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Preparation and characterization study of an olive pomace - polyaniline composite conductor in the recovery of heavy metals by electrosorption and adsorption.

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ABSTRACT/RESUME

Abstract:*In this work, we have prepared a new composite material based on olive pomace (OP) and polyaniline (PANI) by the in situ-polymerization chemical method, demonstrated the ability of our material to conduct current through electrical conductivity tests.We used the spectral analysis techniques to characterize the material as well as Laser Particle Size and scanning electron microscopy to show that the PANI was successfully attached to OP.We have applied our new material in the recovery of Hexavalent Chromium by carrying out electrosorption tests at a positive potential imposed. We compared the recovery with chemical adsorption under the same conditions.*

I. Introduction

Significant processes were widely studied to eliminate heavy metals, pollutants of waste water in high concentrations [1,2] resulting in a remarkable improvement in water quality.Some of these processes are: coagulation and flocculation, floatation, membrane process, adsorption and chemical and electrochemical oxidation processes.Among these techniques adsorption has been shown to be an effective process with its applicability on a large scale to remove impurities from waste waters. However, time required for adsorption is sometimes long [3].On the other hand electrosorption; which is generally defined as potential-induced adsorption of molecules on the surface of charged electrodes is promising technology for faster and more effective removal of pollutants from waste water [4,5]. In this respect, electrosorptionoffers some advantages such as its environmental friendliness, and the in-situ regeneration of adsorbent with less energy consumption, [6,7].Diverse materials as cellulose, rubber, and textile were used as good supports for the electrosorption because of their distinguished physical properties.Nevertheless, we have selected a new material not used yet in this area, namely the olive pomace. Olive pomace is one of the most abundant materials in nature; it possess several advantages such as low cost, low density, non-toxicity, renewable nature, biodegradability [8] It was been reported to be a suitable adsorbent material[9].However, to achieve anelectrosorptionwith natural material, we must make it conductive. To this end the conductive polymer is used.In terms of conductivepolymers, polyaniline (PANI) is extensively studied conjugatedpolymer with particular properties such as good environmentalstability, high electrical conductivity, ease ofpreparation, lowsynthesis costsand reversible acid/base doping/dedopingcharacteristics[10,11] .

The main objective of this work is to fabricate a composite based on olive pomace, tomake it conductiveand to study its applicability in the

electro-enhanced removal of chromium hexavalent from aqueous solution.

II. Experimental

II. 1. Materials and methods

The olive pomace resultsfrom seedscrushed during the production process of the olive oil. They are washed at first several times in the tap water then in the distilled water subsequently, exhausted by the hexane to eliminate the residual oil, washed with some hot distilled water, driedand crushed in a diameter between 500 µ and 1000 µ [12].The chemicals we have used to produce our samples such as Aniline monomer and Ammoniumpersulfate (APS) have been purchased from BiochemChemopharma Inc. and the Hydrochloric acid (HCl) from Chem-lab Inc.

II.2. Synthesis of olive pomace–PANI composites

We have considered two grams (2g) quantity of olive pomacein the preparation of the composites samples according to the In situ polymerization process. We added to this quantity2.0 mol/L of HCl solution dissolving aniline monomer (10:1by molar ratio), stirred for 50 minutes, then added 0.025mol/L of APS aqueous solutiondrop wise to oxidativepolymerize aniline [13]. The mixture was stirred for 20 hours aticetemperature [14]. The reaction product was filtrated and washedwith 0.2 mol/l of chloride acid solution [15], andthen with absolute ethanol till the filtrate become colorless.Finally, the composites were dried at 40 °C in an air oven for 5 hours.We have produced with the same process described earlier six samples by progressively increasing the aniline, HCL, and the persulfate as shown in table 1 bellow.

