Adsorption (2007) 13: 579–586
DOI 10.1007/s10450-007-9024-6

Electrochemically assisted adsorption/desorption of bentazone on activated carbon cloth

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Received: 24 April 2007 / Revised: 3 July 2007 / Accepted: 11 July 2007 / Published online: 22 September 2007

Abstract This paper investigates the use of electrochemical techniques for the removal of a common herbicide, bentazone, from water streams using a carbon-based electrode. Activated carbon cloth with high surface area and narrow micropores was used as electrode. For a better understanding of the process, adsorption was investigated under both open circuit and controlled polarization conditions, the latter in anodic and cathodic directions. It was found that anodic polarization enhances the kinetics of adsorption of the herbicide on the carbon cloth, the extent of which is strongly related to the applied current value. At converse, cathodic polarization induces the reversible desorption of the compound. Moreover, in-situ UV spectra recording on the solution did not show any structural change of the herbicide upon polarization, demonstrating the reversibility of the process for the regeneration of the adsorbent and the recovery of the compound. Based on these experiments, a mechanism is proposed to interpret the reversible sorption of bentazone under polarization.

Keywords Electrosorption · Electrodesorption · Activated carbon cloth · Pesticides · Liquid phase adsorption

Abbreviations

C	solution concentration (mg L^{-1})
C_o	initial solution concentration (mg L^{-1})
DFT	density functional theory
DR	Dubinin-Radushkevich method
E	equilibrium reduction potential (Volts)
I	applied current (mA)
K_{ads}	adsorption rate constant (min^{-1})
NHE	Normal Hydrogen Electrode
p	pressure
p_0	saturation pressure
pH_{PZC}	pH of the point of zero charge (pH units)
PSD	pore size distribution
q	amount of bentazone adsorbed (mg g^{-1})
q_e	amount of bentazone adsorbed at equilibrium (mg g^{-1})
S_{BET}	apparent specific surface area obtained from the nitrogen adsorption data at −196°C by the BET model ($\text{m}^2\ \text{g}^{-1}$)
t	time (min)
U	working electrode potential (Volts)
V_{CO2}	narrow micropores volume, obtained from the CO_2 adsorption data at 0°C, by the DR method ($\text{cm}^3\ \text{g}^{-1}$)
V_{meso}	mesopores volume, obtained from the nitrogen adsorption data at −196°C, by the DFT method ($\text{cm}^3\ \text{g}^{-1}$)
V_{micro}	micropores volume, obtained from the nitrogen adsorption data at −196°C, by the DFT method ($\text{cm}^3\ \text{g}^{-1}$)
V_{TOTAL}	total pore volume obtained from the nitrogen adsorption data at −196°C, and evaluated at $p/p_0 \sim 0.95$ ($\text{cm}^3\ \text{g}^{-1}$)

1 Introduction

Dependence and abuse of chemical pesticides and fertilizers entails one of the most adverse aspects of intensive

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agriculture and industrial activities, due to negative environmental and health impact. The amount of pesticides used in the world exceeded 2 million tons in 2001, herbicides accounting for the largest fraction of total usage in the European Union (EU) (European Communities 2005; EPA 2004). Their repetitive utilization has lead to an increase in their occurrence in wastewater from industrial and human activities. Moreover, pesticides are considered by the EU as priority pollutants (Directive 2000/60/EC) as they are highly noxious (most are carcinogens), long-term persistent and highly mobile throughout the environment. Although environmental concerns on pesticides use in developing countries were raised thirty years ago, hazardous pesticides are still used with little or no protection. Therefore, the issue of their removal is a field of growing interest.

Particularly, the use of bentazone, a post-emergence herbicide, has become very popular for the control of broad-leaved weeds and crops since 2003 after the ban of atrazine in the EU. In association, the herbicides alachlor and bentazone can be substitutes for atrazine to provide the same spectrum of action on weeds in cereals grains crops (mainly maize and rice), resulting in new environmental risks (Dousset et al. 2004). Moreover, bentazone has a relatively high mobility in water.

Faced with an increasing contamination of natural waters by pesticides, adsorption on activated carbon has become widely used in water treatment plants (Bandosz 2006), due to its high depuration efficiency. The design of an adsorption treatment has to be based on (i) the transient force which drives the targeted pollutant from the aqueous phase towards the adsorption sites in the adsorbent and (ii) the technological and economical feasibility of regeneration of the spent adsorbent.

