



## Sorption of zinc ions from aqueous solutions on regenerated activated carbons

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Received 25 November 2002; received in revised form 27 May 2003; accepted 29 May 2003

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### Abstract

This paper discusses the increasing use of activated carbons to remove wastewater contaminants and their contribution to producing wastes abundant in heavy metals. Considering their threat to the environment and their possible recovery, research on the regeneration of spent carbon sorbents and catalysts is advisable. It has also been shown that it is possible to recover activated carbon from spent catalyst ( $CC_{\text{spent}}$ ) generated by vinyl acetate synthesis by using supercritical extraction with carbon dioxide or by leaching with hydrochloric acid or a solution of nitric acid assisted by microwave energy. Activated carbon recovered in this work had a high sorption for zinc ions that was comparable to that of fresh commercial activated carbon.

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*Keywords:* Spent activated carbon; Chemical regeneration; Supercritical extraction; Sorption

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### 1. Introduction

Sorption of metal ions from aqueous solutions on activated carbons has been studied by numerous researchers [1–5]. Its popularity as a treatment process results from the usefulness of the process in treating both waters and wastes coming from different industries. Moreover, both the removal of metal ions as impurities and the recovery of valuable metals from wastes produced by, for example, electrochemical or hydrometallurgical industries, are of importance [6–8].

The investigations on the sorption of metals from aqueous solutions on activated carbons cited here deal in principle with fresh sorbents which, after being found useful in the treatment process and being used, become wastes. In practice, there are no data on further

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treatment of wastes of this kind. Papers on processes of regenerating spent carbon sorbents and on their reuse are not numerous. Meanwhile, considering a wide and increasing use of carbon sorbents and catalysts containing metals and metal compounds on their surface [9–12], the amounts of wastes produced are considerable and will grow. It is then necessary for researchers to consider not only the uses of carbon sorbents and catalysts, but also their management as wastes. It is to be noted that the content of metals in such wastes may be as high as 30 wt.% [12].

The methods of regenerating activated carbons include thermal and chemical, electrochemical, catalytic and biological processes [13,14]. However, in the case of regeneration of spent carbon sorbents and catalysts, only chemical methods are to be considered. This limitation results from the fact that both the sorption of metals from aqueous solutions on activated carbons and the impregnation of carbons with metal compounds, when catalysts are obtained, are chemical processes. Therefore, thermal regeneration is of no practical use in the removal of metals from spent carbon whereas such methods as electrochemical and biological processes are basically have no commercial application.

Chemical methods of regenerating spent carbon sorbents and catalysts consist mainly of removing of metals and their compounds bounded on the surface of the carbon via extraction with organic and inorganic solvents [15–19] as well as with supercritical solvents [20–27]. In extraction wastes are treated with properly selected solvents of defined volume. To increase the efficiency of the removal of metals from the surface of these porous substances, it is necessary to use considerable volumes of solvents in the extraction process resulting in obtaining aqueous solutions of metals that are dangerous wastes themselves.

In the case of extraction with solvents in the supercritical phase, the most popular extraction solvents are: carbon dioxide and  $\text{CHF}_3$ ,  $\text{C}_5\text{H}_{12}$ ,  $\text{C}_4\text{H}_{10}$ ,  $\text{CH}_3\text{OH}$ ,  $\text{H}_2\text{O}$ ,  $\text{N}_2\text{O}$ ,  $\text{NH}_3$ ,  $\text{CO}_2$  [20–27]. However, the foregoing are of no use as far as the removal of metals from spent carbon sorbents and catalysts is concerned. Crown ethers, perfluoro macrocycles and porphyrines can be used as modifiers of carbon dioxide in the supercritical phase [26]. Along with metals, they form complexes soluble in carbon dioxide in the supercritical phase and make metal removal from the carbon surface possible. Unfortunately, these extraction solvents are still very expensive.

Despite all these shortcomings, currently available methods of regeneration contribute to removing metals and their compounds from the surface of spent activated carbons and to regenerating the carbon [16,19,27]. In view of the fact that these carbons can be reused, investigations of chemical methods of regeneration should be carried out and their operational shortcomings eliminated as well as lowering costs.

