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RECOVERY OF SAWDUST DISCHARGES FOR THE TREATMENT OF INDUSTRIAL WASTEWATER

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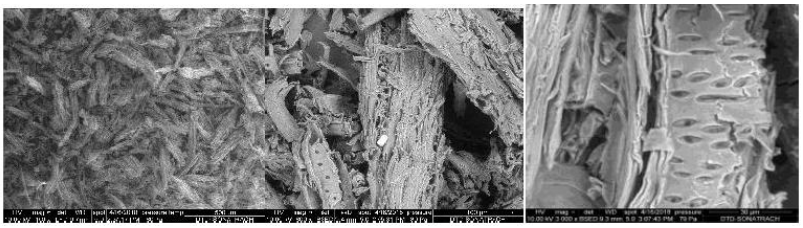
Large capacity and continuous operation of many petroleum units produces several amounts of solid, liquid and gaseous wastes including oily sludge, wastewater, volatile organic compounds, heavy metals, and others. However, these discharges can have serious effects on the environment, human health, terrestrial and aquatic fauna and flora. In this work, a biodegradable natural material of wood flour (WF) obtained from an Algerian separation center was used for treating petroleum wastewater. Our adsorbent has undergone to post-treatment steps (drying, grinding and sieving) before use, in order to obtain a thin material with a high adsorption capacity. After characterization of both samples (adsorbent and wastewater), variation of operating parameters (WF amount mass, pH, temperature, contact time and agitation speed) was considered. Under favorable conditions of pH = 7, mass of WF = 10g, T = 36 °C, V = 250 RPM and t = 30 min, our sample was able to eliminate a large quantity of organic matter, SM, dissolved mineral matter including heavy metals, sulfate, nitrite and phosphate ions. So, the characterization analyses of the WF sample after adsorption process have proved these results.



INTRODUCTION

Petrol and its fractions are considered as essential components of wide variety of materials,[1] it presents a significant source of the world's energy production.[2,3] However, several solid, liquid and gaseous wastes are generated during the processes of their exploration and exploitation, these discharges can provoke harmful impacts on the environment and on human health, by direct pollution of air,[4] soil and surface water, or by propagation and infiltration to rivers, lakes and groundwater causing a diffuse pollution of water.[1,5,6] Therefore, it is necessary to consider the management of this waste by applying effective treatments (biological, physical, chemical, thermal and other treatments) for remedy these dangerous problems.[5] Among these techniques, adsorption treatment is the most recommended technique for the purification of several types of pollutants such as heavy metals,[7,8] reactive dyes,[9–11] hydrocarbons[12] using different types of adsorbents, such as activated carbon,[13–15] clays,[16–18] metal oxides,[19] vegetable fibers[20–22] or synthetic polymers such as composite and biocomposite polymers.[23]

Indeed, wood is a natural composite material that is composed by fibers of complex structures, which

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are grouped essentially into three types of amorphous polymers; lignin, hemicelluloses and cellulose.[24,25] Wood also contains a small amount of wide variety structure of organic compounds with lower molecular weights. Nevertheless, the industrial transformation of wood generates wastes in different forms of small pieces or sawdust, which can be valorized as; an energy source by its combustion, or as a clean adsorbent thanks to their high adsorption capacity.[26]

Wood sawdust (WS) offers a promising and sustainable solution against water contamination, notably heavy metal, thanks to its various advantages, including low cost, availability, porosity, mechanical properties, easy chemical modification, and easy recycling. Furthermore, chemical modification treatments can improve its adsorption capacity.[27,28] The removal of several pollutants from water using WS was reported in several previous studies. In the works of Rani *et al.* (2023), the sawdust chemically modified by formaldehyde and by sulfuric acid were demonstrated an effective elimination of Zn(II) from aqueous solution with an equilibrium adsorption capacity of 45.87 mg/g.[29] Indeed, a high adsorption of Cd(II), Cr(III) and Pb(II) at activated WS by phosphorus oxychloride, reaching capacities of 244.3, 325, and 217 mg/g respectively.[30] So, in the studies of Nurnabi *et al*. (2020), the adsorption of Pb (II) and Cu (II) at modified WS of *Albizia lebbeck* by sodium methylate, epichlorohydrin and n-dodecylamine was important with the calculated maximum sorption capacities of 17.37 mg/g and 9.4 mg/g for both ions respectively.[31]

Moreover, modified WS can be used in the removal of oil from wastewater by hydrophobic magnetic composite, this material was composed by WS and magnetic clusters coated with a silicon dioxide (SiO_2) layer. The resulted material showed high removal effectiveness (93–97%) against the four types of tested oils (fresh engine oil, engine oil, vacuum pump oil and cooking oil) with adsorption capacities ranging from 1.93 to 2.32 g/g.[32] In the studies of Sadaqat *et al*. (2025), the WS was used as a filter synthesized with different material; Red clay, WS, WS ash, and calcium silicate for the treatment of textile and tannery industry wastewaters. The filter has presented a dyes elimination percentage of 75% from textile effluent and 65.89% of chromium removal from tannery effluent.[33]

The aim of this work is the estimation of WF effectiveness for the treatment of an industrial effluent obtained from an oil industry. For this reason, a sample of WF obtained from discharges of a wood carpentry workshop was used as adsorbent material. This sample was purified and subjected to various characterization analyzes namely; Scanning Electronic Microscopy (SEM), Fourier-Transformed Infra Red (FT-IR), X-ray Diffraction (XRD), X-ray fluorescence (XRF) and granulometry. On the other hand, the sample of petroleum wastewater was examined by different analyzes to determine the rate of suspended matters (SM), conductivity, turbidity, chemical oxygen demand (COD), biological oxygen demand (BOD), FT-IR, determination of some mineral pollutants concentrations (sulfates, phosphates, nitrites and Zn, Fe, Pb and Cd). Subsequently, the petroleum effluent sample was treated by WF adsorbent using a Jar test. Optimization of some operating conditions was also considered.

MATERIALS AND METHODS

Samples and reagents

The wastewater sample was taken from the fluid separation center of the petroleum installations in Ain Amenas region, Algeria. The samples were collected in PVC (Poly Vinyl Chloride) plastic bottles previously washed and rinsed with water to be examined. In the other hand, the wood samples were obtained from the discharges of a woodworking workshop located in Boumerdes, Algeria. This sample is a mixture of different types of wood that are obtained from different trees of the region (pine, almond, eucalyptus).

The reagents used in this work are specific for the apparatus according to the analysis to be performed, which are; reagents for measurement of COD using adapter kit (Cat no = 44799-00) of COD for DR/2010 spectrophotometer device, Capsules of Phos Ver3 for measurement of phosphorus ions concentration, Nitri Ver3 for measurement of nitrites ions concentration, and Sulf Ver4 for measurement of sulfur ions concentration. All these reagents are specific using a HACH DR 2010 spectrophotometer. In addition, solutions of NaOH and HCl (both reagents are obtained from Sigma Aldrich supplier) were used at 1M concentration.