In this connection, electrosorptive techniques employing high surface area electrodes of nano-textured carbons have been recently investigated as a potential technology for removing toxic pollutants from aqueous solutions. The working principle of electrosorption is based on imposing an external electric field in order to force charged species (ions) to move toward oppositely charged electrodes. This technology offers several advantages such as the reversibility, which enables the possibility of the regeneration of the exhausted activated carbon. So far, electrochemical techniques have been successfully applied for the removal of ions (Alfarra et al. 2002; Ying et al. 2002; Afkhami and Conway 2002) and a few organic molecules (Niu and Conway 2002a, 2002b, 2002c, 2003, 2004; Ayranci and Conway 2001; Han et al. 2006a, 2006b), as well as for the regeneration of adsorbents (Brown et al. 2004; Zhou and Lei 2006). In contrast, scarce works report their use for the electro-adsorption/desorption of pesticides or more complicated polycyclic aromatic compounds (Niu and Conway 2003; Ania and Béguin 2007).

The main objective of this research is to explore the application of an electrochemical technique for the adsorption and desorption of bentazone, using an activated carbon cloth as electrode. For the sake of comparison, the adsorption process was also investigated under open-circuit conditions (i.e., without polarization). Special attention was paid to the different factors (heterogeneity of the carbon surface, solution pH, current) that affect the complex process of adsorption and electrosorption from solutions.

2 Experimental

2.1 Materials

A commercial activated carbon cloth -*AX*-, supplied by ACTITEX (France) was used. The sample was washed with distilled water at 60°C during one week, dried at 80°C overnight and stored in a desiccator until use. Bentazone [3-Isopropyl-1H-2,1,3-benzothiadiazin-4(3H)-one-2, 2-dioxide] with the highest purity specification (analytical grade, 99.1% purity) was obtained from Aldrich.

2.2 Textural and chemical characterization of the carbon cloths

Nanotexture of the carbon cloth was characterized by N_2 adsorption at -196°C (Autosorb-1, Quantachrome). Before the experiments, the samples were outgassed under vacuum at 120°C overnight. The isotherms were used to calculate the specific surface area, S_{BET}, total pore volume, V_T, and pore size distribution using the density functional theory (DFT). Additionally, the distribution of pores smaller than 0.7 nm (narrow micropores) was assessed from CO_2 adsorption isotherms at 0°C with the DR formalism. The materials were further characterized by the determination of the point of zero charge (pH_{PZC}) by mass-titration as described elsewhere (Noh and Schwarz 1989).

2.3 Kinetics of adsorption/electrosorption/electrodesorption

Kinetics of adsorption/electrosorption was determined in 20 mL of bentazone aqueous solution (initial concentration 20 ppm) with a fixed mass (11 ± 0.5 mg) of adsorbent. The carbon cloth was cut to desired dimensions (disc shape, ca. 1.2 cm diameter) and attached to a gold disk acting as current collector for the electrosorption experiments. In the latter case, due to the low electrical conductivity of the pesticide solutions, 0.01 $\text{mol}\cdot\text{L}^{-1}$ Na_2SO_4 was used as inert supporting electrolyte (pH of the resulting solution ca. 5–6). A platinum grid served as auxiliary electrode, and Hg/Hg_2SO_4 as reference electrode ($E = 0.614$ V vs. Normal Hydrogen

Electrode, NHE). Small samples of the solution (2 mL) were taken out at predetermined time intervals to measure the pH evolution and the concentration of pollutant during sorption, using a UV spectrometer (Uvikon Xs, Bio-TEk Instruments) at 224 nm. The extracted samples were re-introduced in the cell in order to avoid changes in the total volume of solution. This procedure enabled to investigate the kinetics of adsorption under both open-circuit (OC) and polarization at different current values of +1, +2 and +5 mA (i.e., galvanostatic mode). The adsorption rate constants were determined from the first-order adsorption kinetics equation:

$$\log(q_e - q) = \log q_e - \frac{K_{\text{ads}} t}{2.303}$$

where K_{ads} (min^{-1}) is the adsorption rate constant and q_e and q are the amounts of bentazone adsorbed (mg g^{-1}) at equilibrium and time t (min), respectively.

