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Treatment of tannery wastewater by infiltration/percolation process using natural clay combined solid wastes

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ABSTRACT

This work aims to treat the raw effluents from a tannery using raw coal waste (fly ash and bottom ash) and bentonite clay as less expensive adsorbents and percolating agents. Their combination constitutes effective filters to retain heavy metals, dyes, and organic matter. After analysis of the raw wastewater, a significant pollutant load was detected; the chemical oxygen demand (COD) is around 3,400 mg·L^{-1}, the biological oxygen demand about 240 mg·L^{-1} and suspended matter of 490 mg·L^{-1}. These waters are also heavily loaded with heavy metals especially chromium with a high value of 981 mg·L^{-1}, and they have an acidic pH of 4.2, which greatly exceed the specific limit discharges values, applicable to discharges from Moroccan tannery industries. Their treatment with fly ash, bottom ash, and bentonite clay leads to an almost complete elimination of Cr, Fe and Zn with a discoloration of 96% where the pH becomes neutral to slightly alkaline. The COD analysis gives an abatement rate of more than 72% while suspended matters are considerably reduced with a turbidity rate of 99%. Thus, the treated water can be used for irrigation or as wash water in industries.

Keywords: Tannery wastewater; Raw coal wastes; Bentonite clay; Heavy metals; Dye; Infiltration/percolation; Adsorption

1. Introduction

Rapid economic development and population growth have led to environmental deterioration. Indeed, industrial effluents and many other human activities have brought significant amounts of organic and inorganic pollutants into aquatic systems, causing various environmental problems such as eutrophication, metal contamination, and loss of biodiversity and dangerous for human health [1–4]. With the development of the tannery industry, its wastewater has become one of the main sources of pollution loaded with organic matter and heavy metals [5–7], which requires prior treatment. The characteristics of leather tanning wastewater depend on the nature of the tanning process. There are two methods of tanning; the modern technique used chromium in the finishing process and the traditional method

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using plant extracts to stabilize leather [1,8]. With the both processes, a large number of organic and inorganic compounds are employed, such as natural or synthetic tannins, organic acid, as well as chloride, chromium III, and sulphide salts [9,10].

Various treatments are used for tannery wastewater like physico-chemical methods such as sedimentation [11], electro-flotation [12] coagulation–flocculation [13], filtration [13], membrane, and adsorption [14,15], while the chemical and biological methods are considered very expensive in terms of energy and reagents consumption. Among them, the treatment of effluents containing heavy metals revealed that adsorption is a very effective technique [16]. In addition, the quantities of fly and bottom ashes produced annually by the Jorf Lasfar TAQA Thermal Power Plant (Morocco) are significant, respectively around 400.000 and 40.000 tones [17] and pose many problems related to their storage, which requires their management. Thus, our study consists of the development of inexpensive water treatment techniques while recovering industrial waste and natural adsorbents. The treatment of tannery wastewater by filtration on fly and bottom ashes from a thermal power station located in El Jadida (Morocco) has been studied in the removal of some heavy metals and dyes. The association between bentonite clay and the fly ash/bottom ash matrices has also been discussed for water treatment, thanks to their specific surface area, smaller particle size and abundance. Using the selected adsorbents, the vertical percolation/infiltration process was tested. We also tested the adsorbent performance of different matrices composed of natural porous materials (clay and sand) and industrial waste (coal fly and bottom ashes from thermal power plants).

2. Sampling and experimental evaluation

2.1. Sampling

The tannery effluents were collected in the sewer at the entrance to the collection basin of the Mohammadia tannery before any pre-treatment. Samples were taken every 2 for 24 h. Sample bottles were rinsed in the laboratory with distilled water and rinsed again with sampled water before collection. Samples were collected in 5 L plastic containers, transported to the laboratory and stored at 4°C. Their characteristics are given in Table 1 with specific limit discharges values applicable in Morocco.

2.2. Analytical methods and equipment used

The crystalline phases of the raw materials was determined by a X-ray Diffractometer (Bruker D8, Germany) operating at 45 kV/35 mA and equipped with $Cu_{K\alpha}$ radiation ($\lambda_{K\alpha}$ = 1.54056 Å). The chemical composition determined by X-ray fluorescence spectroscopy using a wave dispersion spectrometer (WD-XRF) type axios. The specific surface areas of adsorbents were determined from the isotherms of N_2-sorption at 77°K using Micromeritics apparatus (ASAP 2020, USA).