Table 1. *Samples prepared by In situ polymerization*

Samples	Amount of aniline (mmole)	Amount of HCL (mmole)	Amount of Persulfate Ammonium APS (mmole)	Amount of olive pomace (g)
1	2	20	2.5	2
2	4	40	5	2
3	8	80	10	2
4	12	120	15	2
5	16	160	20	2
6	20	200	25	2

II.3. Characterization

We have applied the Fourier transform infrared spectra (FT-IR) to the samples usingthe EQUINOX-55 FT-IR spectrometer (ὰBrukerlazer) and recorded the Spectra in the range 4000–400 cm^{-1}. As well as the DMAX-Ultima III X-ray diffractometer in the range from 5º to 120º to obtain the X-ray diffraction (XRD) results of the composites.Furthermore, the size distribution of PANI nanoparticles in olive pomace was analyzed by using Gatan Digital Micrograph software. Finally, scanning electron microscopy (Philips ESEEM XL 30 SEM, JEOL, Inc., JSM- 6500F) wasused to characterize the surface morphology andstructure of the samples.The electrical conductivity (EC) ofthe composites was measured at room temperature by the standardfour-probe method. The samples were compressed to13 mm diameter and 0.08–0.2 mm thickness pellets for themeasurements.

III. Results and discussion

III.1. Structure and morphology analysis

III.1.1. FT-IR characterization

The FT- IR spectra of olive pomace (1), PANI-HCl (2) and olive pomace – PANI composite (3) are shown in Fig1. The characteristic broad bandfor O–H group (being in presence of H-band) of olive pomace appears at3330 cm^{-1}[16,17,18],a peak around 1730cm^{-1}due to functional groupC=O of lactones [16] and the band 1234cm^{-1} corresponds to the vibrations of the function OH and NO_2.So the band 1031cm^{-1} indicates the presence of the connections of strain C- OH primary alcohol and C-N.The broad absorptionband between 1200 and 935 $cm^{-1}$is attributed to the contribution ofvarious functional groups, such as C–O and C–O–C.The typical featureof pure PANI is also well known in literature. The peaks correspondingto out of bending vibration ofN–H and C–H band of p-di-substituted benzene appears at 497 cm^{-1} and 790cm^{-1}. The band 1079 cm^{-1}indicates the presence ofthe connections of strain C- OH primary alcohol and C-N.The1278cm^{-1}band corresponds toanNHbendingout of planeandstretching vibrationsofC-O phenolic bondand C-N on thebenzoidecycle[18].The peaks around 1457 and1557 cm^{-1} result from stretching vibration of N–A–N and N=B=Nstructures, respectively (where –A– and =B= stand for benzenoid andquinoidmoieties in the PANI chains) [19,20].The1780cm-1 bandis characteristic of theconducting formofPANI.The characteristicabsorptions of pure cellulose (3331, 1234 and1031cm^{-1}) and PANI (1780,1557, 1457and1278 cm^{-1}) have been both present in the spectrum ofthe composite [15].

The FT-IR spectrum of OP/PANI indicates that the compositing of OP and PANI is successful.

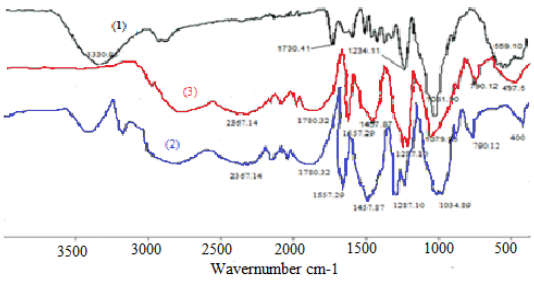

Figure1.*FT-IR spectra of olive pomace (1), PANI-HCl (2) and OP/PANI-HClcompositewith39,42% of PANI (3).*

