The adsorption of HCrO$_4^-$ on activated carbon of date pits and its photoreduction on the hetero-system ZnCo$_2$O$_4$/TiO$_2$

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Abstract
The adsorption properties of activated carbon were successfully tested toward the elimination of hazardous HCrO$_4^-$ (30, 50, 70 and 100 mg L$^{-1}$). The material was prepared from Algerian date pits by physical and chemical activations of ZnCl$_2$/CO$_2$ in the goal to develop the microporous volume. The characterization by N$_2$ adsorption at 77 K, the mercury intrusion porosimetry and scanning electron microscopy showed important textural properties. CO$_2$ increases the specific surface area (1192 m$^2$ g$^{-1}$) and pore volume (0.96 cm$^3$ g$^{-1}$). The HCrO$_4^-$ adsorption is described by a pseudo-first-order kinetic model, and the equilibrium data are fitted by the Langmuir model with a maximal adsorption capacity of 46.72 mg/g within 30 min and a constant $K_L$ (0.12 L g$^{-1}$). The remaining HCrO$_4^-$ concentrations (8, 23, 32.5 and 43.5 mg L$^{-1}$) were photocatalytically reduced on the hetero-system ZnCo$_2$O$_4$/TiO$_2$ down to 5 mg L$^{-1}$. The spinel ZnCo$_2$O$_4$, prepared by co-precipitation from the nitrates precursors and characterized by photoelectrochemistry, gives a conduction band of $-1.49$ V$_{SCE}$, more cathodic than the HCrO$_4^-$ level (0.53 V). Therefore, the photoelectrons transfer toward HCrO$_4^-$ species is achieved through TiO$_2$ located midway between the spinel and chromate levels. The photocatalysis is investigated by varying the catalyst dose and HCrO$_4^-$ concentration. ZnCo$_2$O$_4$ has a gap of 1.82 eV and the best reduction efficiency (82%) was obtained under visible light (50 mW cm$^{-2}$) and optimal conditions (HCrO$_4^-$ 23 mg L$^{-1}$, pH ~ 7, ZnCo$_2$O$_4$/TiO$_2$ 50/50%) and follows a first-order kinetic with a rate constant of $3.86 \times 10^{-3}$ min$^{-1}$.

Keywords Date pits · Chemical and physical activation · Adsorption · Chromate · Photoreduction · Hetero-system ZnCo$_2$O$_4$/TiO$_2$

Introduction
In the water treatment for the potable water production, the adsorption on activated carbons remains an attractive challenge for the environmental protection. Activated carbon (AC) is composed of a substantially amorphous carbon material (Bansal et al. 1988; Bouchemal et al. 2012; Tawfik Saleh 2015), delivering highly porous texture, a large specific surface area, a good adsorption capacity and a thermal stability (Skodras et al. 2008; Li et al. 2013; Alswat et al. 2016). Recent developments in the physical adsorption have highlighted the applications of AC. In this regard, the adsorption of gases (Wang et al. 2012; Abiko et al. 2010; Balarak et al. 2017), the uptake of organophosphorus pollutants (Jayson et al. 1982), the retention of bacteriologic pollutants in aqueous electrolytes (Oya et al. 1993), the elimination of heavy metals (Lu et al. 2014; Gomez-Eyles et al. 2013) and chromate photoreduction (Dermèche et al. 2017) are mainly documented in the open literature. AC is also used in bleaching and deodorization of certain liquids and can further play a role as catalyst support in heterogeneous catalysis (Polcaro et al. 1993; Qi et al. 2012). As the water quality is more stringent by the World Health Organization, an increased interest was focused on adsorbents (Ghasemian et al. 2015),
which reduces the chromate ions to less harmful forms (Cr\(^{3+}\)).

Analysis is a clean alternative for the water decontamination inefficent at low concentrations. At this level, the photocatalysis (Saleh and Gupta 2012; Gupta et al. 2012). Indeed, the adsorption yield increases the abatement but becomes inefficient at low concentrations. At this level, the photocatalysis is a clean alternative for the water decontamination which reduces the chromate ions to less harmful forms (Cr\(^{3+}\)) by photoelectrons (Rekhila et al. 2016). In this regard, the semiconductors with medium forbidden band are promising for the exploitation of a large part of the solar energy (Rekhila et al. 2013). The study focuses on the HCrO\(_4^-\) reduction under visible light on the hetero-system ZnCo\(_2\)O\(_4\)/TiO\(_2\). The charge transfer occurs iso-energetically and TiO\(_2\) acts a bridge between the conduction band (CB) of ZnCo\(_2\)O\(_4\) and adsorbed HCrO\(_4^-\) ions. Nano-crystallites produce larger active surface areas with more photoelectrochemical sites, and the spinel ZnCo\(_2\)O\(_4\) was prepared by chemical route at lower temperatures. It was selected because of its low cost, light absorption in the visible region and environmentally friendly characteristics (Guo et al. 2014).

