



Textile industry effluents treatment by wet catalytic oxidation with hydrogen peroxide in granitic laterite presence

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Abstract: Although water is useful for life, 80% of wastewater continues to be discharged into the aquatic environment without prior treatment, and industry bears a large part of this responsibility for the discharge of pollutants. This situation is sparking protests across the world due to rapid growth in environmental pollution caused by growing industries and population. Wet oxidation is one of the methods used to address this concern. In this present work, iron-rich granitic laterite was used as a catalyst with hydrogen peroxide as an oxidant for its affordable cost. The objective is on the one hand to determine the optimal conditions for the treatment of wastewater from the textile industry and on the other hand to apply it to a real effluent. Thus, pH influence's study showed that the oxidation reaction is observed more in acidic and neutral environments than in basic environments. The study continued under these conditions made it possible to study the influence of the quantity of laterite in these environments (acid and neutral). The results indicated that in a neutral medium with 5 g of laterite, a discoloration of at least 95% is obtained and the more the mass evolves, an interference is observed between the color of the treated water and that of the laterite. On the other hand, in an acidic environment, the more the mass of laterite increases, the more the discoloration improves with a reduction of 96% at 20 g of granitic laterite. In order to reduce the costs linked to lowering the pH and its correction after treatment in an acidic environment, research in a neutral environment was carried out to determine the best reduction rates as well as the various associated parameters for good color degradation. Thus, by varying the volume of the oxidant, the results showed that the latter must be 10% of the volume of water to be treated in order to obtain a good reduction. The possibility of reusing the catalyst in the treatment process also gave very satisfactory results in seven tests with at least 90% reduction rate of discoloration.

1. Introduction

Textile industries are faced with the management of waste released into nature without prior treatment. According to [UNEP \(2017\)](#), the main sources of organic pollutants in Africa's water resources are due to these industries and agricultural development. Indeed, in these industries, the dose of dye

used exceeds that which is necessary to obtain vivid textures for socio-economic reasons. Thus, to improve the performance of the texture, companies create stable dyes which combine chemical structures which are difficult to treat by traditional treatment methods water depollution with the consequences of disturbances in the pH values of the aquatic environment, diseases carcinogens, degradation of the photosynthetic ecosystem (Aravananan *et al.*, 2022; Medjahed *et al.*, 2013). Given this situation, the search for adequate treatment methods to effectively eliminate or transform these pollutants into organic matter capable of being eliminated by traditional water treatment methods is essential. To treat industrial wastewater, physical techniques such as adsorption are used, but they are limited to transferring the pollutant from the effluent to the adsorbent without degrading the pollutant (Kpinsoton, 2019; Rais *et al.*, 2008). Methods such as advanced oxidation processes and biological approaches have also experienced limitations in the treatment of textile industrial wastewater (Zahid *et al.*, 2020; Oudghiri-Hassani (2018)). However, even the most resistant organic pollutants can be transformed into inorganic compounds (water, carbon dioxide and salts) through chemical oxidation processes (Stephen *et al.*, 2023). Wet oxidation processes are one of these chemical oxidation processes. With the use of synthetic or natural catalysts (laterites), these processes are very effective in optimizing degradation yields (Benjelloun *et al.*, (2017); Titi *et al.*, 2020). Wet catalytic oxidation is one of the known oxidation processes operating on the basis of the catalytic decomposition of hydrogen peroxide by ferrous ions at high temperatures and pressures. Granite laterite, which is typically found in African countries exposed to equatorial climates, including Ivory Coast, could serve as a catalyst to supply the ferrous ions. Therefore, the main objective of this study is to develop low-cost catalytic materials to create a sustainable and efficient catalytic oxidation effluent treatment process in a Sub-Saharan African context using granitic laterite.

2. Methodology

2.1 Granitic laterite sampling

Two samples were taken for fundamental mineral analysis. These samples were stored in boxes for analysis in the laboratory. It is important to note that these samples were washed with water to remove the top layer of sand. After that, they were dried for 24 hours in an oven at 105°C before being crushed and sieved to reach a particle size of between 40 and 80 µm. Each sample was duplicated, one for analysis and the other for controls.