Preparation of WF sample

The wood sample has undergone to series of processing step prior their use, in the first time the sample was dried at 50 °C–60 °C in oven (Memmert mark) overnight. After that, the wood sample was

grinded using Retch crusher device and sieved in a series of decreasing sieves, in order to obtain very fine particles. The sample was washed several times with distilled water and then with ethanol (obtained from Sigma Aldrich supplier) for eliminate all organic matters traces and at the end twice time with distilled water. In the next step, the sample was filtered and dried at oven at 50 °C for two days.

Characterization of the crude WF sample

Several characterizations analyses were performed such as; size determination by laser granulometry of type LA 950 device, mineralogical structure determination by X-ray fluorescence (XRF) spectrometer of the type S8 TIGER type XR and chemical structures measurement by Fourier transformer infra-red (FTIR) spectrometer using JASCO FTIR 4100 device. Moreover, the morphological surface and crystalline form were examined by scanning electron microscopy (SEM) of Quanta 650 type and X-ray diffraction (XRD) using Philips X'Pert-MPD X-ray diffractometer respectively.

Preliminary examination of the crude wastewater sample

The wastewater sample has undergone to preliminary examinations of color, odor and SM. This latter, was determined by two distinct methods; vacuum filtration and centrifugation. In addition, the turbidity was measured by optical turbidimeter of HACH 2100N. Moreover, pH and conductivity of the effluent and all other solutions were determined by pH meter-conductometer of HACH HQ 40d.

Chemical analyzes of organic pollution

COD measurement

The chemical oxygen demand (COD) was measured using standard colorimetric method of reflux heating (back dosing of potassium permanganates). The principle of this method consists to introduce 2 mL of wastewater sample previously stirred in inclined COD-specific tube and placed in the COD-reactor previously heated at 150 °C for 2 hours. After that, the tubes were cooled at the room temperature and then we have measured their absorbance by spectrophotometry of HACH DR2010 type at a wavelength of 620 nm.

BOD_5 measurement

The biological oxygen demand was measured after 5 days of incubation that was considered as a significant period for global organic matter biodegradation process, it expressed by mg of O_2/L. Therefore, BOD_5 value could be preliminary estimated from the measured COD value (table .01) that can be used for the test sample amount determination.

Table 1

Recommended BOD_5 volumes values of the test sample according to COD values

COD Margine	Test sample volume (mL)	Conversion coefficients
0–40	432	1
0–80	365	2
0–200	250	5
0-400	164	10
0–800	97	20
0–2000	43.5	50
0–4000	22.7	100

The wastewater was introduced in the dark flask containing a stirrer and covered up by a rubber stopper containing two NaOH pellets. After one hours of stirring at 20 °C, the time measurement was triggered and the value of oxygen concentration was daily noted. At the fifth day, the value on the scale was multiplied by the conversion coefficient that corresponds to the oxygen consumption expressed by mg/L. The measurement was effectuated by manometric apparatus of type of VELP SCIENTIFICA. So, the microorganisms responsible of this degradation are present in the wastewater sample, they can consume the dissolved oxygen expressed by mg/L, which is constantly replaced by oxygen from the air volume below the sample. The carbon dioxide formed is trapped by potassium hydroxide placed in the reservoir attached to the neck of the bottle. Each bottle is equipped with a pressure gauge that records the pressure drop caused by the consumption of O_2 and the binding of CO_2.

Chemical analyzes of mineral pollution

Dosage of sulfates, nitrites and phosphates ions

For the dosage of sulfates, nitrites and phosphates, 25 mL of the wastewater was introduced in the cell of the spectrometer apparatus with one capsule of Sulfa Ver4, Nitri Ver3 and Phos Ver3 respectively.

Therefore, sulfates ions react with barium ions of the sulfa Ver4 reagent and product insoluble barium sulfate that proportional to the sulfate concentration. The barium sulfate can be examined by spectrophotometer at wavelength of 450 nm.[34]

Moreover, for high nitrites concentrations, ferrous sulfate reduces nitrite ions to nitrous oxide in acid medium to produce a greenish-brown complex can be determined by colorimetric at a wavelength of 585 nm.[34]

Furthermore, orthophosphate reacts with molybdate in acid medium and thus products phosphomolybdate complex, which was reduced by ascorbic acid to molybdenum blue. This later can be dosed by spectrophotometer at wavelength of 890 nm.[34]

The lecture of all these analyses were performed at UV-Visible spectrophotometer of type of HACH DR/2010.

Heavy metal dosage by atomic absorption

Some heavy metals were analyzed in our sample of wastewater, there are Pb, Fe, Zn and Cd. For that, the wastewater sample was aspired at a constant flow rate in a pneumatic nebulizer. It was sent in the form of a mist through a flame composed by acetylene/air. At certain sufficient temperature, the ions in the solution pass to atomic state (atomization) that are subjected to specific wavelength of radiation. An electronic signal was resulted from light amount absorbed by the atom. The analyses were performed by a device of Analyst 700 PERKIN ELMER Atomic Absorption Spectrometer.

Wastewater treatment by WF

The mixture solution was introduced in a series of five beakers containing 200 ml of wastewater with a certain quantity of our adsorbent material under well-defined operating conditions, using Jar test to maintain these stable conditions throughout the process.

Indeed, the experimental conditions of coagulation-adsorption processes were optimized such as; initial WF mass, temperature, pH, rotator shaking rate and contact time. Thereby, after each optimization assay, the solution was decanted and the liquid fraction was analyzed by determination of different ions concentrations (SO_4^{2-}, NO_2^-, PO_3^{2-}, Cd, Pb, Zn and Fe). Also, turbidity and conductivity were determined too. Moreover, for the parameter of WF mass, COD and BOD_5 were dosed.

After optimization of these conditions, an adsorption test under favorable conditions was carried out. Thus, the solid fraction was recovered, dried at room temperature and subjected to SEM and FTIR analyzes.

Parametric study of coagulation and adsorption of pollutants on WF

In this section, we have determined some pollution indicators, namely turbidity, conductivity, residual concentrations of nitrites, sulfates, phosphates and some heavy metals during all the optimization experiments (initial WF amount, pH, temperature, shaking rate and time), in order to select the optimal parameters for a significant adsorption. The operational conditions fixed for each experiment are described in the following sections and the results of each pollution index are presented in Fig. 5.

So, determination of optimal WF mass for the wastewater treatment aims to show the sufficient amount that can eliminate a maximum quantity of pollutants from the medium. For this experience, different initial amount of WF (1 g, 2 g, 5 g, 10 g and 20 g) was put in 5 beakers, which contained respectively 200 mL of petroleum wastewater, maintaining the other operational parameter at; pH = 6.63, T = 25 °C, t = 1 h and shaking rate = 250 RPM.

Therefore, medium pH is an important parameter that can affect the removal reactions of several types of pollutants. For our assay, pH of the wastewater solution was varied in the range of; 3, 5, 7, 9 and 11 under ambient temperature. So, the pH was adjusted by hydrochloric acid solution and sodium hydroxide solution. Thus, the mixture of the wastewater samples and 10 g of WF are put in beakers of the Jar test under a shaking rate of 250RPM during 1 hour.

