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Treatment of textile dyeing wastewater using advanced photo-oxidation processes for decolorization and COD reduction

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ABSTRACT

In this study, advanced oxidation processes were applied to the treatment of a textile dyeing wastewater. The effluent presents a dark blue color, with a maximum absorbance peak at 594 nm, alkaline pH (pH = 12.4), high organic contents (COD = 1,400 mg L^{-1}). Experiments were conducted on a lab-scale prototype using UV lamp as light source and pillared natural clay with Fe and Al as catalyst. All the processes examined lead to an effective decolorization and mineralization, but the most efficient process was the photo-Fenton, contributing to 93% decolorization and 88.8% mineralization after 3 h of reaction under UV irradiation. Characteristics of wastewater after treatment were COD below 250 mg L^{-1}, pH 8.5 and uncolored. Thus the resultant water has conditions that are suitable for releasing in public sewage systems.

Keywords: Textile dyeing wastewater; Photo-Fenton; UV-light irradiation; COD mineralization; Decolorization; FeAl-pillared clay

1. Introduction

Today, the high pressure on freshwater sources leads to their contamination by various pollutants. Industrial sector was considered the principal source of contamination because of the generation of various types of pollution [1,2]. Industries such as petrochemicals, food, and textile produce huge quantities of effluents that are highly charged, hardly biodegradable and generally bio-recalcitrant [3]. Textile industry is one of the most water-consuming industries [3]. It consumes an average of 10.5 billion L of water per day and it is responsible for 17%–20% of water pollution in the world [4] with an annual production of nearly 800 tons [5]. This industry generates recalcitrant and toxic organic molecules, which are responsible for organoleptic and aesthetic pollution, health problems and contamination of groundwater, soil and vegetation [6,7].

The treatment of industrial waste is, however, difficult because organic dyes cannot be degraded by conventional means (biological treatment). To reduce these processing difficulties, several physical processes have been envisaged, in particular membrane filtration [8], precipitation/coagulation of dyestuffs [9] and adsorption on activated carbon [10]. But, these methods have the disadvantage of moving simply pollution in large amounts of sludge, to be ineffective for certain types of specific dyes and to be relatively expensive [11]. It is, therefore, necessary to create and implement simple and inexpensive solutions in order to treat industrial effluents.

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Chemical methods were thus perceived as a solution to the problem of treating industrial effluents. However, these processes using traditional oxidants (Cl_2, O_3, etc.) do not allow the organic compounds to be completely removed, their role being mainly disinfection. In addition, if the pollutant contains halogens, oxidation generates toxic by-products such as trihalomethanes, which are perceived as carcinogens [11].

For all these reasons, the researchers concentrated their efforts on more powerful oxidation processes: the advanced oxidation processes (AOPs). These technologies have already shown their potential in the treatment of bio-recalcitrant organic pollutants [12,13]. Many authors use these technologies in the treatment of textile effluent: H_2O_2/UV [14]; TiO_2/UV [15,16]; TiO_2/H_2O_2/UV [17]; homogeneous Fenton reaction [18,19] and homogeneous photo-Fenton reaction [20]. However, the costs involved with the energy requirements and chemicals, especially when complete mineralization is the main objective, are expensive and eventually make these processes inapplicable at the industrial scale [13]. For these reasons, the development of newer eco-friendly methods that are able to destroy these pollutants has become an imperative task such as photo-Fenton reaction using pillared clay as heterogeneous catalyst and UV irradiation as light source. The use of modified clay by iron and aluminum offers significant advantages in separation because the catalyst can be easily recovered by sedimentation or filtration and further used [21]. Also, it is not necessary to acidify the solution before the reaction.

In our earlier works [22,23], the photocatalytic activity of Fe/Al-pillared clay for the decolorization and degradation of Congo Red diazo dye has been studied and found that Fe/Al-pillared clay was the most effective catalyst for the degradation of dye. In continuation of our studies in dye photodegradation [22,23], we investigated the treatment of textile dyeing effluent collected from a local textile company.

Therefore, this study compares the efficiency of different advanced oxidation processes in the treatment of real textile dyeing wastewater at lab-scale under UV light irradiation. The main goals of this work are to test the color and COD removal of textile effluent by photo-Fenton oxidation after the adsorption process, to investigate the influence of the photo-Fenton parameters and finally to reduce the effluent characteristics to an acceptable level that can be discharged to the environment with minimal adverse effects.