2.2 Measurement with a Niton XL3t® portable XRF spectrometer

Before analysis, the samples were taken using a spoon and placed in a dish, the lower part of which was covered with a transparent 6 µm mylar film to allow the X-ray to pass through. In order to obtain a flat surface conforming to the dimensions of the cup, the samples were compacted and tamped. Before the start of the measurements, a calibration of the device was carried out with the “cal Cheik” standard sample. After turning on the device and following the appropriate procedures, the “Allgeo and Soils & Minerals” measurement mode was used. In the Allgeo mode, the selection of the soil option was made in order to know the concentrations of the elements in ppm. The measurement was carried out for 90 seconds, each 30 seconds of which corresponded to each excitation energy level, hence three (3) filters. To obtain the same percentage concentrations, the “Soil and Ores” mode was then used to choose the Ores option. This measurement is carried out for 120 seconds, of which each 30 seconds also corresponds to an excitation energy level, including a total of four (4) filters. The most sought-after element is the iron concentration in rocks. The results obtained were analyzed using NDT and Excel software.

2.3 Characterization of wastewater from the textile industry

TEX-CI wastewater from the production chain is collected through channels to a collection point. Our samples were therefore taken at this grouping point noted respectively J1P1; J2P2 and J3P3. The characterized parameters and their measurement methods are recorded in [Table 1](#).

Table 1: Parameters analyzed on textile wastewater

Parameters	Methods
Temperature (°C)	<i>in situ measurement with HANNA multiparameter</i>
pH	
Conductivity (µS/cm)	
rH (mV)	Nephelometric NF EN ISO 7027
Turbidity (UNT)	
SS (mg/L)	<i>Membrane filtration NF T 90-105-2</i>
COD (mgO ₂ /L)	Potassium dichromate titration
BOD ₅ (mgO ₂ /L)	NF T90-101
	Respirometric (Oxi-Top))
T OC (mg/L)	NF EN 1899-1
TN (mg/L)	Determination by TOC meter
	TN of N/C

2.4 Spectrophotometric scanning

Spectrophotometric scan made it possible to determine the concentration and to know the maximum wavelength at which the samples can be read for the rest of the work. Not having worked on synthetic samples and not knowing the initial concentrations, we did not establish a standard. Our samples are composite and constitute the composite sample of one day of production at TEX-CI. Thus, the scanning was done in the visible (400 nm and 800 nm) in order to globally see the maximum wavelengths specific to each sample.

2.5 Wet oxidation protocol

Initially the reactor is rinsed with distilled water. The heating plate is connected to the mains to adjust the set temperature (150°C in our case), then it is stirred at 400 rpm. After that, we took 100 milliliters of the solution to be treated from the reactor and added the amount of laterite corresponding to the reaction, followed by the amount of peroxide to the reaction medium. To avoid explosions during the reaction, the reactor is closed by checking whether the seal and the cover belt are properly installed. Once completed, the reactor is heated for ten minutes to the set temperature (150°C) before determining the reaction time which varies between 60 and 90 minutes. Before collecting the treated liquid, the reactor must be cooled to 50°C at least once the reaction is complete. To prevent hydroxyl radicals from continuing their activities, a few drops of ethanol are added to the treated water. The absorbance reading is taken with a spectrophotometer at the specific wavelength of the effluent. Concerning the choice of the clean oxidant for the gentle oxidation of wastewater from the textile industry, a sub-stoichiometric quantity of hydrogen peroxide (approximately 10%) is used by a hot catalyst (120°C) and under pressure (2 bars).