The work described here was performed to assess the sorption of zinc ions from aqueous solutions on activated carbons recovered from spent catalysts ( $\text{CC}_{\text{spent}}$ ) used in vinyl acetate synthesis. The fresh catalyst used in vinyl acetate product contains even up to 35 wt.% of zinc acetate on the surface of activated carbon [28]. After its use, the catalyst becomes a waste that contains approximately 26 wt.% of zinc occurring in an acetate, oxide or a metallic form as well as organic compounds which are by-products of vinyl acetate synthesis [29]. The catalyst is composed of granular activated carbon of high mechanical resistance and porosity. Spent catalysts are, on the one hand, dangerous wastes, but, on the other hand, they are valuable secondary materials.

Based on literature reports [16,18,24,29,30–34], the following methods of recovering activated carbon from spent catalysts of vinyl acetate synthesis were chosen.

- Extraction with organic solvents with a view to removing organic substances deposited on the surface followed by leaching zinc and its compounds with a solution of hydrochloric acid.
- Extraction with carbon dioxide in the supercritical phase with a view to removing organic substances followed, as above, by leaching zinc and its compounds with a solution of hydrochloric acid.
- Leaching with water and solutions of HCl and HNO<sub>3</sub> assisted by microwave energy (frequency 2450 MHz); this method has not yet been used with spent carbon catalysts, but it is proposed here to combine the decomposition of organic impurities with the transfer of the metals to a solution using a single process.

The use of the currently available methods to remove both organic impurities and a metal from the surface of spent vinyl acetate catalysts resulted in the recovery of the carbon, which was then used in the sorption of zinc ions from aqueous solutions.

## 2. Experimental

In the investigations, a spent vinyl acetate synthesis catalyst marked CC<sub>spent</sub> was used. It contained 26 wt.% of zinc acetate and organic substances which were the by-products of synthesis. Representative samples of the dried spent catalyst and regenerated activated carbon were obtained using the procedures recommended for solids [35]. Samples were weighed with an accuracy of ±0.001 g. The data given here are the arithmetic mean of the values obtained for five independent trials.

### 2.1. Removal of zinc from the surface of spent carbon catalysts leaching

Ten grams of the CC<sub>spent</sub> and 100 cm<sup>3</sup> of 0.1 mol/dm<sup>3</sup> of HCl solution were put into a 250 ml round-bottomed flask. Washing with acid was done eight times. The activated carbon was then washed with 100 cm<sup>3</sup> doses of boiling distilled water until the reaction with AgNO<sub>3</sub> to the presence of chloride ions disappeared. After washing the carbon was dried at 373 K for 1 h. The amount of zinc removal from a spent catalyst, determined with AAS method, is given in Table 1.

### 2.2. Removal of organic compounds from the surface of spent catalysts heating with organic solvents

To remove organic compounds present on the surface of a spent catalyst (CC<sub>spent</sub>), a three-step procedure was utilized: acetone, *n*-hexane and ethyl acetate were employed as solvents for the contaminated material. The process was carried out as follows: 10 g of the catalyst were placed in a 250 cm<sup>3</sup> round-bottomed flask, covered with 100 cm<sup>3</sup> of *n*-hexane and heated while employing reflux condenser for 1 h. Washing with the forementioned solvents was done four times. After filtering the solvents, the sample was dried at 343 K for

Table 1

Amount of zinc removal from spent catalyst  $CC_{\text{spent}}$  according to the procedure involved

Catalyst	Zinc removal procedure	Amount of zinc removal from spent catalyst (wt.%)	Activated carbon obtained
$CC_{\text{spent}}$	Initial heating with organic solvents then leaching with a solution of HCl	72.3	C1
	Supercritical extraction then leaching with a solution of HCl	93.7	C2
	Leaching with solutions mentioned below and assisted by microwave energy		
	Leaching with H <sub>2</sub> O	68.3	C3
	Leaching with HCl 1:1	94.2	C4
	Leaching with HNO <sub>3</sub> 1:1	94.7	C5

2 h. After drying, the washing procedure was repeated using acetone as a solvent, and the sample was dried at 333 K. In the third stage of washing, ethyl acetate was used. Finally, the sample was dried at 353 K. After this initial treatment zinc acetate was removed according to the above-mentioned procedure (the one with the use of HCl). The degree of zinc removal is shown in Table 1. The activated carbon obtained was marked C1.