Suspended solids (SS) are determined according to AFNOR (T90-105). After filtration of a test on filters, Whatman GF/C (0.45 mm) previously dried and weighed. The filters were placed in an oven at 105°C for 2 h, weighed to assess the suspended solids. Turbidity was measured by a nephelometric turbidity meter HANNA HI 93703C, it measures the intensity of light deflected at an angle of 90° to the incident light that passes through the straight sample.

Biological oxygen demand (BOD) features the biodegradable fraction of organic matter involving bacteria in wastewater. BOD_5 measurement is performed on raw samples respirometric method. It was determined using a manometric device (BSB Model 602) incubated in a thermostatic chamber at 20°C according to the procedure indicative by Rodier (1996). The chemical oxygen demand (COD) was analyzed using a COD-meter thermo-type reactor Behr-TRS300 and heavy metals were determined by inductively coupled plasma-atomic emission spectroscopy (ICP-AES, USA).

2.3. Wastewater characterization

As shown in Table 1, the tannery wastewater is characterized by an acid pH with a very high COD and BOD_5 as well as rich in suspended solids. This high load of organic matter is due to the skins of biogenic materials and the precursors used. Even if the wastewater from the tanneries is rich in organic matter, the BOD_5/COD ratio (<0.1) confirms that it is a matter of effluents that are difficult to biodegrade. With the exception of three metals (Cr, Zn, Fe) considered excessive, in particular Cr, the others are negligible. The SO_4^{2-} content is very high due to the use of chromium sulphate as tanning agent. According to Moroccan regulations and requirements, these values greatly exceed the limit values applicable to discharges from tannery industries. For this, the treatment of wastewater is necessary before it is discharged into nature to reduce its consequences on the environment. In this context, the use of less expensive porous and natural adsorbents is very useful. Thus, coal fly ash (CFA) and bottom ash (BA), collected at the thermal power plant of Jorf Lasfar (El Jadida-Morocco) associated with bentonite clay and sand were selected for the retention

Table 1
Some characteristics of tannery wastewaters and discharge limit values

Parameters	Values	Discharge limit values [18]
Temperature (°C)	21	30
pH	4.5	6.5–9.0
Conductivity (μS)	1,000.96	2,700
Turbidity (NTU)	915	–
SS (mg·L^{-1})	490	50
COD (mg·L^{-1})	3,402	500
BOD_5 (mg·L^{-1})	241	100
SO_4 (mg·g^{-1})	10,738	600
Color	Dark green	Clear
Cr total (ppm)	981	2
Zn (ppm)	9.29	5
Fe (ppm)	7.48	3
(Pb+Ni+Mn+Al+Cu+Se) (ppm)	1.88	–

of metals in excess and discoloration in order to establish the proper characteristics of the discharge water. A characterization of these materials was carried out to control the active sites capable of fixing the pollutants detected in the wastewater.

2.4. Structural characteristics of low-cost adsorbents

Coal fly ash (CFA) and bottom ash (BA) were collected from thermal power plant of Jorf Lasfar located in the province of El Jadida (117 km from Casablanca, Morocco). Fly ash is the fine powders (0.5–315 µm) from burnt pulverized coal in boilers in the form of isolated or clustered spheres [19]. Bottom ash is the residue from the combustion of coal, they appear as porous dark grains more or less gray, and they are coarser than fly ash the particle size distribution of CFA ranges from 1–10 mm. Before any use, these selected materials were dried for 24 h at 105°C and sieved to have fractions in 80–200 µm range, this improves their surface properties to better retain heavy metals and dyes.

Natural bentonite clay was obtained from the mine in the Nador region in the north of Morocco; this mineral is in the form of a white powder grounded, sifted, and dried at 40°C for 48 h while samples of sand were collected from El Oualidia beach. They were, washed several times with distilled water then dried at 90°C at the oven, sifted to determine the various sizes. Their chemical composition are given in Table 2.

Fig. 1 shows X-ray patterns of fly ash and bottom ash revealing several crystalline phases, including quartz (SiO_2), mullite ($Al_6Si_2O_{13}$), calcite ($CaCO_3$), and hematite (Fe_2O_3).

This can be explained by the composition of the coal used in the thermal power plant, it generally consists of silica crystallized in the form of quartz and phyllite minerals of the clay group. During combustion, these minerals change their structure and give rise to a crystallized fraction in the form of mullite and quartz and an amorphous part [20]. Fig. 1 also shows the diffractograms of bentonite clay, which composed of three principal phases (montmorillonite, kaolinite, and anorthite). Due to its mineral composition, BC can be classified as sodium bentonite (Na-bentonite) [21].