**Experimental setup**

**Preparation of activated carbons (AC)**

Date pits (origin Algeria) are used for the preparation of activated carbons AC. At first, the stones were thoroughly washed with distilled water and dried in an air oven at 120 °C; such protocol was effective to facilitate crushing and grinding. A fraction particle size of between 0.5 and 1 mm was used for the preparation of activated carbons. AC prepared by combining physical and chemical activations was referred in the text as DZCO2: The precursor was impregnated with 7.32 mmol of ZnCl\(_2\) as chemical adjuvant in a solid form and then pyrolyzed at 800 °C (heating rate of 5 °C/min) under low N\(_2\) flow, followed by physical activation (800 °C) under CO\(_2\) flow (20 L h\(^{-1}\), 1 h) and cooled to room temperature. The sample was treated by HCl solution (0.1 M) under reflux ebullition (150 °C, 3 h) in order to eliminate the alkaline compounds and adjuvant excess. DZCO2 was recovered by filtration and thoroughly washed with distilled boiling water until the AgNO\(_3\) test on the filtrate became negative. Finally, DZCO2 was dried in air oven at 120 °C (24 h) and stored in glass bottles until use.

The specific surface area was determined with a MicroMetrics ASAP 2010 equipment at 77 K. Before measurement, the powder was degassed under vacuum (150 °C, 12 h). The morphology of DZCO2 was studied by scanning electron microscopy (SEM) using a JEOL, JSM-6360LV microscope.

The HCrO\(_4^-\) stock solution (1 g L\(^{-1}\)) was prepared by dissolving the accurate amount of K\(_2\)Cr\(_2\)O\(_7\) (Merck, 99%) in distilled water. The solutions with initial concentrations C\(_o\) (30, 50, 70 and 100 mg L\(^{-1}\)) were made up by dilution. Batch adsorption tests were carried out in 100-mL Erlenmeyer flasks placed in a thermostat shaker bath at 25 °C for 2 days. The adsorption study was realized at natural solution without any pH adjustment in order to avoid the interferences.

The kinetic study was realized for the adsorption equilibrium according to the following protocol: 50 mg of DZCO2 samples immersed in 100 mL of HCrO\(_4^-\) solutions with different concentrations C\(_o\) for predetermined time intervals. The solutions were separated from adsorbent by centrifugation (4000 rpm, 5 min), and the HCrO\(_4^-\) concentration was analyzed with a UV–visible spectrophotometer (Jasco V-630); HCrO\(_4^-\) absorbs at 348 nm (ε = 1770 cm\(^{-1}\) mol\(^{-1}\) L). The absorbance calibration curves were plotted versus the concentration of HCrO\(_4^-\). The sampling was performed every 15 min during the first hour and more spaced beyond 1 h. The adsorbed amount \(Q_t\) (µmol/g), at time \(t\), was calculated from the relation:

\[
Q_t = \frac{V}{m} (C_0 - C_t)
\]

where \(C_t\) is the liquid concentration at time \(t\), \(V\) the volume of the solution and \(m\) the mass of AC. The adsorption isotherms are useful for the knowledge of the adsorbate/adsorbent interactions, and several models for analyzing the adsorption equilibrium data exist.

The second part was devoted to the HCrO\(_4^-\) reduction under visible light on the hetero-system CoZn\(_2\)O\(_4\)/TiO\(_2\). The spinel ZnCo\(_2\)O\(_4\) was prepared by co-precipitation; stoichiometric amounts of Co(NO\(_3\))\(_2\)·5H\(_2\)O (purity 99%, Merck) and Zn(NO\(_3\))\(_2\)·6H\(_2\)O (99%, Prolabo) were dissolved in distilled water. The solution was evaporated on a hot plate and heated at 150 °C for the nitrate decomposition until disappearance of brown NO\(_2\) fumes. Then, the
powder was ground in an agate mortar and reheated in a programmed furnace at 650 °C (10 °C min−1, 16 h) with three intermediate regrindings. TiO2 (99%) was supplied by Merck company. The phases were identified by X-ray diffraction (XRD) with a Mini-Diffractometer MD-10 using Cu Kα anticathode (λ = 0.15418 nm).

The forbidden band ($E_g$) was determined from the diffuse reflectance using a dual-beam spectrophotometer (Specord 200 Plus), and PTFE was used as standard. The point of zero charge (pzc) was simply obtained from the equilibrium pH of aqueous solution containing an excess of TiO2 powder using a digital pH meter (Schott CG 825).