The possibility of regenerating the carbon cloth was also explored by the reversal of polarization (i.e., cathodic). The carbon cloth was pre-loaded at open-circuit conditions (OC) and subsequently polarized applying a cathodic current (i.e., −5 mA) for 3 hours to provoke the pollutant desorption; the same electrochemical cell described above was used for the regeneration assays. After desorption, the sample was allowed to rest in the same cell until saturation was again attained, before starting a new desorption cycle.

3 Results and discussion

3.1 Characterization of the carbon cloths

Detailed nanotextural characteristics of the as-received cloth are presented in Table 1. The analysis of the pore size distribution (PSD), combining the information of N_2 and CO_2 adsorption isotherms, revealed that the pore volume determined by CO_2 data is larger than the corresponding volume of micropores "seen" by N_2; this indicates that the microporosity of the carbon cloth is mainly composed of pores narrower than 0.7 nm. Moreover, the mesopore volume accounts for less than 10% of the overall porosity.

In order to explore the possible changes in the texture and/or surface chemistry of the carbon cloth during the electrochemical treatment, a blank experiment was carried out by exposing the sample to both anodic and cathodic polarization in the absence of the pollutant. For these experiments, the carbon cloth was initially polarized between −600 and +200 mV vs. Hg/Hg_2SO_4; then it was progressively submitted to more negative (*AXcath*) or to more positive (*AXanod*) cut-off potentials, down to −1200 mV and up to +600 mV, respectively. It is observed that the nanotexture of the carbon cloth remains almost unaltered after cathodic polarization treatment (Table 1); in contrast, surface area, micropore and mesopore volumes slightly (ca. 8%) decreased after anodic polarization. At the same time, the pH_{PZC} decreased by 2 units.

Table 1 Textural and chemical characteristics of the pristine carbon cloth (*AX*), after electrochemical polarization in the absence of bentazone (*AXanod*, *AXcath*), after bentazone exposure (*AXsat*), and after several cycles of cathodic regeneration (*AXreg*)

Sample	S_{BET} [$m^2\,g^{-1}$]	V_{TOTAL}[a] [$cm^3\,g^{-1}$]	V_{micro}[b] [$cm^3\,g^{-1}$]	V_{meso}[b] [$cm^3\,g^{-1}$]	V_{CO2}[c] [$cm^3\,g^{-1}$]	pH_{PZC}
AX	1018	0.432	0.318	0.033	0.353	5.7
AX anod	938	0.423	0.294	0.056	0.311	3.2
AX cath	1076	0.457	0.321	0.034	0.340	5.5
AX sat	484	0.237	0.148	0.047	0.170	–
AX reg	1003	0.495	0.291	0.031	0.320	6.1

[a] Evaluated at $p/p_0 = 0.95$ in the N_2 adsorption isotherms at −196°C

[b] Evaluated from the DFT method applied to the N_2 adsorption isotherms at −196°C

[c] Evaluated from the DR method applied to the CO_2 adsorption isotherms at 0°C

When the cloth is exposed to polarization between −600 and +200 mV vs. Hg/Hg_2SO_4, a pure capacitive behaviour occurs (reversible charge/discharge of the double layer), which is confirmed by the rectangular shape of the voltammogram (Fig. 1). At potentials below −600 mV, a fast current leap related with water reduction and formation of nascent hydrogen is observed, along with an increase in anodic current which is attributed to the reversible electrochemical oxidation of adsorbed hydrogen (Jurewicz et al. 2004). This effect is more pronounced as the cut-off potential goes down, due to an increasing kinetics of hydrogen production. It is important to remark that the carbon cloth has a reversible response; after applying negative potential values (ca. −1200 mV), the voltammogram between −600 and +200 mV superimposes with the initial one. This is reasonable, taking into account that (i) anodic polarization of the cloth was restricted up to +200 mV and (ii) H_2 and OH^- are liberated at the working electrode during cathodic polarization according to:

$$2H_2O + 2e^- \rightarrow H_2 + 2OH^-$$

On the contrary, anodic polarization (*AXanod*) at potentials higher than +200 mV vs. Hg/Hg_2SO_4 causes a slight oxidation of the carbon cloth (Kinoshita 1988), which is confirmed by the appearance of a reversible hump in the voltammogram, due to redox transfer reactions involving the oxygen-containing functionalities created in the carbon matrix (Fig. 1). The position of the cathodic and anodic peaks confirms the existence of the quinone/hydroquinone pair (reduction potential close to −0.15 V vs. Hg/Hg_2SO_4 for