2. Experimental methodology

2.1. Materials and chemicals

2.1.1. Real textile dyeing effluent

Wastewater sample was collected after the textile dyeing process from a local company: SITEX. This textile industry is located at the east of Tunisia, specialized in yarn manufacturing, indigo dyeing process and textile finishing, before being subjected to any form of treatment. Table 1 presents the main physico-chemical characteristics of the sample used in this work. The selected effluent presents a dark blue color, resulting from the mixture of different dyes (such as indigo, sulfur dyes, vat dyes) and other chemical products (wetting agents, surfactants, dispersing agents). The maximum absorbance peak of this effluent is at 594 nm (Fig. 1). Wastewater sample was kept away from light in an opaque container at 4°C.

2.1.2. Chemicals

Treatment assay of wastewater was performed employing prepared catalyst based on natural clay (belong to the smectite family), hydrogen peroxide (35%) and all other chemicals in this work were of analytical grade and used without any further purification.

2.1.3. Preparation of the investigated material

Raw clay used in this study was extracted from soils of Tboursouk (situated in the North-West of Tunisia). This clay belongs on smectite family; it was prepared and purified as described in our previous studies [22,24]. The structural formula is $Ca_{0.22}Na_{0.422}K_{0.174}(Si_{7.58}Al_{0.42})(Al_{2.86}Fe_{0.748}Mg_{0.48})O_{22}$.

The modification carried out in this clay was done via pillaring process by Fe and Al cations. The pillaring process and the most characteristics were reported in our previous study [22]. Fig. 2 illustrates the different steps of pillared clay preparation via pillaring process. The samples $FeAl_X$-PILC obtained with X = Fe/Al+Fe molar ratio of 0.01, 0.05, 0.1 and 1 are denoted below as $FeAl_{0.01}$-PILC, $FeAl_{0.05}$-PILC, $FeAl_{0.1}$-PILC and $FeAl_1$-PILC, respectively.

2.2. Treatment of real effluent in continuous process

2.2.1. Lab-scale photo-reactor prototype

The optimization of the photo-Fenton reaction parameters was carried out in a lab-scale photoreactor illuminated with UV lamp. The photo-reactor used is a Pyrex cylinder and its volume is 150 mL. A magnetic stirrer was provided to ensure complete homogenization of the solution inside the glass vessel. Irradiation was provided by a 125-W Philips HPK (Eindhoven, Netherlands) UV-lamp (UV-A) placed in a plugging tube. A Pyrex cylindrical jacket located around the plugging tube allows an irradiation with wavelength; λ = 350 nm.

2.2.2. Experimental run

All experiments were done at room temperature (T = 25°C) and without pH regulation (natural pH of

Table 1
Textile dyeing effluent characterization

Parameters	Units	Values
pH	–	12.4
COD	mg L^{-1}	1,400
BOD_5	mg L^{-1}	400
Total suspended solid	mg L^{-1}	200
Oil and grease	mg L^{-1}	10
Chloride	mg L^{-1}	2,000
Color	Pt-co	1,580
Total hardness	mg L^{-1}	100

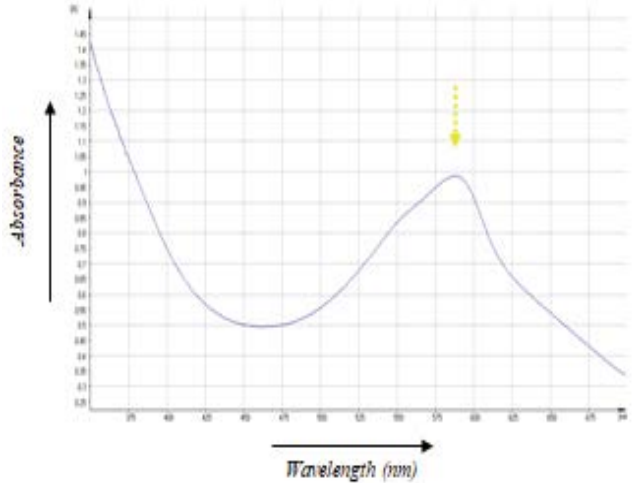

Fig. 1. UV-visible spectra of textile wastewater at pH = 12.4 (natural pH of effluent).