The photoelectrochemical (PEC) study was performed in a conventional cell using Pt as auxiliary electrode. The electrical contact on the back pellet was made with silver paint. The electrode surface of the pellet sintered at 700 °C was polished with emery paper (grade 80). The potential of ZnCo2O4 was monitored by PGZ301 potentiostat (Radiometer analytical) and given against a saturated calomel electrode (SCE). The Mott–Schottky characteristic was plotted at a frequency 10 kHz.

The photocatalytic device was composed by a Pyrex reactor open to atmosphere; the temperature was fixed at 25 °C thanks to a thermostated bath. The catalyst powder (100 mg) was suspended in 100 mL of HCrO$_4^-$ solution at different concentrations (8, 23, 32.5 and 43.5 mg L$^{-1}$) at natural pH (~7). The Erlenmeyers were maintained in the dark overnight and then exposed to visible light under magnetic agitation (400 rpm); the light intensity (mW cm$^{-2}$) was measured by a flux meter, and the concentration was determined as above.

**Results and discussion**

**Characterization of activated carbons (DZCO2)**

**Textural characterization**

The textural characteristics of DZCO2 gave a specific surface area of 1192 m$^2$ g$^{-1}$ from the BET method through the N$_2$ adsorption isotherms. The mesoporous ($V_{meso}$, Ø = 3.7–50 nm), macroporous ($V_{macro}$, Ø > 50 nm) and microporous volumes ($V_{micro}$, Ø < 3.7 nm) were obtained from mercury intrusion porosimetry. The elemental analysis of the spinel confirmed the presence of Zn, Co and O. The crystallite size of ZnCo2O4 ($L \sim 45$ nm) was evaluated from the full width at half maximum (FWHM).

The microstructure of DZCO2 produced from date pits was examined by SEM images. Figure 1 shows a rough surface with narrow cavities; this property is characteristic of lignocellulosic material indicating that the structure of the precursor is preserved after pyrolysis. The external morphology of DZCO2 shows homogeneous cavities on the surface, resulting from the evaporation of the chemical adjuvant during the chemical activation (up to 800 °C), followed by the physical activation under CO$_2$ flow. The combined activations liberate the occupied spaces and increase the specific surface area. These results prompted us to test our sample for the adsorption of inorganic pollutant, and hexavalent chromate was selected for the tests.

**Adsorption of chromate under static conditions on AC**

The adsorption processes from aqueous solutions of K$_2$Cr$_2$O$_7$ on DZCO2 were carried out at 25 °C. The
behavior of DZCO2 obtained by chemical/physical activations was not identical; the volume $V_{\text{micro}}$ increases with raising temperature and reaches a value of 0.63 cm$^3$ g$^{-1}$ at 800 °C. DZCO2 has attractive properties for the HCrO$_4^-$ adsorption which shows a good performance; the adsorption isotherm (Fig. 2) is of L-type according to the Giles classification. To exploit the results, we have determined the maximum adsorption ($Q_{\text{max}} = 46.72$ mg/g) and the Langmuir constant ($K_L = 0.12$ L g$^{-1}$, SM1) from the linearized adsorption isotherms which depend on both the porous texture and chemical nature of the surface. It is worth noting that this value is close to those given in the literature (SM2). At pH ~ 7, the species HCrO$_4^-$ predominates and the small $Q_{\text{max}}$ value is presumably due to the negative charge of chromate which is repelled by the negatively charged sites of DZCO2.

**Photocatalysis**

Chromate remains toxic even at low concentrations, and its presence in water is strongly regulated because it is highly hazardous and presents risks for the human health. The maximum limit was set up at 0.5 mg L$^{-1}$ by the World Health Organization. The environmental photoelectrochemistry is an emerging technology, highly recommended for the pollution abatement (Omeiri et al. 2008). According to the standard powder diffraction data (JPCDS No 00-023-1390), the XRD pattern is characteristic of single phase of the normal spinel Zn[Co]$_2$O$_4$ (Fig. 3) (Yuanyuan et al. 2017; Venkatachalam et al. 2017). The optical gap ($E_g$) of the ZnCo$_2$O$_4$ is determined from the plot $(\alpha h\nu)^2$ versus the incident photon $(h\nu)$ (Fig. 4)

$$(\alpha h\nu)^2 = \text{const} \times (h\nu - E_g)$$

(2)

where $\alpha$ is the absorption coefficient. The intersection with the $h\nu$-axis gives a direct gap of 1.82 eV, due to $d$–$d$
transition coming from the crystal field splitting of $\text{Co}^{3+}$ in octahedral coordination ($t_{2g}^3e_{g}^2$).