### 2.3. Supercritical extraction

A sample of 10 g of spent catalyst was treated by supercritical extraction carried out under the following conditions: temperature, 333 K; pressure, 160 bar; flow of CO<sub>2</sub>, 30 dm<sup>3</sup>/h; amount of passed CO<sub>2</sub>, 167.6 dm<sup>3</sup>; extraction time, 315 min. The extraction solvent was recovered from carbon dioxide in a wash system filled with acetone; temperature in a washer, 268 K.

After extraction zinc acetate was removed from the catalyst using the above-mentioned procedure (the one with the use of HCl). The degree of zinc removal from a catalyst is given in Table 1. The activated carbon obtained was marked C2.

### 2.4. Leaching assisted by microwave energy

A 1 g sample of a spent catalyst was placed in 25 cm<sup>3</sup> of distilled water, HCl 1:1, HNO<sub>3</sub> 1:1 (v/v) and was exposed to microwaves. The procedure was carried out in a UniClever mineralizer (2450 MHz) under conditions designed to avoid the decomposition of the carbon matrix. A two-step treatment procedure was used: (1) the first stage 60% of generator power for 5 min and (2) the second stage 80% of generator power for 5 min. Leaching was done three times under the conditions mentioned above using a new sample of leaching solution each time. The activated carbons were marked C3 (obtained from the sample placed in distilled water), C4 (obtained from the sample placed in HCl), C5 (obtained from the sample placed in HNO<sub>3</sub>). The degree of zinc removal from a catalyst is given in Table 1.

### 2.5. Isotherms of sorption and porous structure

The low-temperature adsorption of nitrogen (77 K) was tested using activated carbon (C2) and commercial carbon (CDex produced by GRYFSKAD at Hajnówka, Poland).

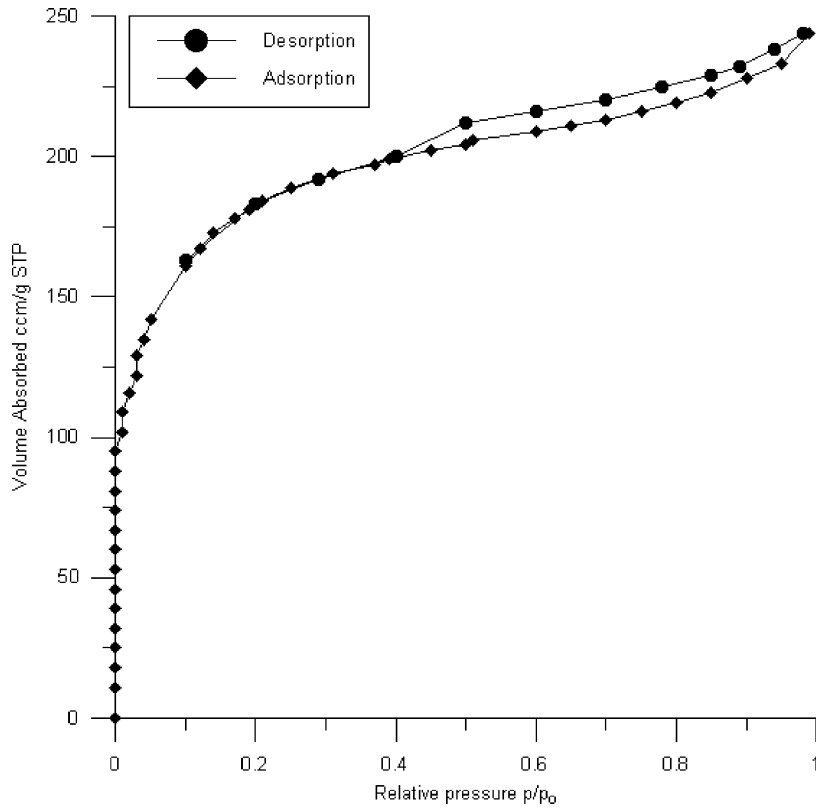


Fig. 1. Isotherm of sorption and desorption of  $N_2$  at 77 K for carbon CDex.

Adsorption and desorption isotherms were determined using a Sorptomatic 1900 apparatus. The isotherms are shown in Figs. 1 and 2.

The specific surface area ( $S$ ) and the total volume of pores ( $V_T$ ) of the tested activated carbons were determined using a simplified BET method described in the paper by Czarny et al. [36]. The results of the analyses are shown in Table 2.