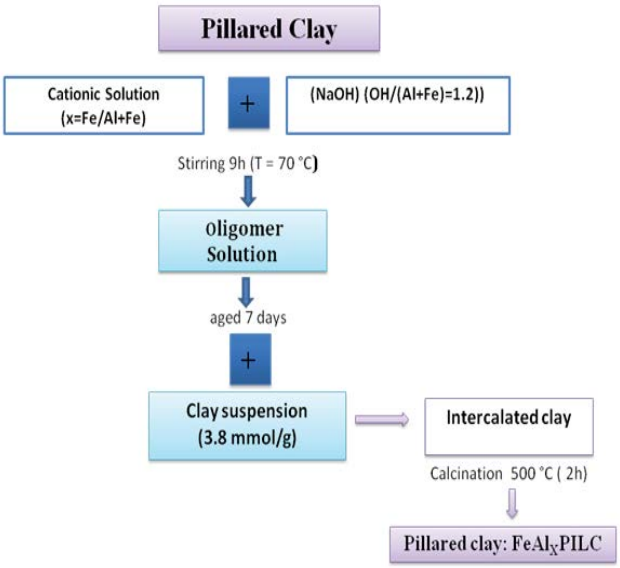

Fig. 2. Scheme describing synthesis process of pillared clay.

effluent). In order to evaluate the photo-Fenton reaction parameters, the photo-reactor was filled with 150 mL of effluent. The desired amount of catalyst was added. Then, the suspension was homogenized for 1 h in the darkness to ensure the adsorption–desorption equilibrium at room temperature. A sample was taken for effluent concentration control, centrifuged and filtered. The lamp was turned ON and the solution was completely exposed to the UV light. Finally, the volume of hydrogen peroxide was added and samples were taken at pre-defined times to evaluate the degradation process. The operational conditions of the experiments are presented in Table 2.

Table 2
Experimental condition for real effluent treatment

Temperature	25°C
pH	pH of effluent (12.4)
Catalyst	$FeAl_x$-PILC (x = 0.01, 0.05, 0.1 and 1)
Catalyst dose	[0.1–0.3 g]
H_2O_2 volume	[1–3 mL]

2.3. Analysis

The decolorization (%) in solution was determined by UV-vis spectrophotometer (Shimadzu Model, PerkinElmer, Waltham, USA), absorbance measurements were performed at the maximum wavelength of effluent, at 594 nm in natural pH of effluent (12.4). Also, UV-vis spectrum was recorded between 200 and 700 nm. COD concentration was measured by using HACH LANGE DR3900 (Loveland, USA). pH was measured using pH meter HANNA. Before analysis, all samples were centrifuged at 4,000 rpm for 15 min and limpid supernatant liquids were collected and filtered for the determination of COD and absorbance.

Unfortunately, due to some limitations, the other physicochemical parameters of effluent were not measured in all the tests.

3. Results and discussion

3.1. Most properties of investigated materials

The most physico-chemical properties of purified clay before and after pillaring modification by Fe and Al polycations are summarized in Table 3.

Furthermore, the morphology of purified and pillared clay (we give only the example of $FeAl_{0.1}$-PILC) is given by TEM (Fig. 3I) and SEM (Fig. 3II) analysis. As can be seen (Fig. 3), TEM micrographs of $FeAl_{0.1}$-PILC show layer structure after pillaring process as the purified sample and SEM micrographs show remarkable changes in the morphological surface after modification with Fe and Al species.

3.2. Adsorption study

In this work, adsorption is an important step in the catalytic act. Hence, to ensure a good catalytic activity of catalyst, it is a key to know the affinity between the adsorbent and the adsorbate and define the necessary time to reach the adsorption–desorption equilibrium. For this experiment, 0.2 g of clay was placed in a 100 mL erlenmeyer flask containing 50 mL of effluent. For kinetic studies of effluent adsorption, 2–3 mL of suspension were collected at various time intervals with a syringe. The clay was separated from the liquid phase by centrifugation and filtered through a 0.45-μm filter. Then, the quantification of effluent was determined by spectrophotometer.