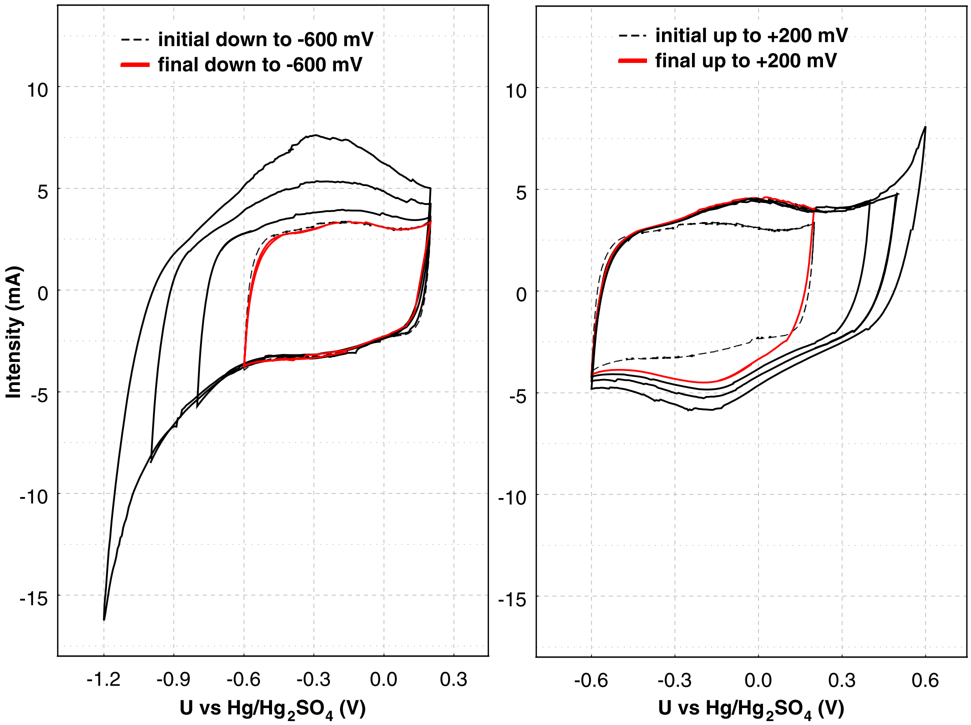

Fig. 1 Series of successive cyclic voltammograms of the carbon cloth material at 2 mV s^{-1} sweep rate taken to progressively more (left) negative and (right) positive, cut-off potential

carbons (Kinoshita 1988) in various environments). Other groups of acidic nature are also created, as inferred by the decrease in the pH_{PZC} of the resulting cloth (Table 1).

In sum, it can be inferred that neither porosity nor surface chemistry of the carbon cloth is altered by negative polarization; on the other hand, positive polarization at potentials higher than +200 mV vs. Hg/Hg_2SO_4 provokes surface oxidation and minor textural changes. This finding is crucial for understanding bentazone adsorption and desorption during polarization (see discussion below), as it should be explained in terms of the occurrence of charges on the carbon surface, rather than to textural changes.

In order to investigate the role of porosity, the nanotextural characteristics of the cloth after bentazone adsorption under polarization were also investigated (Table 1). As expected, the surface area and pore volume of the loaded sample decrease significantly. The fall is particularly important in the volume of micropores (evaluated by both N_2 and CO_2 adsorption), suggesting that adsorption takes place preferentially in the microporosity of the carbon cloth.

3.2 Adsorption of bentazone under anodic polarization

Initially, the electrochemical stability of the pesticide was investigated to ensure that bentazone is not oxidized or reduced during the electrosorption process. Cyclic voltammograms of the aqueous 0.01 mol L^{-1} Na_2SO_4 solution, with and without the targeted pollutant, were recorded at a scan rate of 20 mV s^{-1} in the potential range between −1000 and +1250 mV vs. Hg/Hg_2SO_4; for this aim, the gold disk was used as working electrode and platinum as counter electrode. No differences were found in the voltammograms when the herbicide is present in the solution; this was further confirmed by UV absorption spectroscopy, which indicated that bentazone spectra remain unchanged in this potential range.