Table 2  
Characteristics of activated carbons

Sample ID	Ash content (wt.%)	Specific surface area ( $m^2/g$ )	Total pores volume ( $cm^3/g$ )
CC <sub>spent</sub>	18.9	56	–
C1	13.8	540	0.45
C2	8.6	986	0.72
C3	14.4	450	0.62
C4	11.8	920	0.60
C5	13.9	880	0.60
CDex (fresh)	12.5	1024	0.68

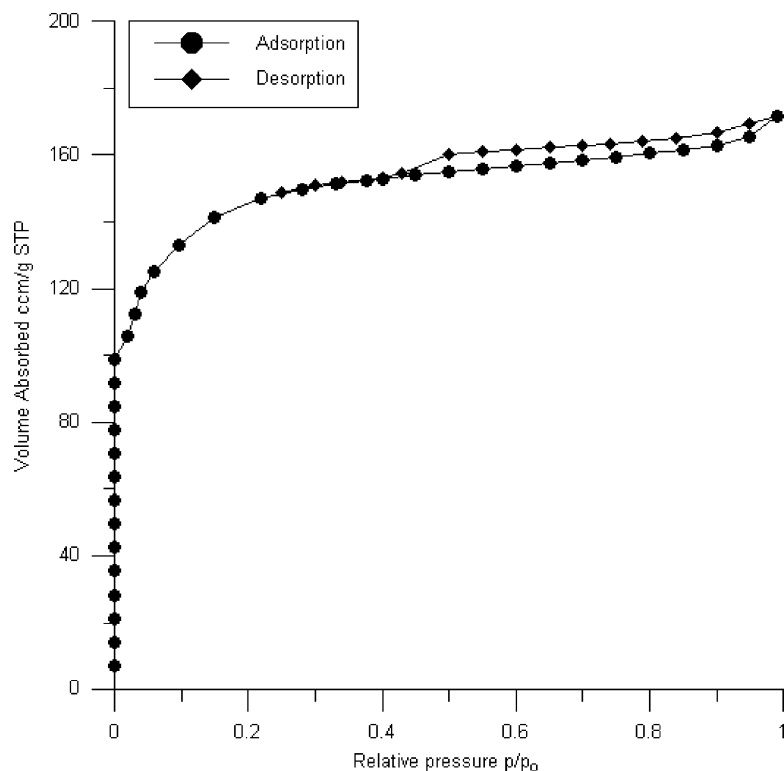


Fig. 2. Isotherm of sorption and desorption of  $N_2$  at 77 K for carbon C2.

## 2.6. Ash content

The samples of spent catalyst and activated carbons weighing approximately 1 g each were incinerated at 1123 K to determine the ash content by the difference between the feed and ash. The results are shown in Table 2.

## 2.7. Sorption of zinc ions from aqueous solutions on recovered activated carbons

Recovered activated carbons marked C1, C2, C3, C4, C5 and, for comparison, commercial activated carbon CDex were used in an adsorption test.

Zinc adsorption isotherms were determined using zinc solutions containing 2; 4; 6; 8; 10; 12; 15; 20 mmol/dm<sup>3</sup> (pH 4–6). In this determination, the time necessary to achieve adsorption equilibrium was determined using a solution of 10 mmol/dm<sup>3</sup> zinc ions and activated carbon C1. The process of sorption was carried out according to the procedure noted below, and the quantity of sorption was assessed after 0.5; 1.0; 3.0; 5.0 and 12 h. The results are given in Table 4.

A 1 g sample of activated carbon was put in a 250 ml round-bottomed flask and flooded with 100 ml of solutions of zinc ions of increasing concentration. KCl in the amount of