For the third parameter, the adsorption process was studied at different temperatures (16 °C, 26 °C, 36 °C, 46 °C and 56 °C) under the operational conditions of (pH = 7, shaking rate = 250 RPM and WF mass= 10 g) with wastewater volume of 200 mL.

Moreover, agitation shaking rate effect on adsorption capacity has been studied. This experience allowed us to demonstrate the wood adsorption capacity of SM and dissolved mineral matter at different rotator shaking. Therefore, the stirring speed was varied from 50 RPM to 300 RPM while maintaining the other parameters at 10 g of

WF mass, pH = 6.63, T = 25 °C, t = 1 h with 200 mL of the sample to be treated.

The last tested factor is the contact time that is considered as an important parameter of the adsorption and SM coagulation kinetics. So, the contact time was varied at 5 min, 10 min, 20 min, 30 min and 60 min, and the other parameters were fixed at (pH = 6.63 WF mass = 10g T = 25 °C, shaking rate = 250 RPM) with 200 mL of the wastewater sample. All optimization tests were realized in a Jar test.

At the end of each optimizing experiment, the mixture was filtered and analyzed in order to determine different pollution indices.

All these manipulations are effectuated in three replicates, the values given in the results are the average between the three obtained values.

Results expression

The rate of suspended matter (SM) found by filtration is given in Eq. 01.

$$SM = \frac{(m_2 - m_1)}{V} \times 10^3 \text{ (mg/L)} \quad (01)$$

where m_1 – the mass of empty filter paper (mg); m_2 – the mass of full filter paper (mg) and V – the volume of test sample (mL).

Otherwise, the rate of total SM found by centrifugation is expressed by Eq. 02.

$$SM_{total} = (M_2 - M_1) \times \frac{1000}{V} \text{ (mg/L)} \quad (02)$$

where M_1 – the masse of empty capsule; M_2 – the mass of full capsule after drying at 105 °C and V – the volume of test sample (mL).

Moreover, the rate of mineral SM found by centrifugation is given in Eq. 03.

$$SM_{mineral} = (M_3 - M_1) \times \frac{1000}{V} \text{ (mg /L)} \quad (03)$$

where M_3 – the mass of full capsule after mineralization at 525 °C.

Indeed, the difference between total *SM* and mineral *SM* is considered as organic SM, expressed by Eq. 04.

$$SM_{organic} = SM_{total} - SM_{mineral} \quad (04)$$

The necessary volume for the BOD_5 sample test is calculated from *COD* value according to Eq. 05.

$$V_{BOD_5} = \frac{COD \times 3}{4} \quad (05)$$

RESULTS AND DISCUSSION

WF sample characterization

Our wood discharges sample is brown solid, after its grinding and sieving, the sample became finer, in the form of flour. So, the measurement of particles sizes by granulometry, presented in the spectra of the Fig. 1, allowed calculating the average and median particle diameter which are equal to 193.8395 µm and 168.683375 µm respectively.

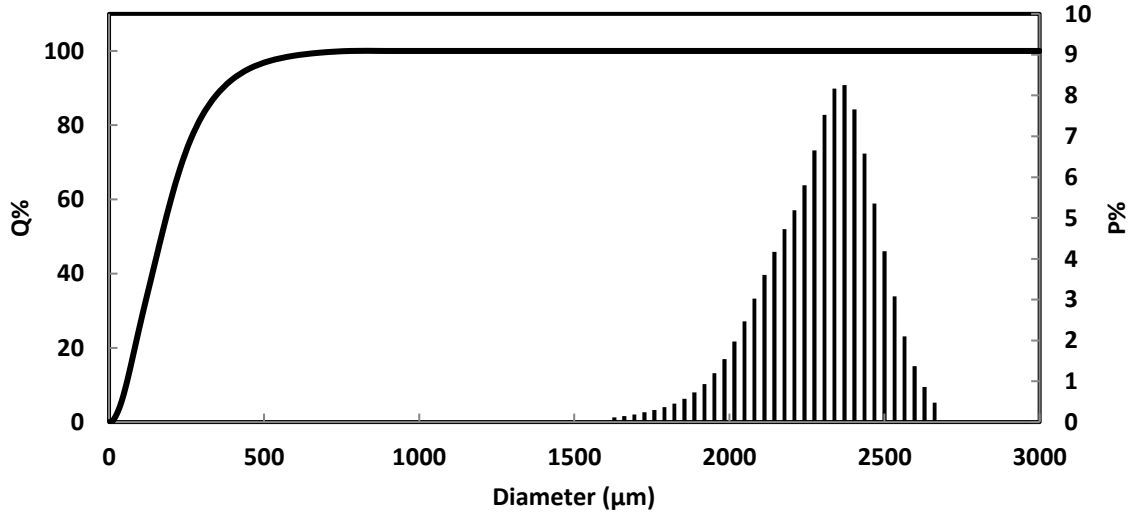


Fig. 1 – Granulometry spectrum of WF sample.

In the other hand, morphological examination of the sample has demonstrated that the surface area is uniform and homogeneous. Indeed, the WF sample presents intercellular spaces and porous cavities in

their surface, which were clearly appeared in the Fig. 2B and 2C at a magnification of × 800 and ×3000 respectively. These pores are considered as active sites of adsorption. In addition, the natural fibers appeared in the images are linked by lignin and noncellulosic materials in the sheet form.[35–37]

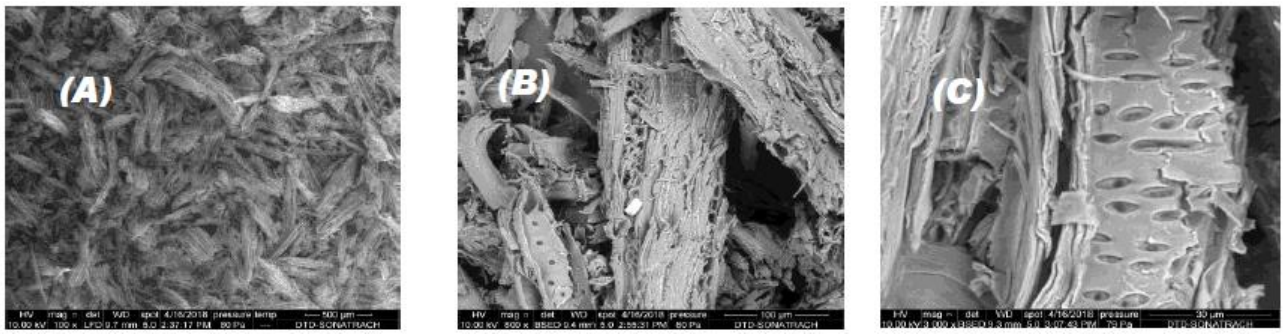


Fig. 2 – SEM micrographs of WF at different magnifications: A) × 100; B) × 800; C) × 3000.