When anodic (positive) polarization is applied to the carbon cloth, the working electrode potential increases promptly and reaches a value that enables the electrolyte oxidation according to:

$$2H_2O \rightarrow O_2 + 4H^+ + 4e^-$$

(according to the Nernst equation, the equilibrium potential at the pH of the electrolytic solution (6.5) is $E = +0.84$ V vs. NHE; +0.226 V vs. Hg/Hg_2SO_4). Varying the anodic current from +1, +2 and +5 mA caused a rise in the electrode potential, reaching values of +0.85, +1.1 and +1.2 V vs. Hg/Hg_2SO_4, respectively.

At the same time, the passage of charge gives rise to a slight pH increase in the cell (up to one unit increase). This is linked to the faradaic production of OH^- ions at the counter electrode during polarization, according to:

$$2H_2O + 2e^- \rightarrow H_2 + 2OH^-$$

($E = -0.38$ V vs. NHE; −0.994 V vs. Hg/Hg_2SO_4)

Although in a non-separated cell the OH^- ions created should be neutralized by the H^+ liberated at the working

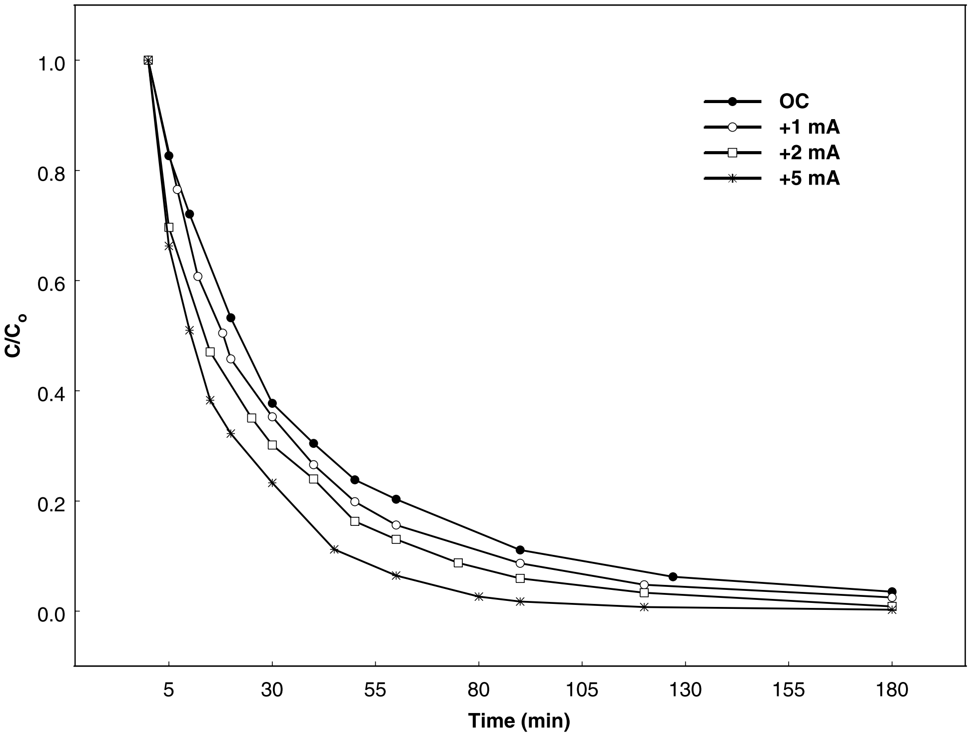

Fig. 2 Concentration decay curves for bentazone under open-circuit (OC) and anodic polarization at +1, +2, +5 mA in a non-buffered solution (0.01 mol·L^{-1} Na_2SO_4 as supporting electrolyte). The initial concentration of pesticide (20 ppm), volume (20 mL) and the mass of carbon cloth (11.5 mg) were the same in all cases. *Symbols* represent experimental data and *solid lines* the fitting to the kinetics model

Table 2 First order adsorption kinetics parameters for bentazone on *AX* in 0.01 mol·L^{-1} Na_2SO_4 under open circuit (OC) conditions and anodic polarization

	q_e [mg g^{-1}]	$K_{ads} \times 10^3$ [min^{-1}]	r^2
OC	27.36	22.54	0.9954
+1 mA	28.62	28.43	0.9970
+2 mA	29.75	37.68	0.9945
+5 mA	30.47	48.18	0.9953

electrode, in this case the pH balance does not apply because the anodic process includes the non-faradaic charging (electrical double layer) of the high surface area carbon cloth. This behavior has also been reported for the electrosorption of azine-type pollutants (Niu and Conway 2002a). Simultaneous trapping of protons in the micropores of the carbon cloth is another effect which could explain the pH increase in the cell. Although the increase is small, this might appreciably affect the efficiency of the electroadsorptive removal of the pollutant.