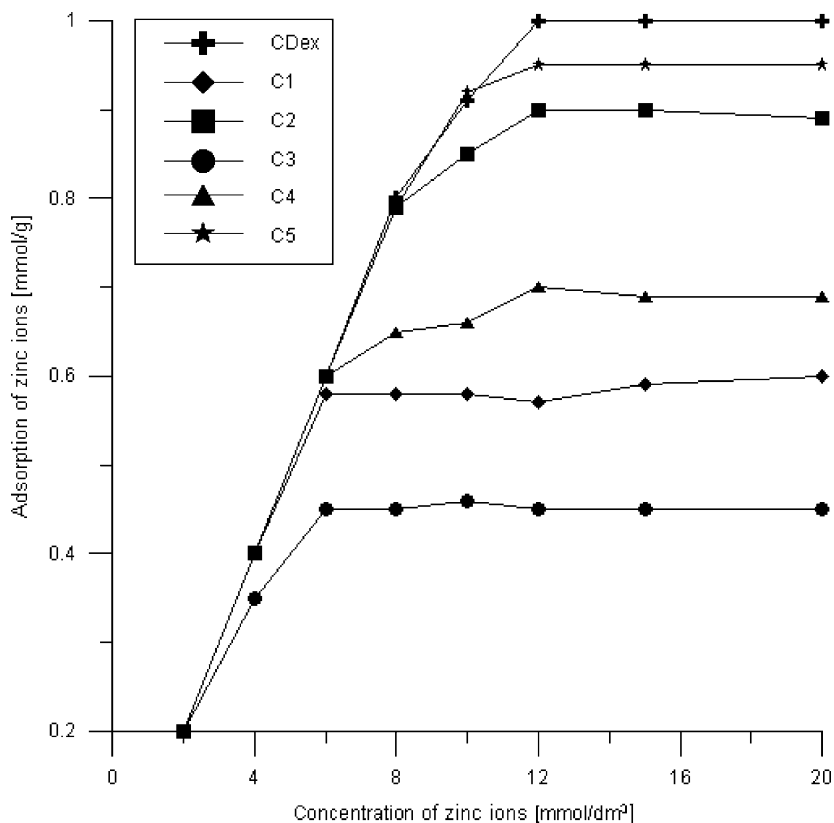


Fig. 3. Comparison of sorption of  $Zn^{+2}$  ions from aqueous solutions on activated carbons obtained and commercial activated carbon CDex according to concentration of solution.

0.2 g was added to each sample. The flask was shaken for 3 h after which the solution was decanted and left for 24 h. Then, 5 ml of a clear layer of the solution was analyzed for zinc. The quantitative determination of zinc ions in solutions after sorption (for each carbon) was performed using an AAS. The results are given in Fig. 3.

### 3. Discussion

As shown in Table 1 using the methods described here, we were able to remove from 68.3 to 94.7% of zinc present in a catalyst. With classical leaching employing a solution of HCl, 72.3% of the zinc was removed in 20 h, whereas for supercritical extraction and leaching with HCl, 93.7% of the zinc was removed in 7 h. The reduction of the leaching time with a solution of hydrochloric acid after supercritical extraction with carbon dioxide has been reported in the previous papers by this author [29,37], because along with organic substances a considerable quantity of zinc is removed in the supercritical extraction process. It can

be concluded that when organic impurities are removed from the surface by supercritical carbon dioxide this removal contributes also to removing metal. However, the nature of this phenomenon has not yet been explained.

Leaching is another method of removing organic impurities and a metal from the surface of a spent catalyst. This process was performed in a pressure vessel with the use of microwave energy. Under such conditions, there are three factors affecting a sample: (1) acidity, (2) boiling temperature of a solution and (3) high pressure. In the process a different degree of zinc removal was obtained depending on the leaching agent involved. The lowest removal (68.3%) was obtained using distilled water in the process, whereas with the use of a solution of hydrochloric acid or nitric acid (V) the zinc removal was approximately 94%. Using 200 cm<sup>3</sup> of solution for 10 g of a sample, the treatment took about 45 min. The shortening of the process and the possibility of using a smaller volume of leaching solution were obtained thanks because the leaching and the decomposition of organic matrix took place at the same time. Assistance in removal was also due to the effective use of energy. The result is the reduction in cost of leaching. In addition, a higher concentration of the metal occurs in the solution after leaching, making its separation easier.

The characteristics of the recovered activated carbons are shown in Table 2. The data indicate that in the process of removing the rest of active phase from the spent catalyst, with the use of proposed methods, organic substances and part of ash are also removed. This result is evidenced by the high specific surface of the carbons and the volume of the pores.

Carbon C2 which was recovered from the spent catalyst in the process of supercritical extraction as well as leaching with hydrochloric acid has the highest specific surface (986 m<sup>2</sup>/g) and the lowest content of ash (8.9 wt.%). This result confirms the high efficiency of supercritical extraction in removing organic impurities from the carbon matrix as well as the possibility of removing metal and ash both during extraction and leaching with HCl.