✓ *Carrying out the wet oxidation process*

Following the procedure, one liter of 10% concentrated hydrogen peroxide solution was prepared, then 20 grams of laterite were mixed with 200 milliliters of raw water in different reactors. Each reactor is assigned a precise heating time in order to achieve the discoloration kinetics over a period of one

hour. This was done at different pHs (pH = 4; pH = 7; pH = 9; pH = 12.6) to observe the pHs that gave the best decolorization rate. The volume of hydrogen peroxide was constant and set at 10 mL per reactor to study the influence of pH. The pH values whose results were satisfactory were retained for the remainder. Concerning the study of the influence of the mass of laterite on the treatment, the masses of laterite chosen varied between 5 and 25 g with 10 mL of hydrogen peroxide fixed at these different pH values (pH=4 & pH= 7). The study of the impact of the hydrogen peroxide concentration was carried out on a pH = 7, because this does not require the purchase of additional reagents to lower the pH to an acidic environment. Indeed, good discoloration was observed at a low dose of laterite, which no longer required continuing the work at pH = 4 characteristics of the acidic environment. In order to observe the evolution of the discoloration, the volumes of peroxide were varied (1 mL, 2 mL, 5 mL, 10 mL, 15 mL and 25 mL). Remember that all of these reactions were carried out at a constant temperature of 150°C using a heating plate whose temperature is controllable and at a pressure assumed to exceed 2 bars, because the medium was hermetically closed and under continuous stirring.

✓ *Tests of reuse of granitic laterite in wet processing tests*

Decolorization kinetics was carried out for a period of 1 hour repeated 7 times with the same laterite under optimal conditions in order to evaluate the reuse rates of the laterite. The treatment began with the same quantity of laterite for one hour, then repeated for seven hours in order to determine when these laterites were able to release the most iron possible into the reaction medium. This number of revolutions of use also informed us at what time it is necessary to change the laterite and also on its effectiveness.

3. Results and Discussion

3.1 Characteristics of granitic laterite at XRF

Figure 1 presents the contents of major elements in granitic laterite. It appears from its exploitation that the laterites used in our work have higher iron contents than aluminum. The results of the XRF analyzes using the Test All geo and Mining modes respectively revealed a content of 123,760.8 ppm or 47.93% of Iron against a content of 102,282.8 ppm of Aluminum or 10.80%. The results also revealed a high silica content (SiO₂) of 39.62% or 102,282.81 ppm, which justifies that the rock is still under alteration. The ratio $R = \text{SiO}_2 / (\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3)$ gives a value of $R = 0.67$, much lower than 1.33.

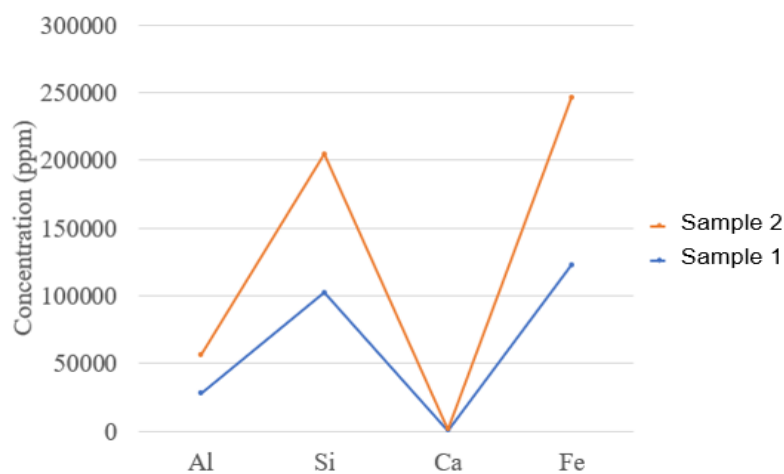


Figure 1. Major element contents in granitic laterite

Figure 2 shows the metallic trace elements found in the rock samples. It shows a large titanium peak. The average value of 16.36 ppm in arsenic shows that Arsenic, whose pollution limit is 40 ppm in the environment, is not polluting. Furthermore, the average value of chromium content of 437.03 ppm is higher than the limit value which is 150 ppm.