III.1.2.X-ray diffraction characterization

Figure 2 shows XRD patterns of pure olive stone, PANI-HCl and OP/PANI-HClcomposite.Thediffractogram of olive pomace exhibits a crystalline major peak at2θ=22.4° [21,22],corresponding tothe(200) crystallographic plane which is attributed to typical cellulose I structurealso callednative celluloseconstitutingthe crystalline partofcellulose[23,24].The other constituentsof olive pomaceare predominantlyamorphouswhich is explainedbythe appearanceof the spectrumfrom2θ=22.4°.For the plane PANI, the major diffraction peaks at 2θ=18.5°, 18.6° and 25.2° are consistent with (020) and (200)crystal planes respectively[25,26].Similar to the PANI prepared by conventional methods[8] , the peaks of our PANI 2θ=18.5°, 18.6° and 25.2° are ascribed to the periodicity parallel and perpendicular to the polymer chains of PANI respectively, which indicates that PANI is partially crystalline as previously reported[8].This crystallinityis also dependent onthe acid usedfor synthesisand the degree ofdoping[27].However XRD of OP/PANI-HCl composite reveals broad diffraction peaks typical to those obtained from the pure PANI (at 2θ=25.51°) and the pure olive pomace (at 2θ=22.28°) indicating a successful coating of PANI over olive pomace[27].From the graph, the emergence ofan additionalpeak at15.87° corresponds to the orientation(010)[28].

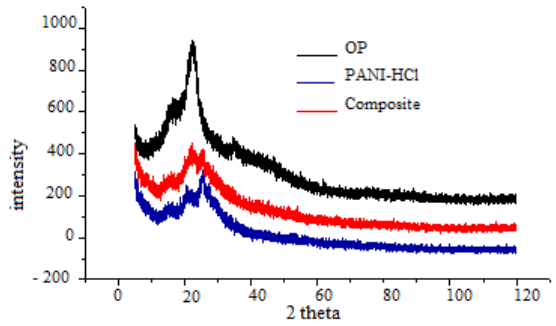

Figure 2. *X-ray diffraction patterns ofolivepomace, PANI and OP/ PANI composite with39,42% of PANI.*

III.1.3.Characterizationby Lasergranulometry

Figures 3.a, 3.b and 3.c showrespectively the size distribution of OP, PANI and OP/PANI compositedistribution of the particle size in olive pomacelocated around24,315µmfor a volume of 50%.From thePANI-HCl, the particle size repartitionwas located around 9,802µmfor 50% of the samples.However, for the OP/ PANI composite, thesize distributionwas40,668µmto50% of the samples.Aftermixing,we notice an increasein particlesize, whichwill allow us tosay that there wereagglomerationbetweenparticles ofolivepomaceand PANI.

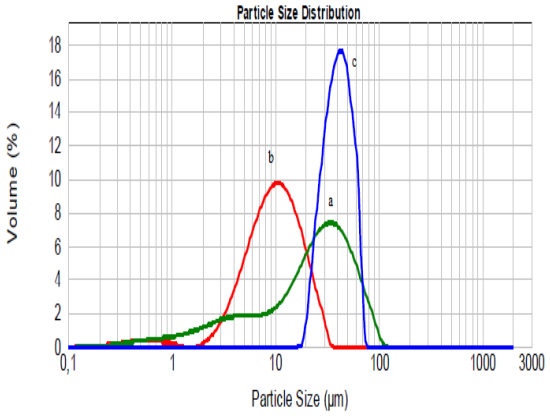

Figure **3.** Particle size distribution of (a): olive pomace, (b): PANI and (c): OP/PANI Composite.

III.1.4.Scanning electron micrographs characterization (SEM)

The percolation limiting thecompositeis related to thehomogeneity of the mixtureof the constituents.
The above resultscan be explainedaccordingtothe composites morphology, based on the characterization by SEM microscopy.Differentimages were taken. Fig. 4.shows an image resulting from SEM-microscopy method.In this image, the spectra (a) of olive

pomace, shows heterogeneous size distribution.Particlegeometryis variable. Thuswe observethe emergence ofapparently well-crystallized grains coexisting withfine powderclusters.For the spectra (b) of PANI, there is a heterogeneous size and form of particles of PANI,as it establishes the existence of a cylindrical form with a smooth surface. Thespectrum (c) shows typical SEM images of the surface of the OP/ PANI composite.We note an apparent uniformity of the surface which leaves us assume that our adsorbent is homogeneous.Also there is an increase in the size of the particle and some resemblance in the form and smoothness of the surfacein comparison with the SEM images of PANI and those of composite. This confirms the results obtained by analysis of Infrared indicate that PANI was able to bind to the surface of olive pomace **[**29].