Table 2

Mineralogical composition of WF ash (%)

Mineral	(%)	Mineral	(%)	Mineral	(%)	Mineral	(%)
CaO	0.24	**Al_2O_3**	0.01	**MnO**	0.002	**ZrO_2**	<0.001
SiO_2	0.06	**TiO_2**	0.01	**Cr_2O_3**	0.001	**MoO_3**	<0.001
MgO	0.04	**Fe_2O_3**	0.01	**NiO**	<0.001	**BaO**	<0.001
SO_3	0.03	**Na_2O**	<0.01	**CuO**	<0.001	**PbO**	<0.001
Cl	0.02	**P_2O_5**	0.0076	**ZnO**	<0.001	**Nb_2O_5**	<0.001
K_2O	0.01	**WO_3**	0.0026	**Rb_2O**	<0.001	**SrO**	<0.001
		V_2O_5	<0.001				

Moreover, the chemical analysis of wood ash sample, realized by XRF, are presented in the Table 2, with the fire loss = 99.57%.

According to Table 2, our WF sample is rich in mineral salts principally calcium, with other mineral in lower proportions, such as; silica, magnesium, sulfur, chloride, potassium and aluminum. The other elements, such as; Na, Sr, Zr, Pb, Ni, Cu and Mn are considered as trace elements because they presented very low percentage (≤ 0.001%). Otherwise, the loss of fire is very important indicated that the WF is rich in organic matters. These later were analyzed by FTIR, the resulted spectrum is presented in Fig. 3.

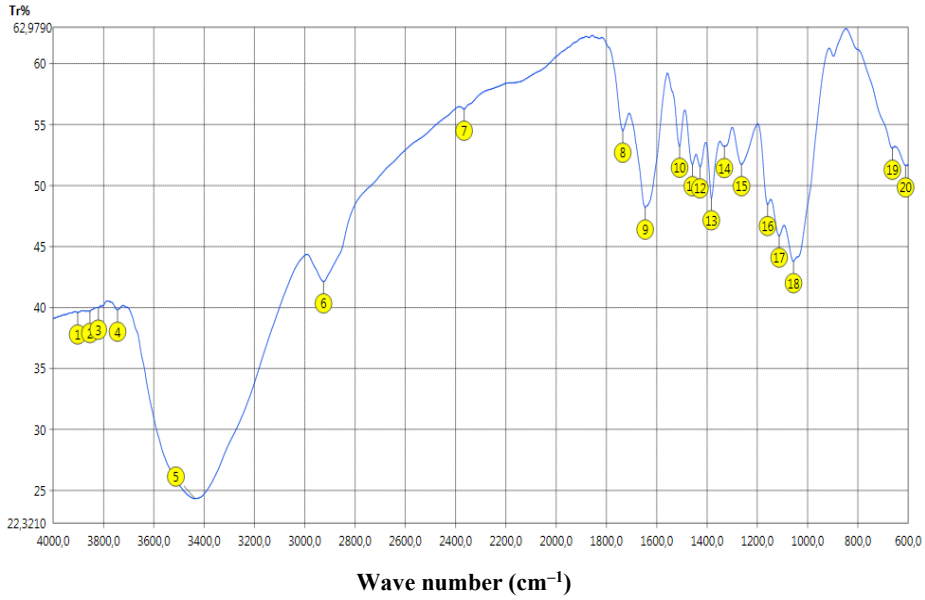


Fig. 3 – FTIR spectrum of the raw WF sample.

According to the FTIR spectrum, a large band with average intensity at 3436 cm^{-1} (peak 5) was noticed that corresponds to stretching vibration of the link O–H associated to alcohol or phenol function of the lignin and hemicelluloses. In the other hand, the asymmetric stretching vibration of systematic C–H links of $–CH_3$ radicals were appeared at 2925 cm^{-1} and 2366 cm^{-1} (peaks 6 and 7 respectively). Peaks of low intensities were observed at 1159 cm^{-1}, 1113 cm^{-1} and 1065 cm^{-1} (peaks 16, 17 and 18 respectively) which correspond to stretching vibration of C–O of primary alcohol. Moreover, the average peaks appeared at 1646 cm^{-1}, 1458 cm^{-1} and 1428 cm^{-1} (peaks 9, 11 and 12 respectively) are due to the stretching vibration of C=C function. Otherwise, the peak of 1509 cm^{-1} (peak 10) demonstrates the existence of aromatic ketone C=O. Thus, the bands observed at 1383 cm^{-1}, 1331 cm^{-1} and 1263 cm^{-1} (peaks 13, 14 and 15 respectively) are attributed to C–O link vibrations of lignin functions and the peak of 1265 cm^{-1} corresponds to valence vibration of C–O and C–O–C of the cellulose.[35–37]

Concerning mineralogical phase's identification, the XRD analysis of our WF was presented in the diffractogram of the Fig. 4.

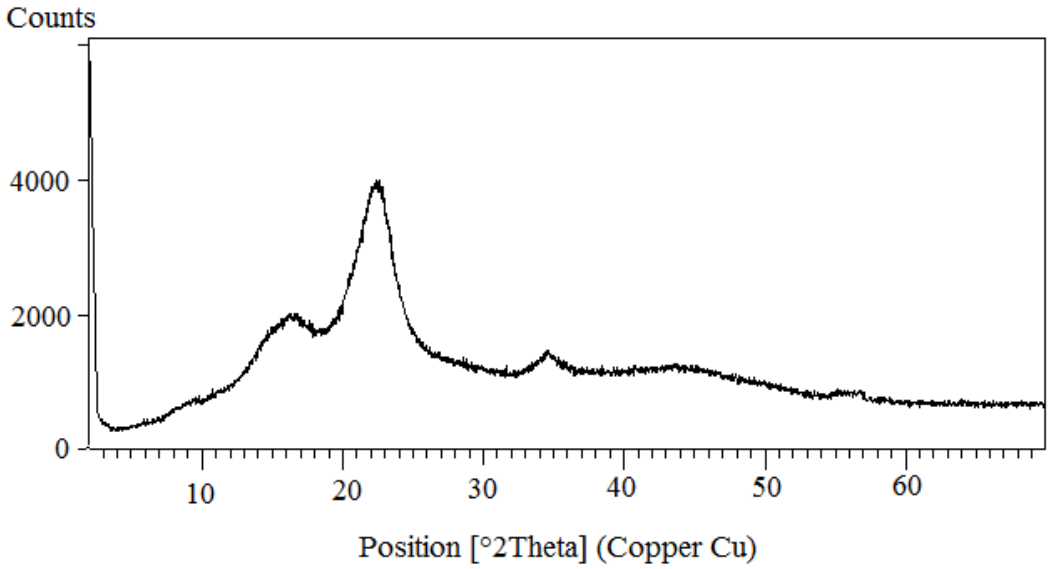


Fig. 4 – XRD patterns of the crude WF sample.

The diffractogram presented in the Fig. 4 demonstrates that the presence of a peak with weak intensity at the neighborhood of 34.6 A°, and other peaks with strong intensities at the neighborhood of 16.5 A° and 22.4 A°. These peaks correspond to the following crystal planes of (002), (110) and (004) respectively which are associated to cellulose structure.[35,38]

Primary characterization of wastewater sample

Our sample is dark brick red in color due to the presence of different hydrocarbons fractions and ferrous iron. Moreover, their odor is similar to the smell gasoil that is related to sulfur, aromatic and aliphatic hydrocarbons. These later are the major pollution precursor.