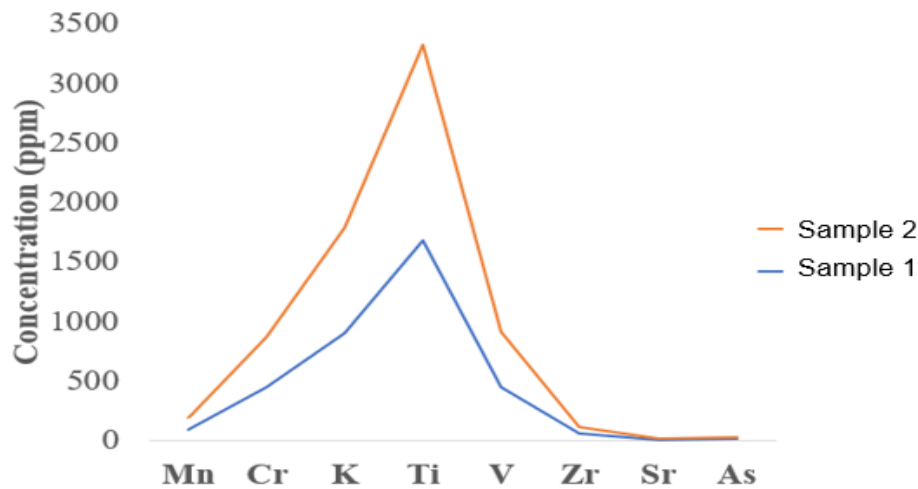


Figure 2. Trace metal contents

3.2. Characteristics of wastewater from TEX-CI production

3.2.1. Physico-chemical parameters of samples

The results of the parameters used to characterize the wastewater from TEX-CI are in table 2. It appears from the analysis of the table that the wastewater from TEX-CI Bouake is strongly basic with pH values between 12, 47 and 12.66 and largely exceeding the limit set by Order No. 01164/MINEEF/CIAPOL/SDII of November 4, 2008 on discharges and emissions into the environment which must be between 5.5 and 9.5 if a chemical treatment is applied. Regarding COD, the values are also high ranging from 3784 to 9578 mgO₂/L. The results show strong electrical conductivity. Suspended solids are relatively low while water turbidity is high.

Table 2. Sample characterization of daily composite samples of TEX-C

Parameters	Samples			Mean ± standard deviation
	J _{1P1}	J _{2P2}	J _{3P3}	
pH	12.60	12.47	12.66	12.57±0.09
T (°C)	28.1	28.8	33.3	30.06 ± 2.82
TDS (mg/L)	60.3	72.3	87.1	72.20 ±13.37
Conductivity (µS/cm)	102.1	120.3	144.7	122.36 ± 21.37
Turbidity (UNT)	87.2	24.9	127	79.70 ± 51.46
Redox potential (mV)	302.4	283.8	291.9	292.70 ± 9.32
SS (mg/L)	14.5	25.5	74	38.00 ± 31.68
COD (mgO ₂ /L)	3784	6804	9578	6722.00 ± 2897.87
TOC (mg/L)	796.6	670.4	2568	1345.00 ± 1061.02
TN (mg/L)	40.4	30.6	197.96	89.65 ± 93.92

3.2.2. Spectrophotometric scanning

The highest peaks are between 600 and 700 nm. The second scan carried out in this interval made it possible to specify the maximum wavelength of each sample. So, for sample J1P1, the maximum wavelength was around 662 nm very close to 664 nm at which the other readings were taken. Regarding sample J2P2 and J3P3, the maximum wavelengths were 660 nm and 680 nm, respectively. **Figure 3** lists the different spectra observed during the scan.