Regarding bentazone uptake, anodic polarization enhanced the adsorption rate (Fig. 2), being the effect more significant with increasing the anodic current from +1 mA to +5 mA. This was corroborated by the parameters of first-order kinetics (Table 2) calculated for the different anodic conditions. Since the porosity of the carbon cloth is just slightly affected after anodic polarization, the enhancement in the adsorption rate should be attributed to variations in the charge of the carbon surface and/or ionization of the adsorbate.

It should be stressed that bentazone is a weak acid with a pK_a of 3.3; therefore, at the pH of the supporting electrolyte (pH $\sim$ 5.5) the anionic form is predominant in the solution, accounting for 98%. By contrast, the neutral molecule is the main structure in solution at acidic pH, representing ca. 95 mol% at pH = 2. Taking this into account, we believe that the generation of protons at the working electrode could be the key factor in the electroadsorption, as it is responsible for the local pH decrease and it causes the protonation of the anionic bentazone form (predominant in the supporting electrolyte solution, as stated above) to the neutral form. According to previous studies, adsorption at open-circuit conditions is controlled by the solution pH and the degree of functionalization of both the carbon surface and the adsorbate (i.e., dissociation state). It was reported that bentazone removal is favored at acidic solution pH, being bentazone preferentially adsorbed in a molecular form (Ania and Béguin 2007).

Therefore, bentazone uptake enhancement upon anodic polarization is a consequence of the local pH decrease that occurs in the electrode during polarization. Similar results, pointing out the pH-dependence of bentazone adsorption at open-circuit conditions on soils and clays have been reported (Boivin et al. 2005; Ayranci and Hoda 2004).

Concerning the mechanism of electrosorption, several factors should be considered. Because of the π-system interaction, it is expected that solute molecules are adsorbed with their aromatic ring adjacent to the cloth surface to maximize the dispersive interactions (Coughlin and Ezra 1968). Such orientation seems to be favoured in most organic aromatics at small coverage conditions (Damaskin et al. 1971). In this flat position, induced dipole-charge interactions might take place easily between the carbon surface and the π-electrons. This is expected since the charge-density of the electrode surface changes upon polarization. As a consequence, this process promotes dipole-charge interactions and it is able to govern both the rates and extents of adsorption (Niu and Conway 2004; Damaskin et al. 1971). A similar mechanism and orientation has been proposed for electrochemical enhanced adsorption of aniline on activated carbon fibers (Han et al. 2006a), with electrosorption taking place through the aromatic ring approaching the charged carbon surface. Attractive electrostatic interactions between the anionic form of bentazone (predominant in the electrolytic solution) and the positively charged carbon surface (induced by the anodic polarization) might also occur to some extent. This would also lead to a faster uptake.

One should bear in mind that a slight oxidation of the carbon cloth takes place upon anodic polarization (due to oxygen evolution), which might result in a partial collapse of the porous network. Nevertheless, the mild experimental conditions of the electrochemical treatment (current up to +5 mA during 3 hours) prevented a large deterioration of the porous texture of the carbon cloth (Table 1). Also, based on the mechanism proposed for adsorption under OC conditions, a modification of surface functionality should have a negative effect on bentazone uptake (Ania and Béguin 2007) due to the occurrence of negative charges on the cloth, arising from the created oxygen groups. Since it is not the case, two factors should be considered. First, although oxygen evolution produces acidic groups (confirmed by decrease in $\mathrm{pH_{PZC}}$), these may be created either in small amounts or as surface groups which dissociation is not pH dependent. On the other hand, the simultaneous liberation of $\mathrm{H^+}$ during anodic polarization provokes the cancellation of the negative charges arising form the dissociation of the acidic groups, so that the negative effect due to repulsive electrostatic interactions is minimized.

3.3 Desorption of bentazone under cathodic polarization

When a cathodic (negative) polarization is applied to the pre-loaded sample, the reversible desorption of bentazone occurs (Fig. 3). This is an interesting finding from the point of view of the adsorbent regeneration. The UV absorption spectra of the solution recorded *in-situ* during cathodic polarization show that the amount of bentazone in solution increased with time, therefore confirming that it is being desorbed from the activated carbon (Fig. 4). This also probes that the bentazone molecule is weakly adsorbed (no strong or irreversible interactions) on the carbon cloth and that it does not undergo decomposition and/or polymerization. Support for the structural integrity of bentazone under cathodic polarization is the absence of a line shift in the UV spectra (recorded *in-situ*), although the electrode potential reached during the electrodesorption experiments is very low (i.e., −1700 mV vs. $\mathrm{Hg/Hg_2SO_4}$).