The rate of SM of our sample determined by two distinct methods of filtration and centrifugation, are equal to 0.3 g/L and 1.7 g/L respectively. In addition, turbidity value is 247 NTU that is superior to the norm value of 50 NTU; therefore, our sample is overloaded by SM.

Furthermore, conductivity of the solution is 103.4 µS/cm indicated that our sample is moderately loaded with dissolved mineral matter because this value is comprised between 100 and 300 µS/cm. Indeed, the conductivity measurement allows evaluating approximately the global mineralization of the water, so conductivity between 0 and 100 µS/cm indicates that the mineralization is low and the values conductivity superior to 300 µS/cm indicating that the mineralization is very high. Moreover, the pH of our sample is slightly acidic that is equal to 6.63.

In the other hand, organic pollution of our sample was determined by COD and BOD_5 tests,

which consist of the measurement of equivalent oxygen to organic matters amount capable to be oxidized by chemical reagents (Eq. 06) or by microorganisms (Eq. 07) respectively.

$$OM + yK_2Cr_2O_7 + zH_2SO_4 \rightarrow CO_2 + vH_2O + nCr_2(SO_4)_3 + mK_2SO_4 \quad (06)$$

$$OM + \text{microorganisms} + O_2 \rightarrow CO_2 + H_2O + \text{energy} + \text{biomass} \quad (07)$$

So, the COD and BOD_5 values of our sample are respectively equal to 24000 mg/L and 184.7 mg/L.

Concerning mineral pollution, concentrations of some ions that can present certain toxicity are determined, such as; phosphate, nitrites and sulfates, obtained values are 7 mg/L, 491 mg/L and 90 mg/L respectively.

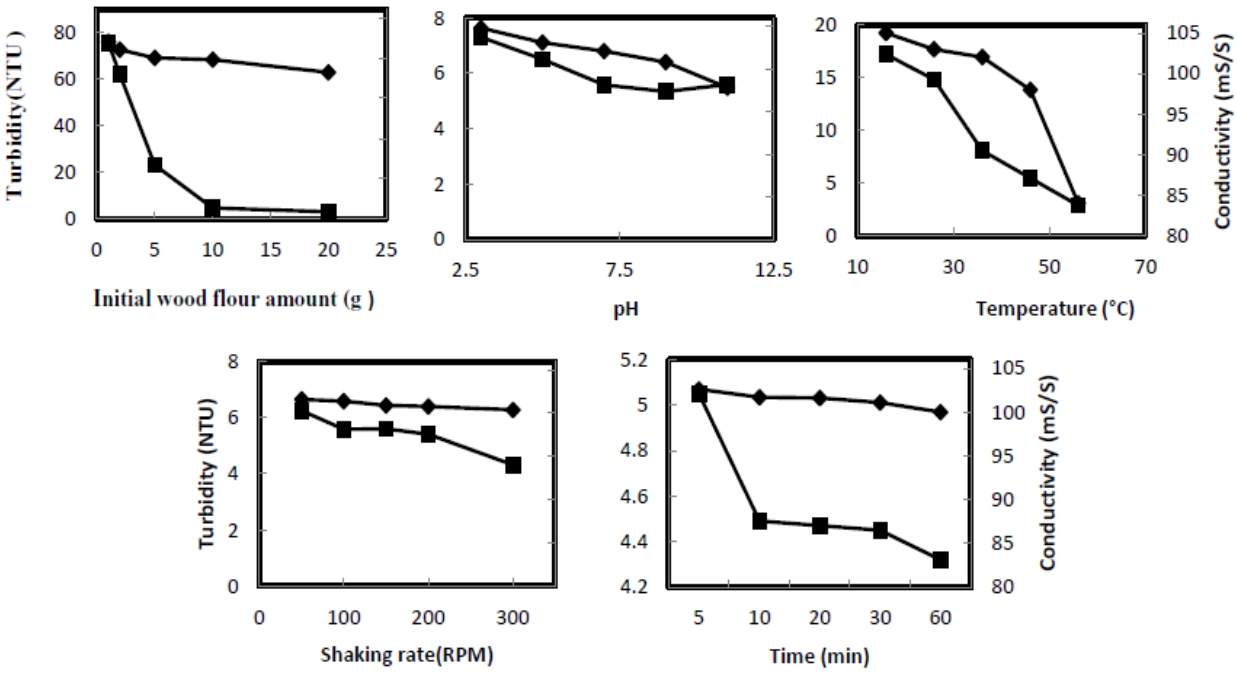
According to these results, our sample is rich in nitrites and sulfates because their concentrations exceeded the regulatory standards values of wastewaters. Similarly, for phosphate ions, despite their concentration is lower that nitrates and sulfates but it was considered as a pollutant concentration because the limit value is around 3 mg/L.

These parameters are similar with that found in several researches because the wastewater generated from petroleum industries is frequently surcharged with aromatic and aliphatic hydrocarbons and other harmful compounds such as; heavy metals, nutritive matters, phenols, aromatic hydrocarbons, heterocyclics, naphthalene and other. These matters can present a several dangers for aquatic and terrestrial ecosystems.[39–42]

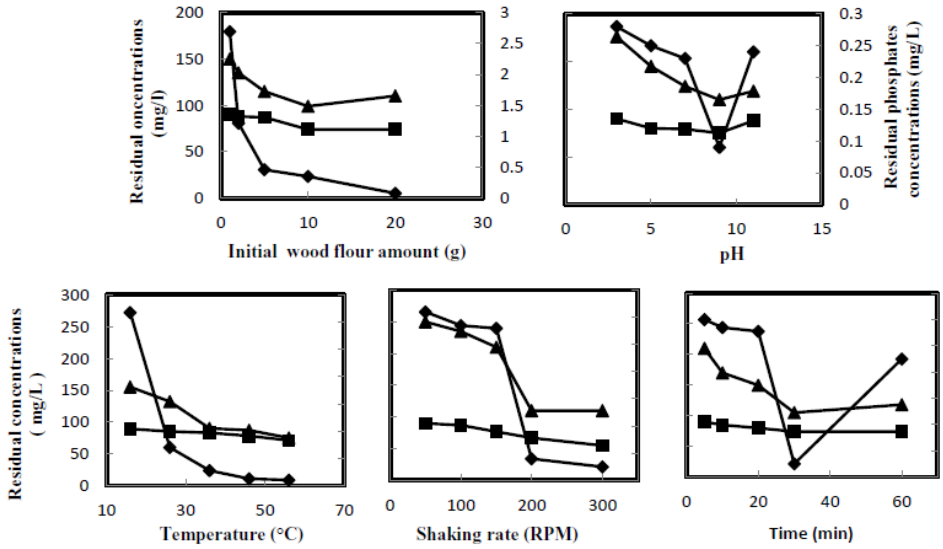
Parametric study of coagulation and adsorption of pollutants on WF

The results of operational conditions optimization of the parameters cited below (in material and methods section) are exploited in the curves presented in the Fig. 5.