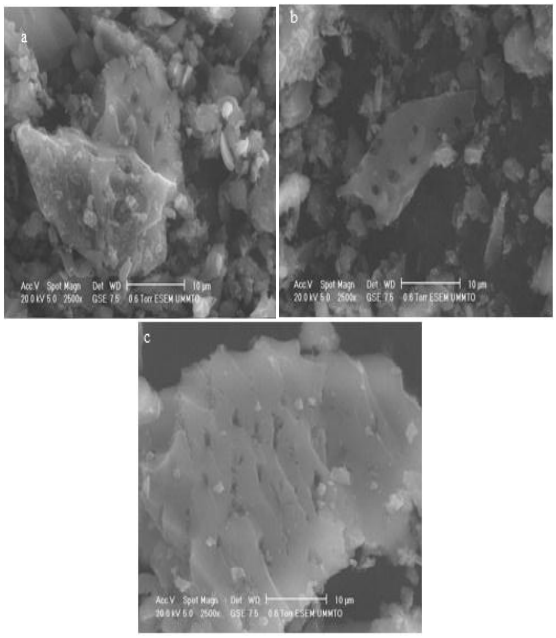

Figure 4. *Scanning electron micrographs of (a) olive pomace (b) polyaniline, and (c) PO/PANI composite.*

III.2. Electrical analysis

III.2.1. Theelectricalconductivity (EC)caractérisation (fourpointméthod)

The electrical conductivity parameter was measured in the case of PANI and the PO/PANI composite prepared with different percentages of PANI, the results are shown in tables 02 and 03 respectively

Table2: *values of electrical conductivity of the PANI-HCL*:

Sample	**e (mm)**	**I (µA)**	**V (volt)**	Resistivity **(Ohm.cm)**	**conductivity (S/cm)**
PANI	0.53	1.989	$1.20.10^{-4}$	14.4851	$6.90.10^{-2}$

The value of the conductivity obtained (σ = 0.06 S/cm) for the synthesized polyaniline, is close to that given in the literature, taking into account the same operating conditions.

Table 3*: values of conductivity of OP/PANI-HClcomposite*

% PANI	σ **(S/cm)**	$Log\sigma$ **(S/cm)**
4.63	1.23.10-4	-3.91
8.23	1.02.10-3	-2.99
19.18	1.5.10-3	-2.82
23.23	2.13.10-3	-2.82
28.15	8.81.10-3	-2.05
39.42	3.20.10-2	-1.49

According to the results found, we note that the insertion of the polyaniline in the olive pomace by the method of in-situ polymerization, confers a conducting appearance.Even low-PANI, composites already express a conductivity of about $1,23.10^{-4}$ S/cm.The samples percolation threshold is observed in the range [25%, 40%] by weight of PANI; it reflects the formation of conjugated system.

III.3.Electrosorptionbehavior

Batch mode electrosorption experiments were performed to investigate the application of the electrosorption process for the removal of chromium (VI) from aqueous solutions, using anOlive pomace-PANI composite with an (PO/PANI) electrode.The exact speciation of the chromium ions depends upon pH, temperature, concentration and the presence of other ions. However according to different authors [30,31], the Cr (VI) is in the form of $HCrO_4^-$, CrO_4^{2-}, or $Cr_2O_7^{2-}$.TheCr (VI) forms would be drawn to the anode. Since these ions are already in a high oxidation state, complexation at the anode, without anodic oxidation is expected. To thiseffectweapply a positive polarization (anodicpotential) to promoteelectrosorptionchromium Cr (VI).In order to efficiently investigate on the relation between the developed conductor material (Olive pomace-PANI composite) and the effect of the electrochemical polarization on the adsorption rate (which is necessary for the accurate design and modelling of adsorption processes), an experiment on adsorption/electrosorption kinetics were carried out starting with the same initial chromium salt concentration of about 20 mg/l of chromium (VI) with anelectrode area of 13.2 cm^2at room temperature and recorded the adsorption at open circuit (OC) and electrosorptionunder 800 mv polarizationas shown in Fig.5.