The ash loss in the treated samples in comparison with the initial material (CC<sub>spent</sub>) has been measured in carbons C3–C5 recovered in the leaching process assisted by microwave energy. The mass loss of the initial sample from the leaching process depends on the solution used (Table 3) and is higher than the ash loss. That finding means that in the leaching process assisted by microwave energy, part of the carbon matrix is decomposed. A similar effect was observed by Fang and Lai [38] who described microwave regeneration of activated carbons contaminated with organic substances. The mass loss is unfavourable but still in this regeneration method it is lower than in the thermal regeneration of activated carbons (where 15% of mass loss was found) [39]. At the same time, activated carbons C4 and C5 had high specific surface areas and the volume of pores was comparable to carbon CDex.

Activated carbon C4 recovered in the process of leaching with HCl has a lower ash content (11.7 wt.%) and higher specific surface (920 m<sup>2</sup>/g) than carbon C5 (ash content,

Table 3  
Mass decrement of sample of spent catalyst (marked CC<sub>spent</sub>) during leaching assisted by microwave energy

Catalyst	Leaching agent	Conditions of before leaching	Mass decrement of sample (wt.%)
CC <sub>spent</sub>	H <sub>2</sub> O	(1) 60% of power, –5 min	4.2
	HCl 1:1		8.4
	HNO <sub>3</sub> 1:1	(2) 80% of power, –5 min	5.5



13.9 wt.% and specific surface area, 880 m<sup>2</sup>/g), which was recovered through leaching of the spent catalyst with HNO<sub>3</sub> solution. The smaller specific surface of carbon C5 is a result of the strongly oxidizing surroundings that destroyed the micropores and the pores having larger radii are created. This phenomenon leads to the lowering of the specific surface [40]. At the same time, the number of the acidic surface functional groups on the surface of activated carbon increases, which should be conducive to sorption of metal ions from aqueous solutions [41].

The recovery of carbon from spent catalysts makes sense only when the carbon will be useful in another process, e.g. for sorption of selected impurities from aqueous solutions or waste gases. The assessment of the sorptive abilities of activated carbons is based on sorption and desorption curves. In this connection, carbon C2, which should have been shown good sorptive properties, was evaluated by a nitrogen sorption isotherm at 77 K. For comparison, an isotherm was also drawn for commercial carbon CDex. The sorption and desorption isotherms for both carbons are given in Figs. 1 and 2. They are comparable thus showing a considerable fraction of the mesopores in the porous structure. The volume of the mesopores is  $V_{\text{meso}} = 0.14 \text{ cm}^3/\text{g}$  for C2, and  $V_{\text{meso}} = 0.2 \text{ cm}^3/\text{g}$  for CDex. The specific surface area is approximately 986 m<sup>2</sup>/g for C2 and 1024 m<sup>2</sup>/g for CDex. The nitrogen sorption isotherm on C2 is indicative of good sorptive properties.

The nitrogen sorption isotherm does not fully assess the sorptive abilities of carbons in aqueous solutions. Therefore, the next stage concentrated on examining the sorption of zinc ions from aqueous solutions on C1–C5 activated carbons. The evaluations were done under static conditions. For the sake of comparison, the sorption of zinc ions on commercial activated carbon CDex which is commonly used in water treatment was carried out. The zinc ion isotherms were determined as a function of the duration of the adsorption process (for activated carbon C1, Table 3 and Fig. 1) and of concentration (Table 4 and Figs. 2 and 3). To minimize the influence of the change of solution ion strength on sorption, the investigations were made with the constant addition of KCl. Sorption was carried out using pH 4–6 solutions. As shown in the literature [6], this solution is optimal for the sorption of zinc ions on activated carbon.

In the first stage of investigation, the time needed to establish adsorption equilibrium was determined. Considering the isotherm of the sorption of zinc ions from the solution of concentration 10 mmol/dm<sup>3</sup> on carbon C1 (Table 4), it was found that the sorption is maximum after 3 h. The sorption time was used for other samples.