The specific surface values obtained are 10.38 and 4.07 $m^2·g^{-1}$ for fly ash and bottom ash, respectively. Compared to the raw coal wastes described, bentonite clay is characterized by a high specific surface area (49.9 $m^2·g^{-1}$) very high water retention capacity and therefore a high swelling capacity and porosity as well as permeability [21], which will allow the retention of heavy metals.

3. Wastewater treatment approach

3.1. Experimental set-up

The water purification experiments were carried out on a laboratory scale using an experimental set-up designed for infiltration and percolation operations as shown in Fig. 2, which consists of a small column of 12 cm height and a radius of 10 cm. We tested the performance of seven matrices composed of two to four layers of adsorbent materials. The volume of wastewater to be filtered is 200 mL. The mass of adsorbents is optimized after several tests and the proportion and the position of each adsorbent are detailed in Table 3. The position of the adsorbents in the columns was chosen taking into account the particle size of the materials in order to avoid clogging. The percolation infiltration tests were repeated five times to ensure the repeatability of our tests. The measures taken into account throughout the filtration operation are as follows: (i) infiltration time or residence time: time necessary for all the volume brought to the system to flow through all the pores of the adsorbents, (ii) the total volume returned: total volume of treated wastewater recovered at the outlet of the system.

3.2. Sewage treatment result

The hydraulic efficiency is related to the effective volume and flow pattern as well as the operating performance of the anaerobic reactor. According to the matrices chosen, the hydraulic characteristics are given in Table 4. This shows that the contact time is higher in filters containing

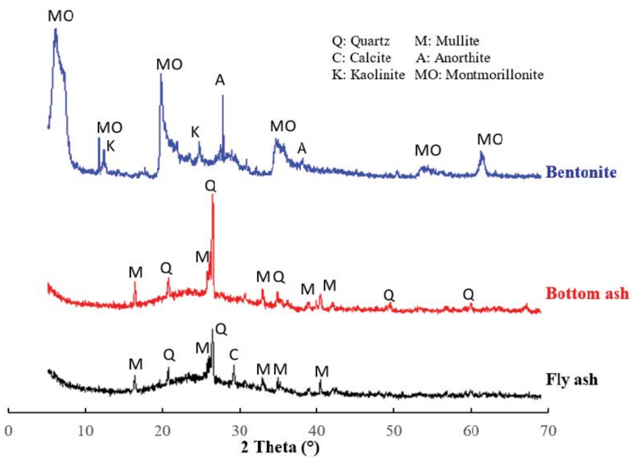

Fig. 1. X-ray diffraction patterns of fly ash, bottom ash, and bentonite clay.

Table 2
Chemical composition of fly ash, bottom ash, bentonite clay, and sand

Oxides	SiO_2	Al_2O_3	Fe_2O_3	K_2O	CaO	SO_3	TiO_2	MgO	Na_2O	(SiO_2 + Al_2O_3 + Fe_2O_3)
Fly ash	56.42	18.51	3.04	2.34	2.45	2.65	1.5	1.9	0.99	77.97
Bottom ash	48.07	21.34	9.86	2.9	2.21	1.89	–	1.19	0.4	79.27
Bentonite	52.6	22.3	1.82	2.02	1.29	–	0.20	1.83	2.52	76.72
Sand	48.53	3.96	3.26	0.84	24.21	–	0.49	0.49	–	55.75

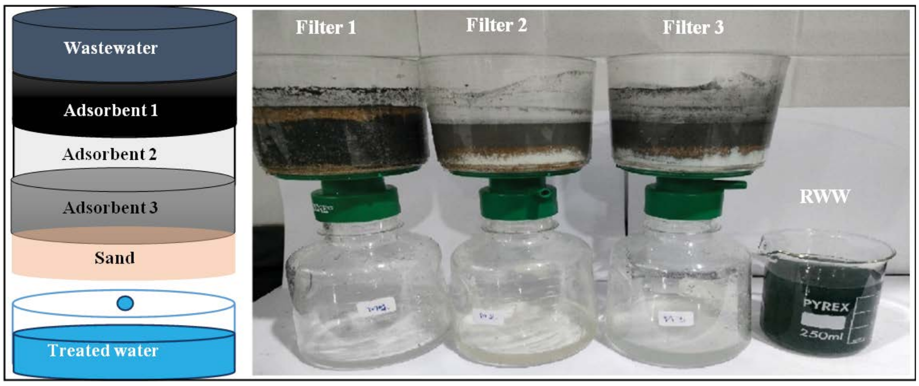

Fig. 2. Experimental set-up used in filtration.