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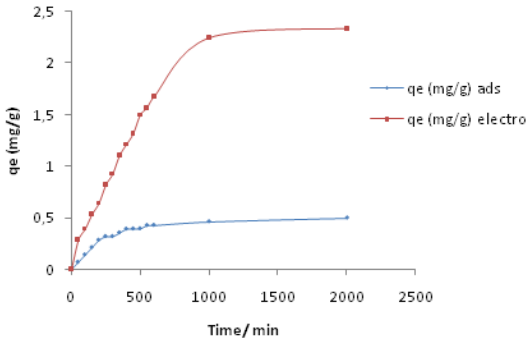


Figure5.*Adsorption kinetics at open circuit (OC) and electrosorption under 800 mv polarizationat C_0: 20 mg/L, T: 25°C, pH: 2.6.*

In order to determine the capacity for electrosorption and regenerability after electrosorption on Olive pomace-PANI composite we have carried another experiment. In this latter, theelectrosorption run from adsorbate solution according to desorption run. The electrosorption data were recorded upon polarization by applying -400 mV for the first 10000 s followed by electrodesorption upon polarization by applying +400mV for the next 1200 s for the possible regeneration of Olive pomace-PANI composite .Then polarization direction was reversed in order to observe the electrosorption. The results are shown in Fig.6.

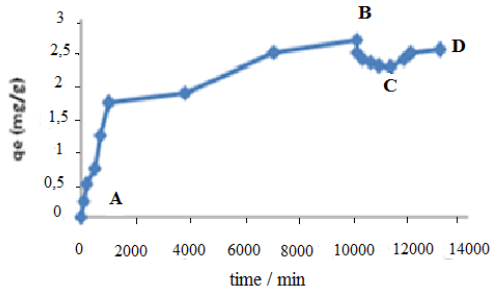


Figure 6.*Successiveelectrosorption/ electrosdesorption (from A to D), electrosorption (from A to B) at+400mV, electrodesorption (from B to C) at -400 mV polarization, and electrosorption (from C to D); mass of OP/PANIelectrodewas 0.2g.*

The electrosorption takes place from A to B upon +400 mV. Adecrease is observed in solution concentration of chromium, resulting in electrosorption of almost 25% of chromium in the first 10000s.When the polarization direction is reversed (-400mV) at point B to C during 1200 s as shown in Fig6,a small amount of Cr (VI) was released during this step. During regeneration step at – 400 mV polarisation, an amount of 0,5 mg g-1of electrosorbed chromium was electrodesorbedinto 800ml of $K_2Cr_2O_7$ solution in 20 min period, corresponding to about 18,5% of the amount of chromium electrosorbed. The remainder is absorbed.A reverse polarization at -1.2 V was required for the partial release of bound Cr (VI).This electrosorptivebehavior of chromium not onlycanbelinked to the electrostaticinteractions betweenChromiumspecies in solution and charged Olive pomace-PANI composite surface in the double layer but alsochemicalcomplexationbetween the dichromate oxyanionswithfunctional groups on the Olive pomace-PANI composite surface, where the relative irreversibility Cr(VI) electrosorption.It is evident that Cr(VI) anions such as dichromate are not removed from the solution by a simple electrostatic (capacitive charging) mechanism. The electrosorption process involves some type of bond formation.Reverse polarizationat -400 mV causedcathodicreductionat the electrodecomplexedwith the Cr(VI) species, therebydestroying the Olive pomace-PANI composite Cr(VI) bonds.After the regeneration, the polarization direction is reversed (+ 400 mV) at point C, the concentration starts to decreasesharply as a result of electrosorption of chromium. However, the adsorbed amount is similar that previously regenerated.