Fig. 4 shows the adsorption kinetics of real effluent by the purified clay and $FeAl_{0.1}$-PILC. As can be seen, the adsorption percentage for pillared clay is about 54% compared with the purified clay which is 7%. This is can be explained by the enhancement of the adsorptive properties of the purified clay after pillaring process. Also, the adsorption–desorption equilibrium was reached in 60 min.

Table 3
Physicochemical properties of the studied clays

Catalyst	S_{BET} ($m^2\ g^{-1}$)	CEC (meq/100 g)	d_{001} (Å)	V_p ($cm^3\ g^{-1}$)	D (Å)	% Fe_2O_3	% Al_2O_3
Purified clay	58	72.93	12.56	0.07	19.11	7.10	20.09
$FeAl_{0.01}$-PILC	61.67	24	19.36	0.08	19.01	9.36	27.32
$FeAl_{0.05}$-PILC	55.43	21.33	19.31	0.07	18.9	10.68	28.23
$FeAl_{0.1}$-PILC	53.85	17.33	19.09	0.08	19.003	12.19	28.87
$FeAl_1$-PILC	50.08	13.33	14.49	0.09	18.85	18.28	20.14

S_{BET}: BET surface area; CEC: cationic exchange capacity; d_{001}: basal spacing of clay; D: average pore diameter; V_p: volume of N_2 adsorbed at $P/P_0 = 0.98$.

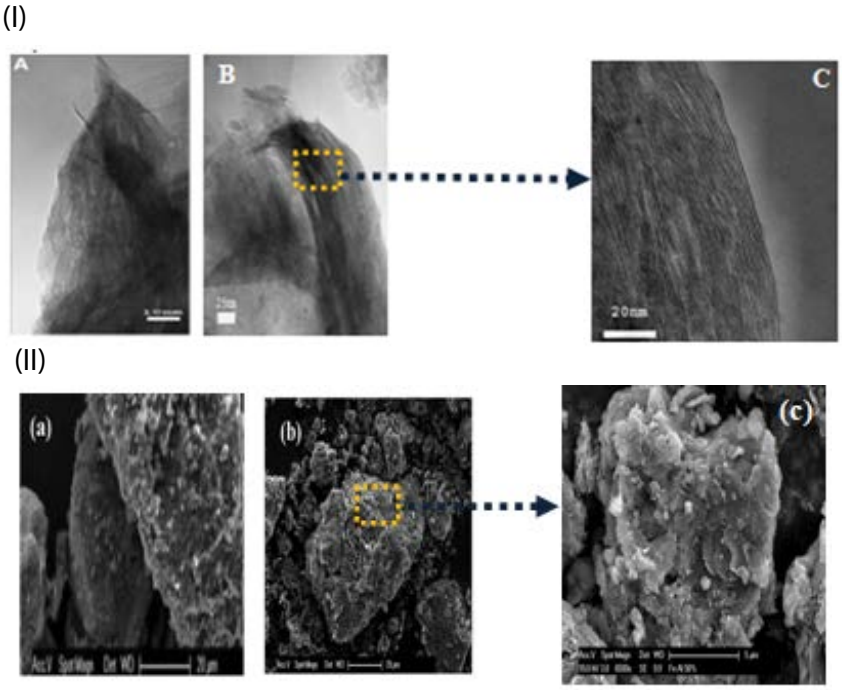

Fig. 3. (I) Morphology of purified (A) and pillared clay ($FeAl_{0.1}$-PILC) (B) and (C) given by TEM. (II) Morphology of purified (a) and pillared clay ($FeAl_{0.1}$-PILC) (b) and (c) given by SEM.

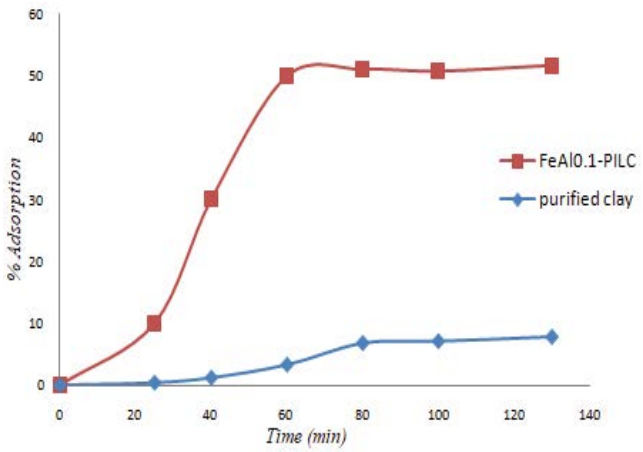

Fig. 4. Kinetics of real effluent adsorption onto purified and pillared clay (experimental conditions: 50 mL of effluent; 0.2 g of adsorbent; pH = 12.4; T = 25°C).