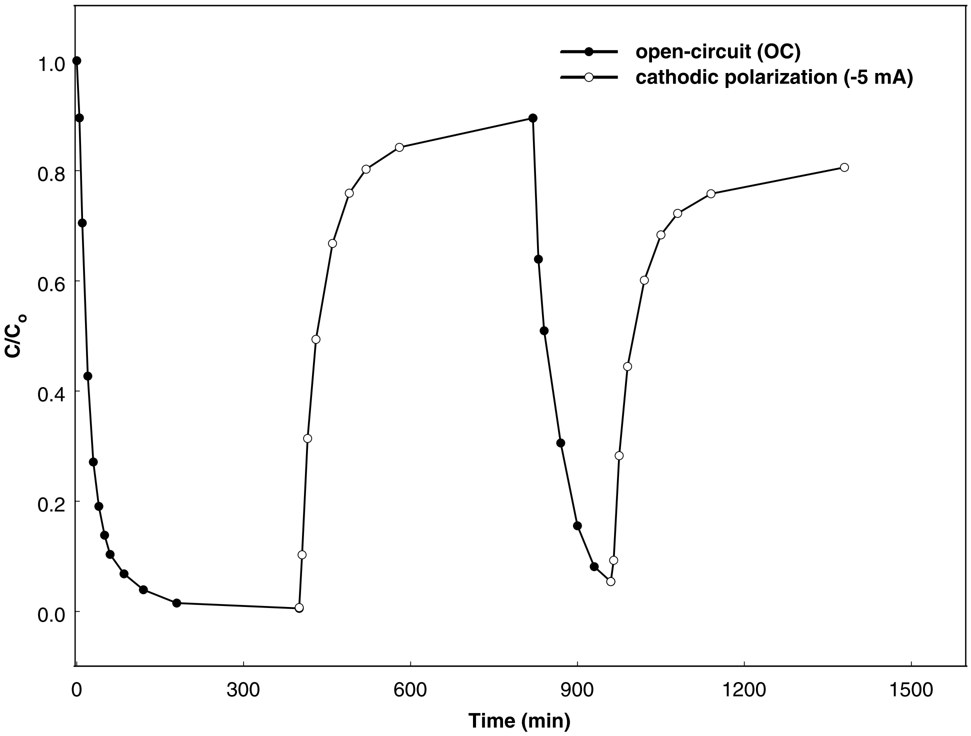

Fig. 3 Bentazone desorption upon cathodic (galvanostatic) polarization on a pre-exhausted carbon cloth electrode, followed by re-adsorption under open circuit (OC) conditions. Mass of carbon cloth: 11.5 mg. Initial concentration: 20 ppm