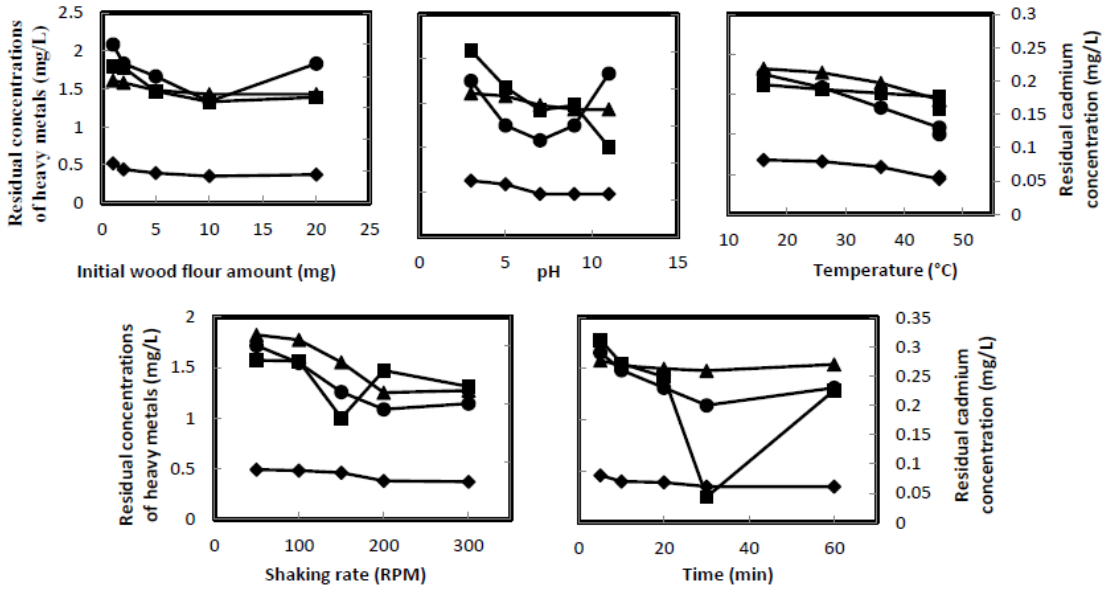
According to the Figs. 5A, 5B and 5C, the optimal parameters that gave the most important results about the pollutants removal are; initial mass of WF = 10 g/L, pH = 9, T = 36 °C, shaking rate = 250 RPM and contact time = 30 min. So, at these parameters values the conductivity and turbidity were notably decreased and the nitrites, sulfates and phosphates concentrations have effectively removed. Similarly, for the tested heavy metals, their elimination was more important at the selected parameters in above.



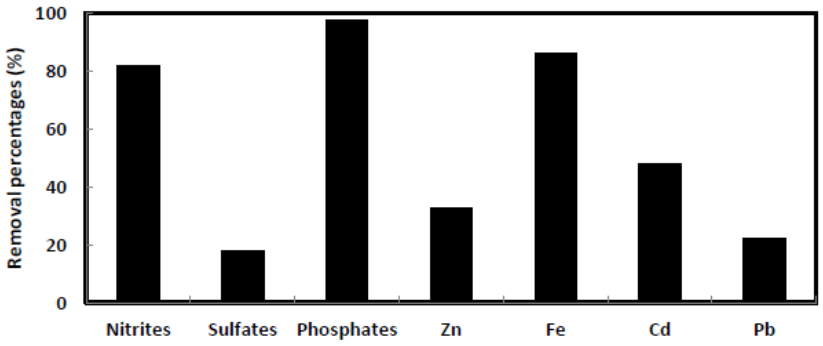
A): (■) turbidity; (♦) conductivity



(B): (▲) nitrites; (■) sulfates; (♦) phosphates



C): (▲) Pb; (■) Fe; (♦) Zn; (●) Cd.



D)

Fig. 5 – Parametric study of coagulation and adsorption of pollutants on WF: A) – turbidity and conductivity measurements during the optimization experiments; B) – residual concentrations of nitrites, sulfates and phosphates measurements during the optimization experiments; C) – residual concentrations measurements of some heavy metals during the optimization experiments; D) – removal percentages of each pollution factor at the optimum operational conditions.

So, in the Fig. 5A, conductivity and turbidity values measured at the tested WF amounts were decreased from 103.4 mS/s to 99.8 mS/s and from 247 NTU to 4.73 NTU respectively. These results demonstrate that mineral matters and SM were effectively eliminated. For the pH effect, the minimum values of these two parameters were observed at neutral and basic medium, where their values are 5.59 NTU and 5.34 NTU for the turbidity at pH of 7 and 11 respectively. Moreover, we have noticed that the increase in temperature results a decrease of the turbidity and the conductivity until attain their minimal values of 2.88 NTU and 84 mS/s respectively at 56 °C, indicating that our process is exothermic. In the other hand, the tenor of turbidity and conductivity decreases with increasing of shaking rate that implies a decrease in suspended and mineral matter concentrations in the aqueous medium. Likewise, for the time factor, turbidity and conductivity decreases significantly with increasing of contact time until 10 min, where they took the values of 4.49 NTU and 101.7 mS/s respectively, after this period the turbidity has reached an almost stable phase and the conductivity has decreased more slowly. Removal of mineral and SM was studied by several previous research using different material and methods. Likewise, use of modified eucalyptus Kraft lignin for the elimination of nickel and cadmium from wastewater was investigated in the literature and gave an important adsorption yield of 96.1% and 80.4% respectively. However, the non-treated Kraft lignin has given an adsorption percentage very low, 24.4% and 20.99% for the cadmium and nickel respectively.[43] Therefore, the electrocoagulation/flocculation is considered as among efficient techniques that can eliminate mineral matters and reduce the turbidity of the treated water. In the studies of Pallier *et al*. (2011) this method was used for the treatment of arsenic ions by two ways; with and without its electro-oxidation and to As(V). So, this electrochemistry method has presented several disadvantages such as the production of SM and a low elimination yield.[44]

Nevertheless, residual concentrations of nitrites, sulfates and phosphates were decreased from 491 mg/L, 90 mg/L and 7 mg/L to 99 mg/L, 74 mg/L and 0.35 mg/L respectively with increasing the initial WF mass to 10 g/L (Fig. 5B). After this amount value, a stabilization phase of the phosphate and sulfates adsorption process was noticed. However, for nitrites, an increase of its concentrations was observed that can attain 110 mg/L at an amount WF mass of 20 g/L. This augmentation can be also related to the saturation of adsorption active sites conducting to thier desorption or it is due to competition between this ion and other elements that are presented in the solution. In the same figure, for the pH study, the results demonstrated that the residual concentrations of phosphates, nitrites and sulfates have decreased from 0.28 mg/L, 176 mg/L and 90 mg/L to 0.09 mg/L, 110 mg/L and 75 mg/L respectively as a function of pH, from acidic (pH=3) to slightly basic (pH = 9). At a strongly basic medium, pH ≥ 9, the adsorption lost their efficiency. Furthermore, the temperature promoted the phosphates, nitrites and sulfates removal up to a temperature of 35 °C, beyond this value, the adsorption remains stable. Otherwise, for the shaking rate and contact time factors, the adsorption process of tested contaminants in this section was proportional to these two parameters. For the contact time the process was rapid during the first 30 min, indicating the saturation of the adsorption active sites. After that, the process was stable for nitrites and sulfates ions, however, the phosphates ions were desorbed after this optimal time. According to the literatures, elimination of nutrients from wastewater is an essential step to prevent eutrophication phenomena of surface water, among these nutrients; nitrites, nitrates, phosphates and ammonium are the most elements that can present a dangerous effect to aquatic fauna. Removal of these pollutants is studied by several researches using several methods, such as adsorption, oxidation or reduction, nitrification and denitrification.[16,17,45,46]

