin carbon. However, the process of regeneration resulted in a significant loss of mass of the activated carbon, especially when Fenton's reagent was used (approx. 20 %). The results for the removal of *p*-chlorophenol from an aqueous solution in the simultaneous processes of sorption and oxidation show that oxidation was the predominant process and included the decomposition of both *p*-chlorophenol and the activated carbon, which indicates the presence of chloride ions and a significant loss of the carbon mass. By extending the reaction time, it is possible to increase the proportion of sorption to oxidation for the removal of *p*-chlorophenol from a solution.

Acknowledgements

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ZASTOSOWANIE POŁĄCZONYCH PROCESÓW SORPCJI I UTLENIANIA DO USUWANIA ZWIĄZKÓW ORGANICZNYCH ZE ŚRODOWISKA WODNEGO NA PRZYKŁADZIE *p*-CHLOROFENOLU

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Abstarakt: W prezentowanej pracy prowadzono badania nad skutecznością usuwania *p*-chlorofenolu z roztworu wodnego, jako symulację eliminowania ze środowiska łatwo rozpuszczalnych związków chlorowcoorganicznych stanowiących jedno z groźniejszych zanieczyszczeń środowiska. Jako metody usuwania wybrano sorpcję, utlenianie nadtlenkiem wodoru i odczynnikiem Fentona oraz symultanicznie realizowany proces sorpcji i utleniania nadtlenkiem wodoru. Wykazano, że nadtlenek wodoru nie utlenia *p*-chlorofenolu, natomiast w reakcji Fentona następuje natychmiastowy rozkład tej substancji. Stwierdzono, że sorpcja na węglu aktywnym jest skutecznym sposobem usuwania *p*-chlorofenolu z roztworu wodnego. Ponadto, zużyty węgiel aktywny można z powodzeniem zregenerować poprzez utlenienie zaadsorbowanej substancji nadtlenkiem wodoru lub odczynnikiem Fentona i ponownie wykorzystać jako sorbent. Jednakże proces regeneracji łączy się ze znaczną stratą węgla aktywnego na skutek jego utlenienia. Znacznie skuteczniejszym rozwiązaniem jest usuwanie *p*-chlorofenolu z roztworu wodnego poprzez utlenienie odczynnikiem Fentona lub nadtlenkiem wodoru w obecności węgla aktywnego. W tych warunkach ma miejsce tak proces sorpcji, jak i utleniania substancji organicznych zarówno w roztworze wodnym, jak i zaadsorbowanych na węglu aktywnym, co równocześnie skutkuje jego regeneracją i umożliwia ponowne wykorzystanie.

Słowa kluczowe: węgiel aktywny, utlenianie, sorpcja, nadtlenek wodoru, *p*-chlorofenol