3.3. Comparison between advanced oxidation processes in the treatment of real effluent

Preliminary treatment studies were performed in order to compare the efficiency of different processes (Fenton, photo-Fenton, UV, H_2O_2/UV, and UV/PILC) under these conditions: catalyst = $FeAl_{0.1}$-PILC, m_{cat} = 0.2 g, $V_{H_2O_2}$ = 2 mL, T = 25°C. All tests were performed without pH regulation, at natural pH of the textile effluent (12.4). Fig. 5 shows the removal efficiency for all the processes tested and Table 4 presents the results of COD removal, decolorization efficiency and pH at the end of reaction.

As can be seen from obtained results (Fig. 5), it is so clear that the photo-Fenton process is the most efficient in all processes studied, achieving 93% decolorization after 3 h and consuming 2 mL of H_2O_2. The self decolorization of effluent under UV irradiation only was negligible. Also, there is no enhancement of decolorization percentage when the catalyst was added without H_2O_2. On the other hand, photolysis of H_2O_2 exhibited a good decolorization

Table 4
Results of effluent treatment and the value of pH at the end of reaction

Process	% Decolorization	% COD removal	pH_f
Fenton	68.2	54.9	9.6
Photo-Fenton	93	88.8	8.5
UV	9	0.6	11.6
H_2O_2/UV	83.5	39.3	9.7
H_2O_2	4.5	0.03	10.32
UV/PILC	39.3	16.5	10.2

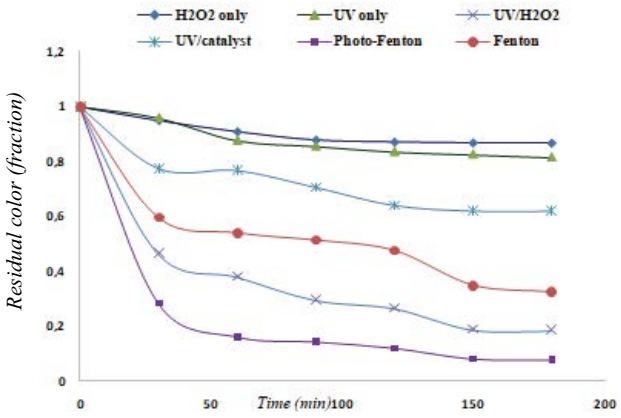

Fig. 5. Decolorization of the textile dyeing wastewater: comparison between the different processes evaluated.

percentage reach of 83.5%. In the dark condition, Fenton reaction allows to 68.2% of color removal only. This is can be explained by the effect of UV irradiation in the enhancement of the photocatalytic activity of $FeAl_{0.1}$-PILC.

Table 4 shows the percentage of decolorization for each treatment proposed. All AOPs resulted in a decolorization greater than 50%, being the photo-Fenton process the most efficient and faster one.

Following the trend of the mineralization process, the photo-Fenton reaction resulted in 88.8% of COD abatement after only 3 h. Similar results have been also reported by Vilar et al. [25] and Soares et al. [26] for the treatment of real textile wastewater.

According to the predicted results, we choose to study the parameter effect of effluent degradation via photo-Fenton process under UV irradiations and with $FeAl_X$-PILC.

3.4. Effect of different parameters

3.4.1. Effect of catalyst mass

The effect of catalyst mass on textile effluent degradation in the FeAl-PILC/H_2O_2 system was evaluated. To investigate the role of the mass on the catalytic process, three experiments (0.1; 0.2 and 0.3 g) were carried out while the other conditions were maintained constant. As the mass of catalyst is increased (Fig. 6), the rate of effluent decolorization increases. This can be explained by more