The flat band potential ($E_{fb}$) is crucial in photocatalysis, and its value (+0.26 V) is reliably obtained from the capacitance versus applied potential, by extrapolation of the linear part to the abscissa axis ($C^{-2}=0$) (Fig. 5):

$$C^{-2} = (0.5 \times e\varepsilon\varepsilon_0 N_A)^{-1}\{E - E_{fb}\} \quad (3)$$

where $N_A$ is the holes concentration and $\varepsilon$ the permittivity of $\text{ZnCo}_2\text{O}_4$. The negative slope characterizes $p$-type conduction for $\text{ZnCo}_2\text{O}_4$ where the holes are the majority carriers. The potential of the conduction band ($-1.49 \text{ V} = E_{fb} - E_g/e + E_a/e$) is negative enough to reduce $\text{HCrO}_4^-$ into trivalent state ($-0.53 \text{ V}$) (Fig. 6).

The charge transfer in heterogeneous system occurs between levels of similar energy, and $\text{TiO}_2$ is used to mediate the electrons transfer since its conduction band is located midway between $\text{ZnCo}_2\text{O}_4$-CB and $\text{HCrO}_4^-$. Coupled semiconductors improve the separation of ($e^-/h^+$) pairs by synergic effect and shift the spectral photoresponse toward longer wavelengths. Nano-crystallites of $\text{ZnCo}_2\text{O}_4$ prepared by chemical route give large active surface areas and more photocatalytic sites. $\text{ZnCo}_2\text{O}_4$ was chosen because of its light absorption over the visible region and chemical stability.

The sun spectrum overlaps the band gap of $\text{ZnCo}_2\text{O}_4$, and the reaction is kinetically governed by the electron flow in the sensitizer $\text{ZnCo}_2\text{O}_4$ because of the small diffusion length; therefore, nano-crystallites are attractive in such a case. The reduction in $\text{HCrO}_4^-$ is non-spontaneous, and the solar energy is used for it reduction to less harmful valence; $\text{Cr}^{3+}$ is less hazardous and can be easily adsorbed. Moreover, the free potential of $p$-$\text{ZnCo}_2\text{O}_4$ is more negative than the flat band potential ($-1.49 \text{ V}$), yielding a spontaneous reduction under illumination. The photoelectrons located in $\text{ZnCo}_2\text{O}_4$-CB ($-1.49 \text{ V}$) are injected into $\text{TiO}_2$-CB ($-0.8 \text{ V}$) and subsequently transferred to $\text{HCrO}_4^-$ species (Fig. 6):

$$\text{HCrO}_4^- + 7\text{H}^+ + 3e^- \rightarrow \text{Cr}^{3+} + 4\text{H}_2\text{O} \quad (E^+ \sim 0.53 \text{ V})$$

The effect of the $\text{ZnCo}_2\text{O}_4$ dose in the hetero-system $\text{ZnCo}_2\text{O}_4/\text{TiO}_2$ on the photocatalytic process was studied, and the maximal efficiency is obtained for a mass ratio (50/50%) (Fig. 7). At low doses; the number of incident photons is larger than that of the photoelectrochemical sites and the photoactivity increases with raising the $\text{ZnCo}_2\text{O}_4$ mass. Conversely, for higher doses, the performance decreases because the catalyst works as optical filter and reduces the light penetration; the light scattering and shadowing effect also account for the decreased performance.

The chromate concentrations in industrial effluents can reach 50 mg L$^{-1}$ and the influence of the remaining

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1 The activation energy ($E_a ~ 0.07 \text{ eV}$) was determined from the electrical conductivity measurement on sintered pellet.
HCrO$_4^-$ concentration (C$_o^+$) is investigated in the range (8–43.5 mg L$^{-1}$) (Fig. 8). The color of the solution weakens over time and turns to white, an evidence of chromate disappearance. The reduction in HCrO$_4^-$ decreases when C$_o^+$ increases, because of the limited number of photocatalytic sites on the spinel surface. The photons flux is fixed, thus keeping constant the number of photoelectrons; so the increase in C$_o^+$ slows down the reduction because the remaining sites become less accessible due the inside porosity of ZnCo$_2$O$_4$ and repulsive forces between the HCrO$_4^-$ ions and the catalyst bulk.

**Conclusion**

This paper has given an overview on the preparation, characterization and application of adsorbent material based on activated carbon from date pits for the treatment of water polluted by chromate. We emphasized that the development of the porous texture of the activated carbon prepared by combined activations depends mainly on the nature of the adjuvant. The HCrO$_4^-$ adsorption of chromate on prepared adsorbent was carried out in a batch reactor, and the data were well filled by the Langmuir isotherm, while the adsorption kinetic followed a pseudo-first-order model. The remaining concentrations remain above the thresholds required by the water standard. Accordingly, the photoelectrochemical HCrO$_4^-$ reduction is reported on the hetero-system ZnCo$_2$O$_4$/TiO$_2$ to lower the concentration. The decay in the HCrO$_4^-$ absorbance is attributed to visible light reduction since no change was observed in the dark. The photocatalysis was optimized with respect to the ZnCo$_2$O$_4$ dose and HCrO$_4^-$ concentration, and a conversion of 82% was obtained.

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