In the other hand, according to the Fig. 5C, Zn, Fe, Cd and Pb residual concentrations were minimal at the adsorbent concentration of 10g/L, which are minimized respectively from 0.52 mg/L, 1.8 mg/L, 0.25 mg/L and 1.61 mg/L to 0.35 mg/L, 1.43 mg/L, 0.16 mg/L and 1.43 mg/L. Nevertheless, the removal of tested heavy metals was improved at a medium close to neutrality (slightly basic) except of iron ions that has

presented an optimal adsorption at a pH strongly basic at of 11 with residual concentration of 1.01 mg/L. Concerning the temperature and shaking rate factors, we noted a decrease in the concentration of these heavy metals with the increase of these factors. Furthermore, we also observed a decrease in the residual concentration of heavy metals (Zn, Pb, Cd and Fe) with the increase of contact time during the first 30 minutes. However, after this period, these concentrations increased due to the long contact time and agitation. Comparing our results with that found in the literatures, removal efficiency of Cd by our crude WF was about 48 %, which was more important that found by crude eucalyptus kraft lignin (24.4%).[43] Likewise, removal of Zn (II) ions from wastewater using alumina nanoparticles was 33.6% that is similar to that found in our work, around 32.69% but their adsorption process was very slow (4.5 h for attain the equilibrium phase) compared to our results with WF adsorbent.[47] However, the percentage yield of Pb removal onto WF was about 22.36% that was less important compared to that found by $FeCl_3$ as coagulant, this later can eliminate 56% of Pb and 41% Zn and by using of floculan that gave a removal yield of 68% Pb and 42% Zn.[48] In addition, iron ions elimination yield was important with modified clays that can attain 99.8% and 91.2% using Ca-Palygorskite and Ca-Halloysite respectively[49] compared to our material that gave a percentage of 86.11%, which was important to. Moreover, different other materials were used for the removal of heavy metals such as; Nile Rose Plant was utilized as adsorbent of Cu^{2+}, Zn^{2+}, Cd^{2+} and Pb^{2+} from wastewater.[50]

In addition, COD and BOD_5 were measured as a function of WF mass in order to show the efficiency of our material for organic matter removal and also to confirm the observations that are found above. The results are presented in the Fig. 6.

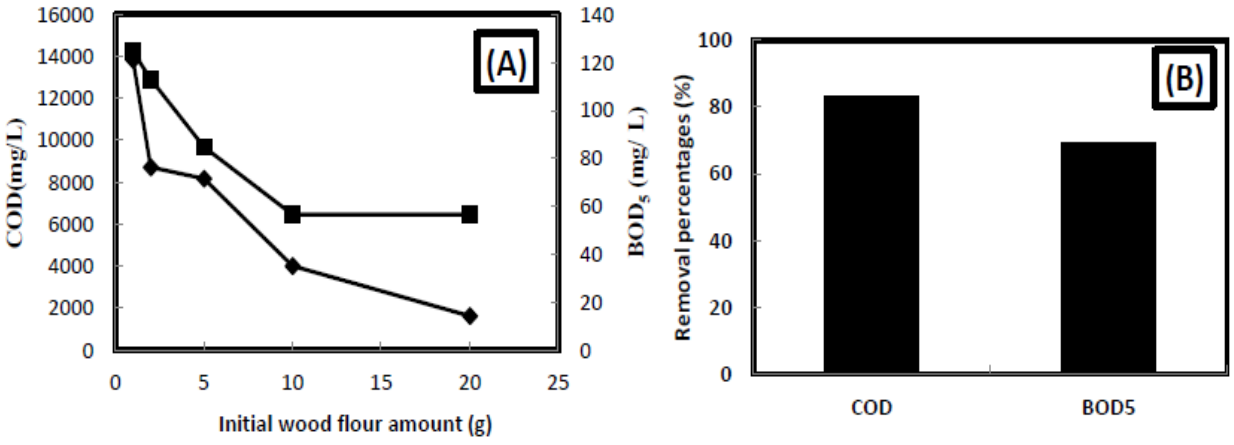
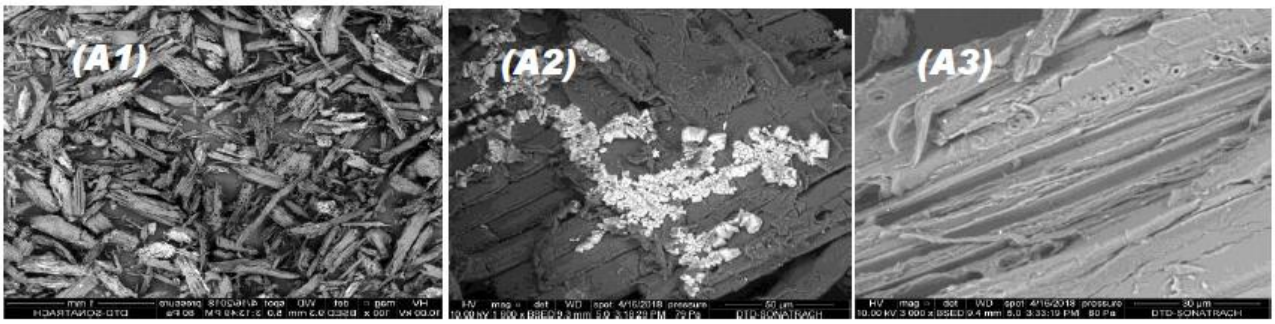


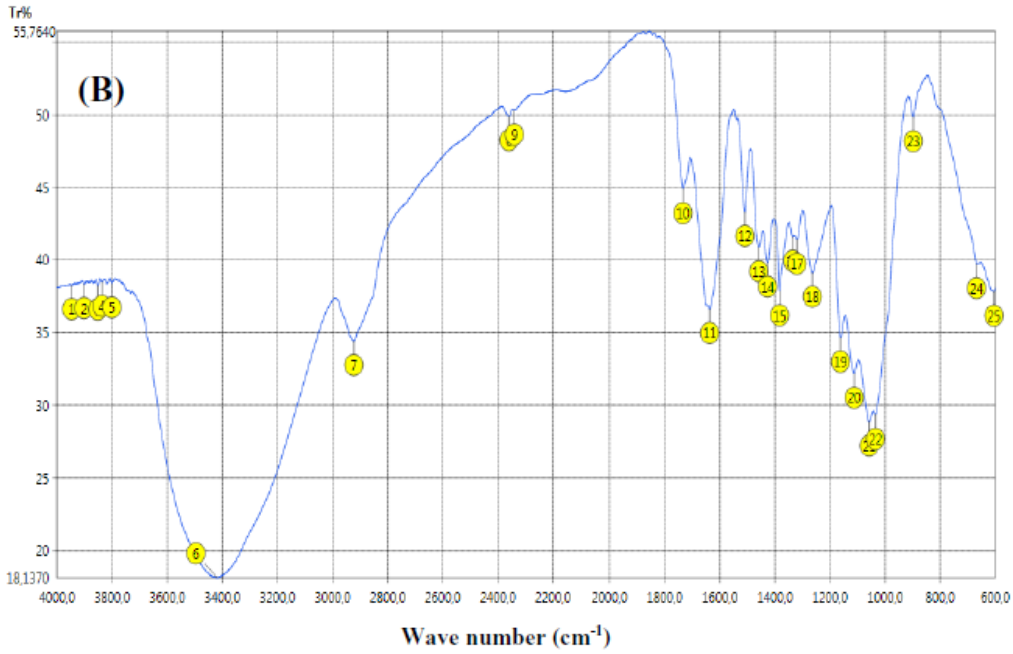
Fig. 6 – A) Variation of COD and BOD_5 as a function of initial WF amount (♦ COD and ■ BOD_5);
B) Removal percentages of COD and BOD_5.