Table 3
Composition of matrix used in filtration

Matrix	Adsorbents
1	40% sand + 30% fly ash + 30% bottom ash
2	40% sand + 30% bentonite clay + 30% fly ash
3	40% sand + 30% bentonite clay + 30% bottom ash
4	20% sand + 30% bentonite clay + 30% fly ash + 20% bottom ash
5	40% sand + 60% fly ash
6	40% sand + 60% bentonite clay
7	40% sand + 60% bottom ash

Table 4
Hydraulic characteristics

Matrix	1	2	3	4	5	6	7
Input volume (mL)	200	200	200	200	200	200	200
Infiltration time (min)	30	35	35	35	30	45	25
Volume returned (mL)	120	100	100	100	120	90	130

bentonite clay and fly ash due to their fine particle size, which affects the retention rate. As a result, the volumetric efficiency is related to the pollutant conversion efficiency and the flow pattern, but it also is influenced by the hydraulic characteristics.

Determination of pH plays an important role in the wastewater treatment process where build-up of toxic chemicals and increased alkalinity levels are common problems in wastewater. The pH control is an important parameter for neutralization, precipitation, coagulation and other biological treatment processes and most recommended analysis for wastewater treatment [22]. Fig. 3 shows the evolution of the pH of treated water (WWT) using different filter matrices compared to that of the selected wastewater. At the start of the operation, it was acidic (pH 4.2) and becomes between pH 7.3–10.6, depending on the nature of the matrix. It is linked to the alkaline nature of adsorbents and to the adsorption of acidic species in wastewater. It may also be related to neutralization of the tannery's wastewater by the carbonates present in different

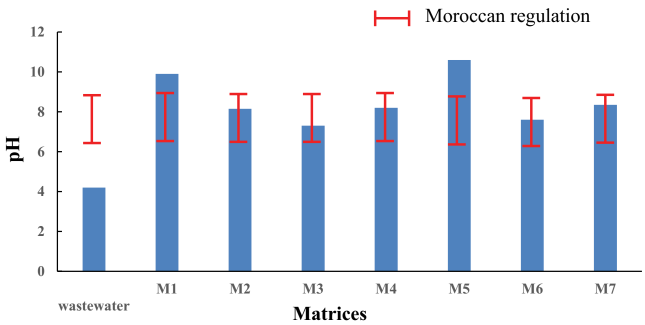

Fig. 3. Evolution of pH solution after water treatment with different matrices.

levels of filters. Note that the pH values of most filtrates are within the pH range of the Moroccan standard for direct discharges [20].

The total concentration of COD in the raw wastewater is 3,402 mg·L^{-1} at the outlet of the filtration columns, while those after treatment vary between 931 and 2,189 mg·L^{-1}. The high elimination rate of which is obtained by the matrix M4 composed of sand, fly ash, bentonite clay and bottom ash with a rate of 72.63% (Fig. 4). In the treatment of wastewater by infiltration-percolation, a major part of the organic matter is eliminated by the simple mechanical role of filtration played by the filter bed through degradation, transformation, and bacterial oxidation. The oxidation of dissolved organic material is related to the availability of oxygen and the residence time of the effluent at the matrix [23].

After the wastewater flows over the filters, its suspended matter (SM) content drops from 490 mg·L^{-1} to values between 29–78 mg·L^{-1} (Fig. 5), with a smallest amount SM was found for filter M4 (29 mg·L^{-1}); which represents an abatement of 94.08% in accordance with Moroccan regulations (50 mg·L^{-1}). The difference in abatement between the different filters is due to the presence of bentonite clay and fly ash where their fine particle size leads to a long residence time of the wastewater allowing better retention of suspended solids. Using the same water treatment, a high turbidity removal rate (99%) was achieved using the M2, M4 and M5 filters, this is due to the removal of colloids and mineral and organic matter. These organic pollutants compete with heavy metals at the active sites on the adsorbent surface [24].