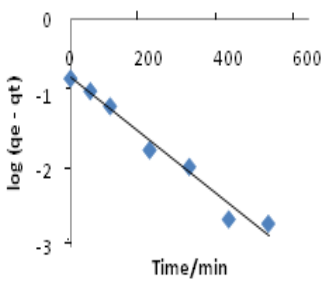
III.4.Comparison of electrosorption with adsorption:

Evaluation of adsorption kinetics is carried out using pseudo-first - order, and intraparticle diffusion models [32, 33].

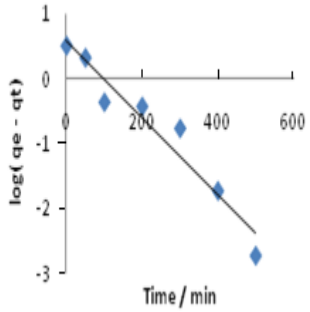
Lagergren pseudo-first order is expressed as:

$$\text{Log } (q_e - q_t) = \log q_e - (K_1/2.303)t \quad \textbf{(1)}$$

Where$q_e$and q_t (mg/g) are the amounts of chromium adsorbent per unit mass of Olive pomace-PANI compositeat equilibrium and at time t, respectively, and K_1(min^{-1}) is the rate constant for pseudo-first-order adsorption. The values of $K_1$and q_e*at* different concentrations can be determined from the slope and intercept when log (q_e– q_t) is plotted against t (Fig.7(a) and (b)).



(a).



(b)

Figure 7.*pseudo-first – order plots for adsorption :* ***(a).*** *under potential condition at 800 mV polarization,* ***(b).****at open electrosorption circuit (OC)*

The rate constants, k1, and corresponding regression coefficients, r, are given in (Table 4) for adsorptionat open circuit (OC) and electrosorption under potential condition at 800 mV polarization.

Table4. *Kinetic parameters of the removal of chromium by adsorption at open circuit andelectrosorptionunder potential condition at 800 mV polarization.*

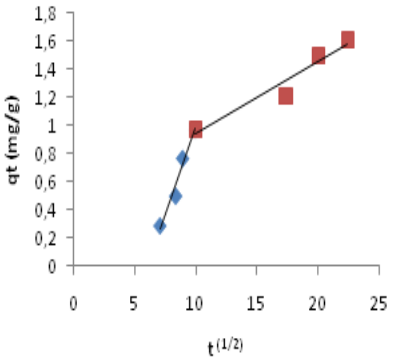
	Adsorption (Open circuit)	electrosorption
Pseudo-first order		
K1 (min^{-1})	0.0092	0.0138
qe (mg/g)	0.465	1.79
r^2	0.976	0.940
$\%R_{eq}$	18.7	86
Intraparticle diffusion		
K_1id (mg g^{-1} $min^{1/(1/2)}$)	0.024	0.243
K_2id (mg g^{-1} $min^{1/(1/2)}$)	0.02	0.051

Positive polarization causes an increase in the rate of adsorption, and equilibrium adsorption capacities compared to open circuit adsorption; 33%,increase was observed in rate constant (k1) and upon +800 mV polarization. This result suggesting an effect of electrostatic interactions by attraction between positively charged of Olive pomace-PANI compositeand negatively charged chromiumionswhichis in the anionic form $Cr_2O_7^{-2}$and also dispersion forces are responsible from the observed enhancement in rate parameter.The intraparticle diffusion model has also been used as a first approach for identifying the limiting adsorption step and the diffusion/transport mechanisms during Chromium adsorption and electrosorption. The intraparticle diffusion model can be represented with equation (2)

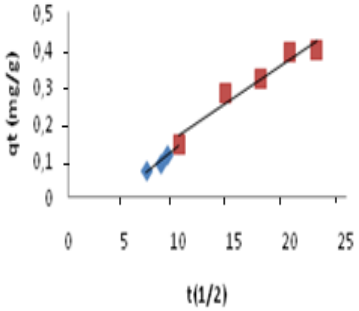
$$qt = k_{id}\, t^{(1/2)} + C_i \quad \textbf{(2)}$$

Where k*id* (mg $g^{-1}min^{-1(1/2)}$) is the measure of diffusion coefficient and Ci (mg g^{-1}) is the intraparticle diffusion constant which is directly proportional to the boundary layer thickness. The diffusion plots of qtvs. $t^{(1/2)}$gives the rate constant, Kid from the slope and Cifrom the intercept.The multi-linear nature of the intraparticle diffusion plots (two differentiated steps were detected in adsorption and electrosorption) indicates the simultaneous occurrence of several adsorption stages.