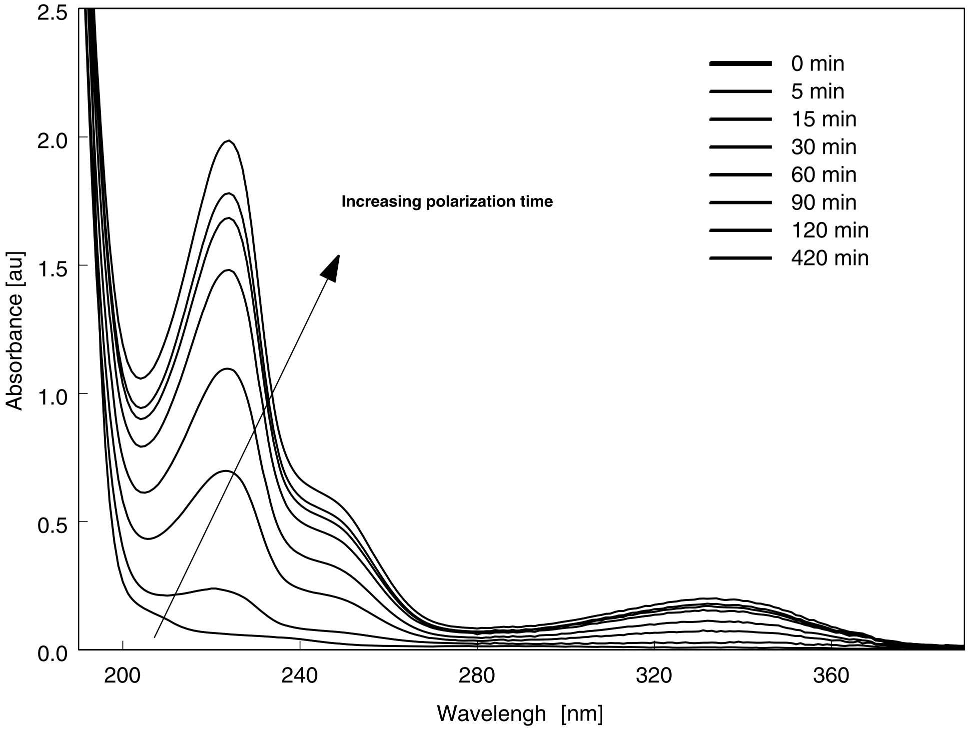

Fig. 4 Changes in the UV spectra of the solution while cathodic polarization (−5 mA) is being applied

The reversibility of the process upon reversal of polarization is in good agreement with the proposed mechanism for bentazone adsorption under both open circuit and anodic polarization (Ania and Béguin 2007). As aforementioned, desorption cannot be attributed to the negligible changes in porosity after applying a negative polarization, therefore it seems reasonable to think that there might exist a connection with the local pH changes. One must bear in mind that bentazone is preferentially adsorbed in a molecular form; at the same time, cathodic polarization generates OH^- at the working electrode, inducing the ionization of the adsorbed neutral molecules into their anionic form. Consequently, electrostatic repulsive interactions arise due to the negative charges of the cloth surface and the anionic form of the bentazone molecules. On the other hand, negative charges on the carbon cloth might affect the charge distribution of bentazone molecules, inducing repulsive intermolecular forces at the interface between the adsorbed bentazone molecules in the flat orientation, thus promoting desorption. The substitution of bentazone molecules by water molecules on the adsorption sites might facilitate the desorption step.

Desorption of bentazone was also confirmed by evaluating the porosity of the regenerated sample -*AXreg*- after cathodic treatment of *AXsat* during three hours (Table 1). Compared to the pre-loaded sample, *AXsat*, the BET surface area and total pore volume significantly increased after cathodic regeneration. However, the values are slightly lower than those in the as-received carbon *AX* suggesting that desorption is non (but almost) quantitative after 3 hours. The decrease is more remarkable in the microporosity, (evaluated by N_2 and CO_2 adsorption) confirming the premise that adsorption takes place preferentially in the narrow micropores of the carbon cloth. Although the results presented above are new and interesting, more investigations are needed to elucidate the exact mechanism of electrochemical desorption of this herbicide. Moreover, further studies are ongoing to evaluate the feasibility of the regeneration process (i.e., regeneration efficiency) over several cycles.

4 Conclusions

Electrochemical technique was investigated for the adsorption/desorption of a common herbicide in aqueous solution, by means of an activated carbon cloth electrode. Applying anodic polarization to the carbon cloth enhances the adsorption rate of bentazone. It seems that the main factor controlling the electrosorption process are the local pH changes occurring in the vicinity of the carbon electrode during polarization, through ionization of the adsorbate and surface charging of the adsorbent.

Bentazone is likely trapped via two mechanisms: (i) π–π dispersive interactions with the aromatic ring approaching to the carbon surface and (ii) electrostatic interactions between the adsorbate and positive charges on the carbon surface. The main difference as a result of bentazone adsorption is a decrease of the micropore volume, underlining the important role of the narrow micropores in bentazone uptake. A similar trend was observed for the adsorption in open circuit conditions.

Reversal of the polarization (i.e., cathodic) results in the desorption of the retained adsorbate. Given the absence of structural changes in the carbon cloth and the structural integrity of bentazone during cathodic polarization, the reversible desorption is attributed to the pH increase and/or to the charges created on the carbon electrode. Based on these results, cathodic polarization is a promising tool for the regeneration of the pre-loaded adsorbent.

Acknowledgements The authors thank Actitex, France, for kindly supplying the activated carbon cloth. COA thanks the CSIC I3P Program, co-financed by the European Social Fund, for a postdoctoral contract. Dr. Khomenko is also acknowledged for fruitful discussions.

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