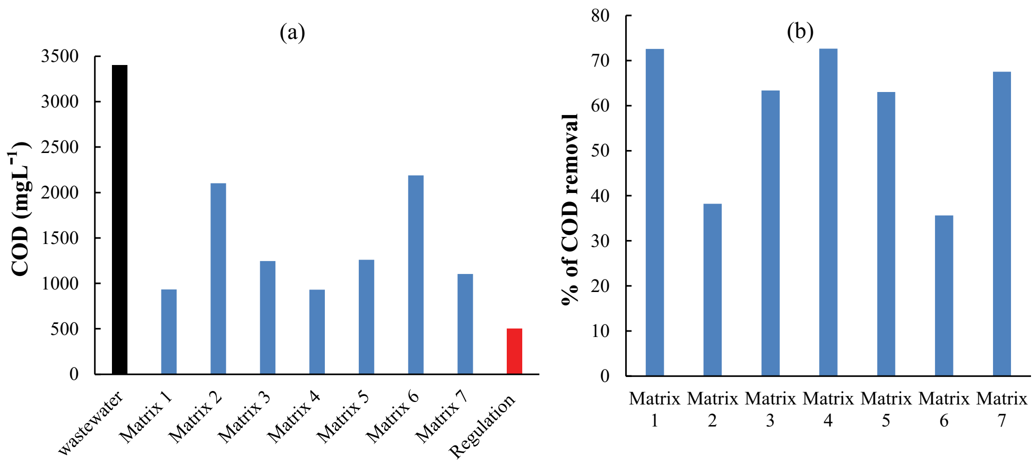

Fig. 4. (a) Variation of COD concentration and (b) abatement.

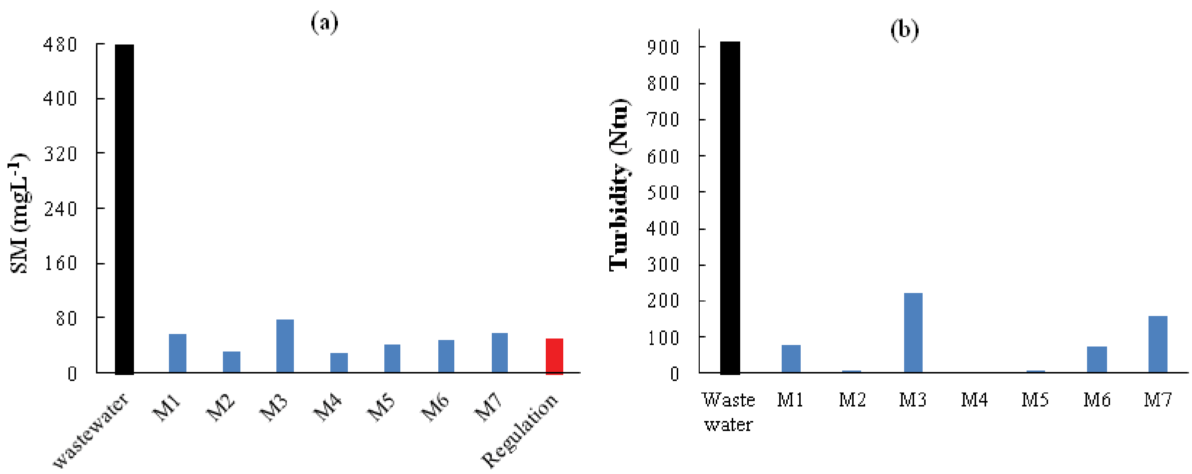

Fig. 5. Abatement of (a) suspended matter and (b) turbidity.

Table 5
Heavy metals concentration (ppm) in wastewater and its removal (%) using the different matrices as well as the Moroccan standards

		Wastewater	M1	M2	M3	M4	M5	M6	M7	Moroccan standards
Cr	(ppm)	981.00	2.33	3.68	14.4	1.49	3.29	7.15	4.26	2.00
	Removal (%)	–	99.7	99.6	98.5	99.8	99.6	99.2	99.5	–
Fe	(ppm)	7.48	0.01	0.04	0.03	0.00	0.01	0.01	0.02	3.00
	Removal (%)	–	99.4	99.5	100	99.8	99.8	99.7	99.8	–
Zn	(ppm)	6.29	0.22	0.17	0.00	0.00	0.53	0.45	0.04	5.00
	Removal (%)	–	96.5	97.2	100	100	91.5	92.8	99.3	–

As described below, the tannery's wastewater is heavily contaminated with chromium (981 mg·L^{-1}) due the use of chromium sulphate as a tanning agent. For the different adsorbent matrices characterized elsewhere, the concentrations of heavy metals before and after treatment are given in Table 5 compared to Moroccan standards for direct discharges [20].