The first linear step represents the adsorbate diffusion in the boundary layer; the second one accounts for the gradual adsorption stage where the molecules of the adsorbate diffuse through the porosity of the composite electrode (intraparticle diffusion).Fig8(a) and (b) presents respectively the plots of chromium electrosorption upon polarization by applied potential of the order of 800 mV and open circuit (OC) versus $t^{1/2}$on Olive pomace-PANI composite. It can be seen from the figure that data points are linked by two straight lines.



(a)



(b)

Figure8 .*Intraparticlediffusionplots:* ***a).****fortheElectrosorptionupon polarization by applyingPotential of the order of 800 mV.* ***b).****for the adsorption at open circuit (OC).*

For the electrosorptionupon polarization by appling potential of the order of 800 mV, the external mass

transfert step occurring at low contact times during the retention of chromium was further explored by determining the rate constant of external mass transfer K_1id (diffusion in the boundary layer) from the initial period of adsorption. The second distinctive linear region marks the stage where intraparticle diffusion starts controlling the rate of adsorption (K_2id). K_1id and K_2id are included in Table 2. It is clear the intraparticle diffusion (K_2id) islower than the external mass transfer (diffusion in the boundary layer). This indicates that the external mass transfer is not the rate-controlling step forelectrosorption of chromium. Our results are similar of the work concerning electrosorptionof basic dyes and pyridine from aqueous solution onto activated carbon cloth electrode [35]; showing that this electrosorption is not diffusion controlled in the boundary layer.Whereas for the open circuit (OC) adsorption, we see that the rate of constant for the external mass transfer step and the intraparticle diffusion are the same (table 4).However, the intercepts of the plots do not pass through the origin. This indicates that intraparticle diffusion is not the only ratelimiting step, but also other kinetic models may control the rate of adsorption [32]. The rate limiting step maybe a complex combination of chemisorption and intraparticletransport [32]
(Table 4).

IV. Conclusion

In the present paper, Olive Pomace /PANI conductive composites were prepared by chemical oxidative polymerization of aniline with natural olive pomace.The FT-IR spectrum of OP/PANI indicates that the compositing of OP and PANI is successful. On the other hand, XRD of OP/PANI-HCl composite reveals broad diffraction peaks typical to those obtained from the pure PANI and the pure olive pomaceindicating a successful coating of PANI over olive pomace.
Agglomerationbetweenparticles ofOlivePomaceand PANI is determined by The Laser granulometrywhich indicatesthat aftermixing;there was an increaseinparticlesize.
Scanning electron micrographs characterization (SEM) confirms results obtained by FT-IR and DRX Laser granulometry analysis. These results indicate that the PANI is successfully fixed on the OP.
The conductive composite wasused in the chemical and electrochemical removal of Cr(VI) from aqueous solutions. The Cr (VI) is in the form of $HCrO_4^-$, CrO_4^{2-}, or $Cr_2O_7^{2-}$, they would be drawn to the anode. Since these ions are already in a high oxidation state, complexation at the anode, without anodic oxidation is expected. To this effect we apply a positive polarization (anodicpotential) to promote electrosorption chromium Cr (VI).An experiment was carried out in which electrosorption run from adsorbate solution were followed by an desorption run indicating that this electrosorption behavior of chromium can also be linked to the chemical complexation between the dichromate oxyanions with functional groups on the Olive pomace-PANI composite surface.Finally, the kinetic data are fitted well with the pseudo-first-order model and intraparticles diffusion model

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