According to the Fig. 6, the COD and BOD_5 values decreased with increasing of the material amounts that involves a diminution of organic matters concentration in the wastewater. The removal process was stable after the flour mass of 10g/L, which are the optimal flour mass selected in the previous experiments, where the COD and BOD_5 values were decreased from 24000 mg/L and 184.7 mg/L to 4000 mg/L and 56.5 mg/L respectively with a yield percentage of 83.33% and 69.4% respectively. The removal of black color G dyes using alumina nanoparticles was 25.6% that is less important compared to that found in our study.[47]

Analysis of the WF sample after the coagulation-adsorption treatment process

After optimization of favorable conditions for the oil wastewater treatment by our WF sample, we have proceeded the adsorption process by setting these parameters at the optimum (t = 30 min, m = 10 g, T = 36 °C, V = 250 RPM, pH = 6.63) with a mixture volume of 200 mL, using Jar test. The analysis results of the recovered solid are shown in the Fig. 7.





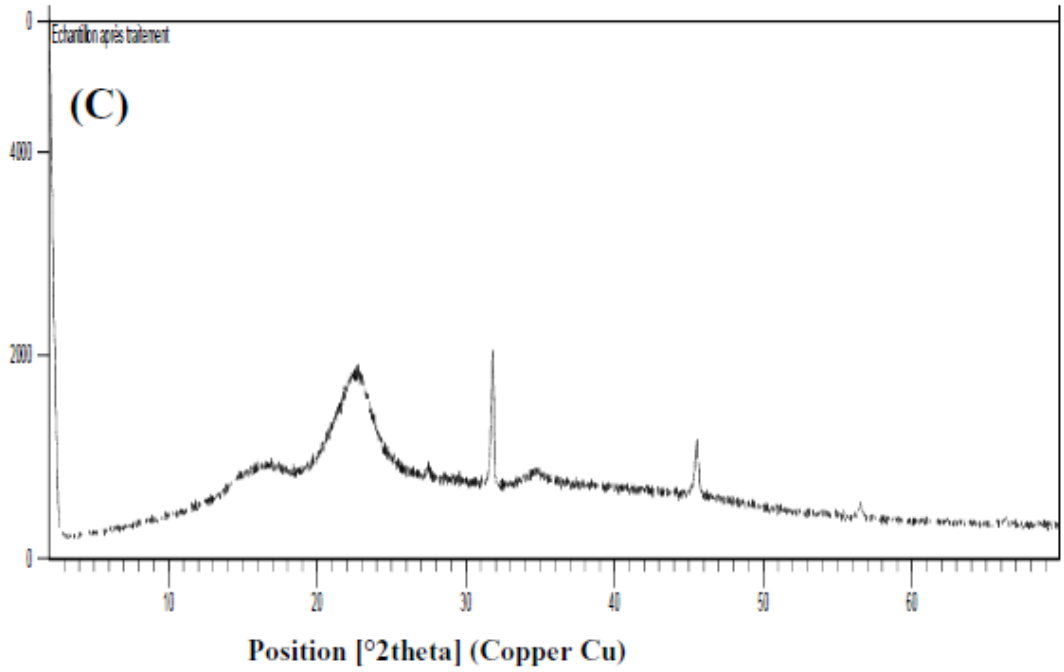


Fig. 7 – Analysis results of the WF sample after their exposure to wastewater; A) SEM micrographs at different magnifications: A1) – × 100; A2) – × 1600; A3) – × 3000; B) – FTIR spectrum; C) – X-ray diffractogram.

In the image of the Fig. 7A2, appearance of the new materials on the surface of the WF and the disappearance of the intercellular voids was observed compared to images of the Fig. 2. These findings are explained by the adsorption and coagulation of effluents on the adsorbent surface voids. Furthermore, these observations are confirmed by a magnification of x3000µm (Fig. 7A3).

In order to confirm and argue these results, the FTIR analysis was realized and presented in Fig. 7B. We observed the appearance and disappearance of some characteristic peaks compared to that of the crude sample (Fig. 3). Indeed, the average peaks located at 1646 cm^{-1}, 1458 cm^{-1} and 1428 cm^{-1} that correspond to the C=C double link were disappeared due to their breaking up and formation of other links. In addition, new large bands, around 898 cm^{-1} and 1383 cm^{-1}, were appeared that can be attributed to the vibration and elongation links of C–Cl and C–F respectively. Moreover, another peak was observed at 3420 cm^{-1} that is due to the deformation vibration of N–H link. These peaks were not recorded on the FTIR spectrum of raw WF (Fig. 3), which indicates that our material has absorbed these elements on its adsorption sites.

Concerning the X-ray diffractogram (Fig. 7C), the appearance of the new peaks was noticed that corresponds to the crystalline phases of Na and Cl. This result implies that our sample of the WF has fixed the elements Na and Cl coming from the petroleum wastewater.

CONCLUSION

The objective of our study which aimed to recover waste for the treatment of industrial effluent obtained from an Algerian oil industry was achieved. The obtained results of the characterization analyses showed that our WF has a heterogeneous structure containing micro pores on its surface and intercellular cavities. Moreover, our material is composed by three natural polymers of cellulose, hemicellulose and lignin, which have a significant adsorption capacity due to their characteristic functions. In addition, our sample contains some minerals materials with different proportions.

Furthermore, the favorable operating conditions for an effective elimination of the tested pollutants are observed at a temperature of 36 °C and rotator shaking rate (250 RPM). In addition, an important adsorption yield was recorded at a WF mass of 10 g, pH = 9 and t = 30 min.

Nevertheless, the FTIR and SEM analyses of wood sample after the adsorption process have proved our previous conclusions. Finally, we can conclude that the results obtained in our work are beneficial for the environment, industries in general and the oil industry in particular. Indeed, in this study, we have targeted two environmental opportunities at the same time, a recovery of a solid waste for the treatment of another wastewater. So, we have seen the possibility of using a WF as an adsorbent and coagulant under well-optimized experimental conditions for the treatment of petroleum effluent.

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