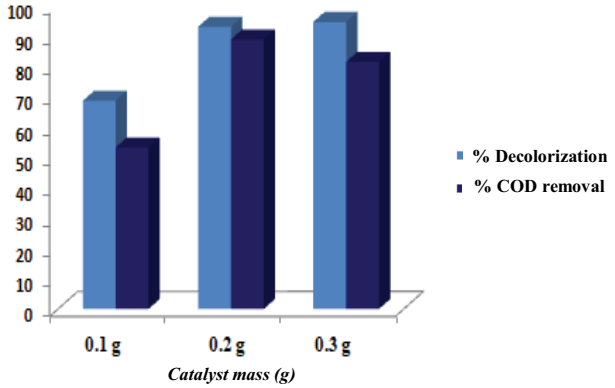

Fig. 6. Effect of catalyst mass on textile wastewater degradation.

accessibility of active site in the catalyst surface, which reacts with hydrogen peroxide to produce more radicals [27]. Hence, 0.2 g has been taken as optimum catalyst loading for further experiments.

3.4.2. Effect of H_2O_2

In the heterogeneous Fenton reaction, the concentrations of oxidant and catalyst mass are essential to the reaction and directly affect the concentration of active radicals produced for pollutant degradation. We investigated the effect of hydrogen peroxide on the degradation process of real wastewater by increasing the volume from 1 to 3 mL. As can be seen in Table 5, the increase of H_2O_2 volume enhances the increase of the rate of decolorization and COD abatement too. These results can be explained to the fact that the increase of oxidant concentration allows the increase of $HO^{\bullet}$ radicals in the reaction medium.

3.4.3. Effect of the amount of iron loading

Iron species in the catalyst provide active sites for the activation of hydrogen peroxide and consequently effluent decomposition. So, the effect of Fe(III) loading on catalytic activity has been evaluated using 0.2 g of catalyst $FeAl_X$-PILC (Fig. 7).

The degree of decolorization increases with the increase of the amount of iron loading. The catalyst $AlFe_{0.1}$-PILC presents the highest % decolorization and the best COD abatement. Also, the catalyst $FeAl_{0.5}$-PILC presents the best activity compared with the other catalysts. This is due to the presence of a high amount of iron, which is the principle element in the Fenton process.

3.5. Textile effluent characterization after photo-Fenton treatment under optimal conditions

According to the above-mentioned results, in this section, we are interested in the photo-Fenton treatment of real effluent under the optimal conditions and we presented the most physicochemical characteristics before and after treatment.

Table 6 shows the most characteristic of the effluent before and after 3 h of treatment with heterogeneous

Table 5
Effect of H_2O_2 concentration on textile wastewater degradation

$V_{H_2O_2}$ (mL)	% Decolorization	% COD removal
1	68.6	34.6
2	93	88.8
3	85.7	65.3

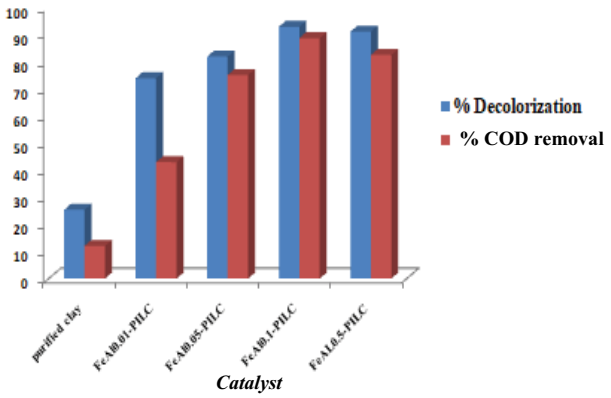

Fig. 7. Effect of the amount of iron loading in the catalyst on textile wastewater degradation.

photo-Fenton reaction under ultraviolet irradiations. It can be seen from Table 6 that the photocatalytic activity of the prepared catalyst varied in the same order compared with the degradation by Fenton-like process. The % decolorization after photocatalytic treatment under UV irradiation clearly indicates a highest activity of FeAl-pillared clay. Also, physicochemical quality indicators of effluents decrease considerably compared with them before treatment. The pH reaches a neutral value; therefore, it can be released in aquatic medium (6 < pH < 8.5) (NT 106.2 (1989)) [28]. Also, it is the case for COD value (88.8% of COD removal) respects the condition for releasing in public sewage systems, yet in aquatic environment. Also, the color removal was achieved 93%, which is considered an efficient result. Fig. 8 shows the raw and the treated effluent at optimum conditions with pillared clay.