Note that matrix 4 (fly ash-bottom ash-bentonite clay) and matrix 1 (coal fly ash + bottom ash) provide the best performance for the removal of heavy metals. For chromium, its elimination is significant (99.85%) and its residual concentration is 1.43 mg·L^{-1}, which complies with the Moroccan standard (2 mg·L^{-1}), whereas complete elimination is obtained for the majority of matrices. for iron and zinc the removal rate is 100%. The negative charges on the surface of clay, coal fly ash, and bottom ash are responsible for the sorption of heavy metals linked to the presence of silicates and aluminates as active sites

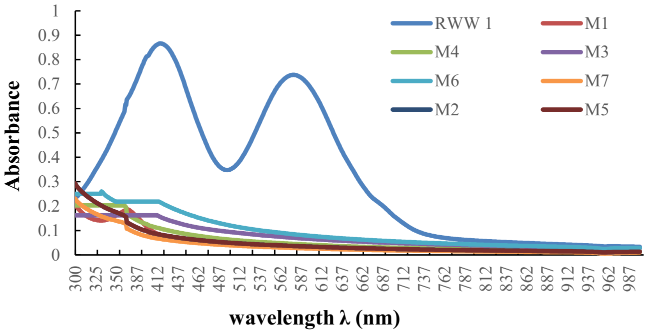

Fig. 6. UV-Visible spectra of raw wastewater (RWW) before and after treatment by the different matrices.

Table 6
Discoloration of wastewaters by the different filters

	Absorbance	Discoloration (%)
RWW	0.7374	–
Matrix 1	0.0358	95.15
Matrix 2	0.0367	95.02
Matrix 3	0.0702	90.48
Matrix 4	0.0367	95.02
Matrix 5	0.0286	96.12
Matrix 6	0.0847	88.51
Matrix 7	0.0453	93.86

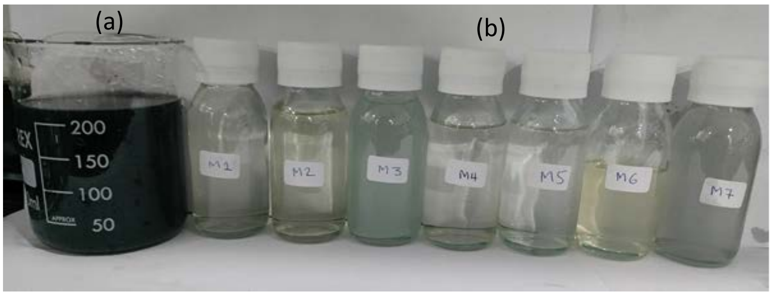

Fig. 7. Tannery wastewater before (a) and after treatment (b) by different filters.

exhibiting an electrostatic interaction with Cr, Fe and Zn metals. In addition, the tanning involving dyeing requires the use of dyes causing significant pollution after the discharge of dark green wastewater without any prior treatment. The degree of coloration before and after treatment was checked by UV-Visible spectrophotometric measurements showing that the effluent strongly absorbs in the visible range at two wavelengths 416 and 578 nm (Fig. 6).

After water treatment using different filters, discoloration varies between 88.5% for matrix 6 and 96.12% for matrix 5 (Table 6). This could be explained by the better adsorption of dye molecules on the active sites of adsorbents by electrostatic attraction (Van der Waals forces, hydrogen bonding, and hydrophobic interactions) [25]. Fig. 7 visualizes the appearance of wastewater and treated water, which this laser becomes completely transparent which improves considerably the quality of treated wastewater. The obtained results improve considerably the effectiveness of fly ash, bottom ash, and bentonite clay to adsorb dyes contained in tannery wastewater samples.

4. Conclusion

Treatment of tannery's wastewater was carried out in applying the infiltration/percolation methods using different combinations of adsorbents (fly ash, bottom ash, and bentonite clay). Results obtained showed that the filters composed of two waste materials and clay have a good retention of metallic elements (Cr, Zn, Fe) and the dyes contained in the selected tannery's wastewater. They can remove up to 99.85% of chromium and 100% of iron and zinc. After water treatment, the pH of wastewater becomes neutral to slightly alkaline. The COD, turbidity, and SM are very significant with values of 72%, 99.48% and 94%, respectively. The high abatement rates obtained on almost all physico-chemical parameters of the filtered water show the interest of the filtration method by adsorption on inorganic support being an economical technique. The characteristics of the treated wastewater will be used for irrigation water and the filtration sludge will be recycled in the field of civil engineering as building materials.

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