The results of the sorption of zinc ions from solutions of different concentrations are given in Fig. 3. The sorption isotherms show that carbon C3 has the lowest sorptive capacity

Table 4  
Sorption of Zn<sup>+2</sup> ions on carbon C1 according to duration of sorption process

Sample	Time of sorption (h)	Quantity of Zn <sup>+2</sup> ions in solution before sorption (mmol)	Quantity of Zn <sup>+2</sup> ions in solution after sorption (mmol)
C1	0.5	1.0	0.58
	1.0	1.0	0.47
	3.0	1.0	0.43
	5.0	1.0	0.42
	12.0	1.0	0.42

(0.4 mmol/g). The removal was obtained by the partially removal of both zinc compounds and organic impurities from the surface of spent catalysts. It is unlikely that one can obtain a good sorbent under current leaching conditions. Better sorption results (0.6 mmol/g) were obtained for carbon C4, but much lower than for commercial carbon CDex, for which the sorption of zinc ions under present conditions removed 1 mmol/g. Carbon C4 was obtained by leaching a spent catalyst with a solution of hydrochloric acid assisted by microwave energy. Like carbon C3, the current conditions do not favour the complete removal of impurities from the surface of catalysts and the reuse of their carriers as sorbents.

The results obtained show that carbons C2 and C5 have the highest sorptive volume (0.9 and 0.95 mmol/g). It is noteworthy that in this work, the sorption on carbons C2 and C5 is comparable to the sorption of zinc ions on fresh commercial carbon CDex.

Activated carbons C2 and C5 were recovered with the maximum removal of metals and other organic impurities from the surface of spent catalysts. To obtain carbon C2, supercritical extraction was used to remove organic impurities. It was combined with leaching using hydrochloric acid. The results of the sorption of zinc ions from aqueous solutions prove that this method is very useful in removing impurities from a waste carbon surface and in recovering carbon carriers which can be used as sorbents.

Another method that is useful in removing impurities deposited on the surface of spent catalysts is leaching with oxidizing acid under high pressure and temperature. In the acidic and strongly oxidizing surroundings, the removal of organic impurities and ash from the surface of spent metal catalyst takes place. Good sorptive properties of this carbon for zinc ions can be explained by the fact that the most of the surface becomes available again for them. The presence of acidic oxygen groups is also beneficial. This method also seems useful in recovering carbon carriers from spent catalysts and is worth more attention in further investigations.

#### **4. Conclusions**

From the literature dealing with activated carbons and their properties and use in sorption and regeneration as well as from investigations presented here, it can be concluded that:

1. a wide and still increasing use of activated carbons and their derivatives such as catalysts containing metals and their compounds makes it necessary to strive for their safe neutralization or management after use. Moreover, the recovery of both substances absorbed on the surface of spent activated carbons and activated carbons themselves, whose sorptive properties are intact despite their repeated treatment, should also be considered;
2. among the methods used here to recover carbon carriers from vinyl acetate synthesis spent catalysts, supercritical extraction combined with leaching with hydrochloric acid and leaching with nitric acid in the system assisted by the use of microwave energy was most effective with over 90% of zinc and organic substances removal;
3. the activated carbons recovered by methods discussed in the work and marked C2 and C5 showed a high sorptive volume for  $Zn^{+2}$  ions in an aqueous solution. Their sorptive properties were comparable to those of commercial activated carbon CDex, used in water treatment;

4. the sorption of zinc ions from aqueous solutions under static conditions shows the usefulness of these methods of removing impurities from the surface of carbon waste and of recovering activated carbon which can be used as a sorbent. Furthermore, the methods can contribute to further investigations on sorption on carbons recovered under dynamic conditions.