According to these results, the water can be re-used in agriculture since it obeys the norms for reuse in agriculture (NT 106.3 (1989)) [29], especially it can be used for vegetation adapted to high salinity.

3.6. Comparison between actual and previous studies

Recently, researchers are more and more interested in heterogeneous Fenton oxidation process and many studies have been published about this method, since it is a simple process, which has several benefits such as: operation at natural pH, using of natural catalysts, cost effectiveness and no risk of secondary pollution. According to our knowledge from literature, there is no work about real indigo dyeing effluent removal by heterogeneous photo-Fenton process using pillared clay. But, there are a few studies that report the treatment of this effluent by electrocoagulation process as can be seen in Table 7. Also, heterogeneous photocatalysis was reported as solution for textile wastewater degradation using TiO_2 and doped TiO_2 as photocatalysts. On the other hand, homogeneous photo-Fenton process was also applied. For this reason (as shown in Table 7), we compared the actual study with other studies, which used Fenton oxidation techniques to remove other textile effluents. In terms of process also, we compared this work with other applied process for real textile effluent.

Table 6
Most characteristics of real wastewater before and after photo-Fenton treatment

Parameters	Before treatment	After treatment	Tunisian standard (for river discharge)
pH	12.4	8.5	6.5–8.5
BOD_5	600	90	30
COD	1,400	200	90
Color	3,500	190	70
Suspended solids	200	Traces	30
Total hardness	100	Traces	5
Oil and grease	10	Traces	20
Chloride	1,700	600	600

Except pH, the units of all other parameters are mg per litre.

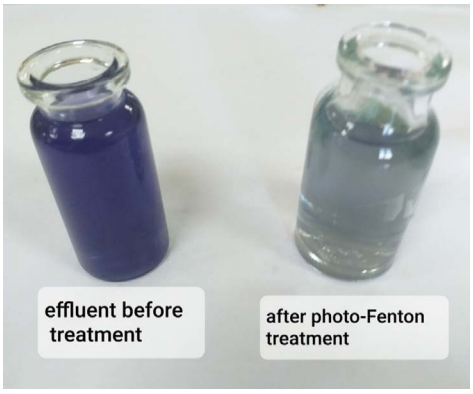

Fig. 8. Textile effluent before and after photo-Fenton treatment at optimum conditions.

4. Conclusion

In this study, textile dyeing wastewater with dark blue color, alkaline pH and presents a high organic load due to the mixture of recalcitrant dyes and chemicals used in the dyeing process, was treated before the discharge into the water bodies. Advanced oxidation processes were tested for the decomposition of textile effluent under UV light irradiation. Obtained results confirm that the photo-Fenton treatment was the most efficient of all processes studied. The developed material used as catalyst is pillared clay with mixed Al-Fe pillars between the clay layers. The photocatalytic activity of as synthesized samples was examined by photocatalytic degradation of real effluent under UV-visible light irradiation. Above-mentioned discussions

Table 7
Comparison between actual and previous studies

Process	Effluent	Used catalyst	% Color removal	% COD removal	Reference
Adsorption onto purified clay	Textile effluent	Purified clay	–	70	[30]
Heterogeneous Fenton process	Textile effluent	Zero valent iron	70	55	[31]
Fenton-like process	Textile manufacturing plant	Scrap zero valent iron (SZVI)	95	72	[32]
Heterogeneous photo-Fenton	Textile effluent	Fe-Al-PILC	93	88.8	This work
Heterogeneous Fenton process	Real textile effluent	Fe/activated carbon	96	66.3	[33]
Homogeneous photo-Fenton	Textile wastewater stream	–	96	20	[34]
Heterogeneous photocatalysis	Wastewater effluent	Ag-doped TiO_2	–	80	[35]
Heterogeneous photocatalysis with H_2O_2	Textile printing wastewater	TiO_2 (Degussa P25, Essen, Germany)	72	58	[36]
Chemical coagulation–electrooxidation	Textile wastewater	–	100	93%	[37]
Electrocoagulation	Textile effluent	–	90	81	[38]
Electrocoagulation	Textile effluent	–	89.2	76.1	[39]

indicate that the prepared catalyst exhibits good photocatalytic activities on mineralization of a real wastewater solution. Finally wastewater presents a COD below 250 mg L^{-1}, pH 8.5 and uncolored. It respects the condition for releasing in public sewage systems.

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