## References

- [1] C.A. Burns, P.J. Cass, I.H. Harding, R.J. Crawford, *Colloids Surf.* 155 (1999) 63.
- [2] Y. Onganer, C. Temur, *J. Colloid, Interface Sci.* 205 (1998) 241.
- [3] C. Namasivayam, K. Kadirvelu, *Carbon* 37 (1) (1999) 79.
- [4] Y.F. Jia, C.J. Steele, K.M. Thomas, *Carbon* 36 (9) (1998) 1299.
- [5] S. Biniak, M. Pakula, S. Szymański, A. Świątkowski, *Langmuir* 15 (1999) 6117.
- [6] O.N. Kononova, A.G. Kholmogorov, A.N. Lukianov, S.V. Kachin, G.L. Paskhov, Y.S. Kononov, *Carbon* 39 (2001) 383.
- [7] S.B. Makhov, V.A. Domrachev, V.I. Budarev, *Zvetnye metally* 1 (1997) 33.
- [8] H. Jankowska, A. Świątkowski, L. Starostin, J. Ławrinienko-Omiecynska, *Adsorpcja jonów na węglu aktywnym*, PWN, Warszawa, 1991, p. 161.
- [9] T. Wigmans, *Carbon* 27 (1998) 13.
- [10] E. Auer, A. Freund, J. Pietsch, T. Tacke, *Appl. Catal. A Gen.* 173 (1998) 259.
- [11] R.C. Bansal, J.B. Donnet, F. Stoeckli, *Active Carbon*, Marcel Dekker, New York, 1988.
- [12] M. Smisek, S. Cerny, *Active Carbon. Manufacture, Properties and Applications*, Elsevier, Amsterdam, 1970.
- [13] M. Sheintuch, Y.I. Matatov-Meytal, *Catalysis Today* 53 (1999) 73.
- [14] R. Taft, *Res. Crt. Rep. No. TWRC-7, Regeneration of Spent Activated Carbon*, US Department of Int. FWPCA, February 1969.
- [15] S.D. Lambert, N.D.J. Graham, C.J. Sollars, in: *Proceedings of the Sixth GNEST International Conference on Environmental Science and Technology*, vol. B., University of the Aegean, Samos, Greece, 31 August–2 September 1999, pp. 1–8.
- [16] V.D. Mundale, H.S. Joglekar, A. Kalam, J.B. Joshi, *Can. J. Chem. Eng.* 69 (1991) 1149.
- [17] V.S. Mishra, V.V. Mahajani, J.B. Joshi, *Ind. Eng. Chem. Res.* 34 (1995) 52.
- [18] S.R. Cline, B.E. Reed, *J. Env. Eng.* 121 (1995) 700.
- [19] B.E. Reed, P.C. Carriere, M. Roderic, *J. Env. Eng.* 122 (1996) 48.
- [20] R.M. Smith, *LC-GC International* 9 (1996) 8.
- [21] S. Higashidate, Y. Yamauchi, M. Satio, *J. Chromatog.* 515 (1990) 295.
- [22] H.Q. Hu, S. Guo, *Fuel Proces. Technol.* 31 (1992) 79.
- [23] A. Wilhelm, A. Hedden, *Fuel* 65 (1986) 1209.
- [24] R.P. DeFilippi, V.J. Krakonis, R.J. Robey, M. Modell, *Supercritical Fluid Regeneration of Activated Carbon for Adsorption of Pesticides*, US EPA Report, Washington, DC, 1980.
- [25] C.S. Tan, D.C. Liou, *Ind. Eng. Chem. Res.* 27 (1988) 988.
- [26] E.K. Wilson, *C&EN*, April 15 1996, p. 27.
- [27] S. Wang, S. Elshani, C.M. Wai, *Anal. Chem.* 67 (1995) 919.
- [28] L. Dąbek, *Przem. Chem.* 80 (2001) 290.
- [29] L. Dąbek, *Adsorp. Sci. Technol.* 20 (2) (2002) 141.
- [30] A. Baker, E.J. Poziomek, *Carbon* 13 (1975) 347.
- [31] T. Chiou, P.J. Recroft, *Carbon* 15 (1977) 49.
- [32] Xijun Hu, Lecheng Lei, Hiu Ping Chu, Po Lock Yue, *Carbon* 37 (4) (1999) 631.
- [33] Y.L. Wang, Y.Z. Wan, X.H. Dong, G.X. Cheng, H.M. Tao, M. Wen, *Carbon* 36 (11) (1998) 1567.
- [34] R. Leyva Ramos, J. Ovalle-Turrubiarres, M.A. Sanchez-Castillo, *Carbon* 37 (4) (1999) 609.
- [35] G. Brands, *Fresenius Z. Anal. Chem.* 314 (1983) 646.
- [36] Z. Czarny, J. Wolczko, L. Dąbek, *Przem. Chem.* 69 (1990) 404.
- [37] L. Dąbek, A. Świątkowski, J. Dziaduszek, *Adsorp. Sci. Technol.* 20 (7) (2002) 623.
- [38] C.S. Fang, P.M.C. Lai, *Chem. Eng. Comm.* 147 (1996) 17.
- [39] F. Salvador, C. Sánchez Jiménez, *Carbon* 37 (4) (1999) 577.
- [40] C. Namasivayam, K. Periasamy, *Carbon* 37 (1) (1999) 79.
- [41] J.-W. Shim, S.-J. Park, S.-K. Ryu, *Carbon* 39 (11) (2001) 1635.