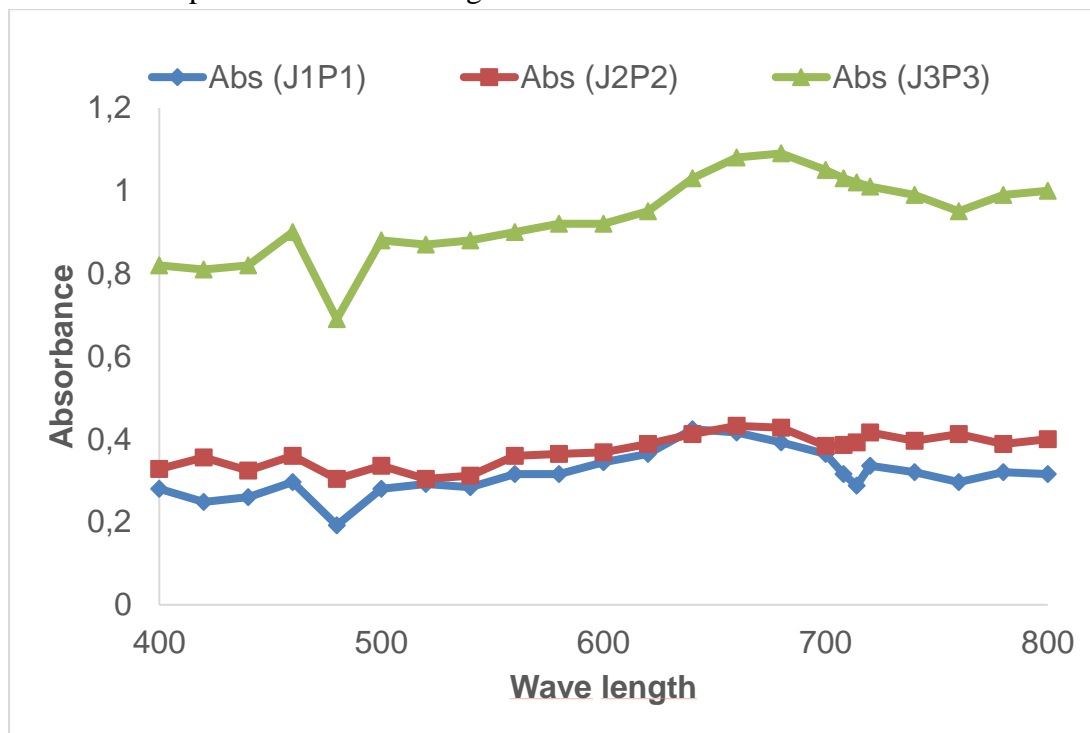


Figure 3: Spectrophotometric scanning of raw effluent

3. Determination of the operating parameters influencing the wet oxidation reaction

3.3.1. Study of the influence of pH variation

As discoloration is observed over time, the results obtained are illustrated in **Figure 4**. It presents the rate of color degradation as a function of pH. It appears from its operation that in the first ten minutes, the discoloration is more extensive, reaching more than 70% for pH = 4 unlike pH = 12.6 which reaches this percentage around the following 10 minutes. In addition, the results show a high reduction rate in acidic medium and in neutral medium of 96.45% and 94.75% respectively while that obtained in basic medium is 84.96%. Thus, we conclude that the more acidic the environment, in the presence of laterite and peroxide there is a good degradation of the color. Likewise, at pH = 9, a good and significant discoloration is also observed which can also be exploited to limit the costs linked to the use of chemicals to lower the pH to an acidic environment when it comes to treat large quantities of water.

3.3.2. Study of the influence of the laterite mass

Using laterite as a catalyst, very rich in iron previously proven by XRF analyses, the results obtained are presented in **Figure 5**. It presents the different reduction results obtained at different masses. The analysis shows that as the mass increases, the reduction improves. For the acidic pH, the mass of laterite giving the best reduction rate is 20 g. The other low results in acidic environments are justified by the crumbling of the laterite giving its color to the treated water, which reduces the

reduction despite filtration. Concerning the neutral pH, we already notice that at 5 g of laterite, a reduction of 95.5% is obtained after one hour of treatment. By changing the masses, the same observation made at the acidic pH level is also observed, but this time with increasingly weaker coloring than in an acidic environment. We can understand that it is the quantity of residual iron in the medium after treatment which influences the coloring of the water after treatment and consequently the reduction of discoloration. We can also remember that in a neutral environment the mass of suitable laterite is 5 g for a volume of 100 mL of raw water to be treated. So, in the rest of the work, we continued with this mass of laterite.

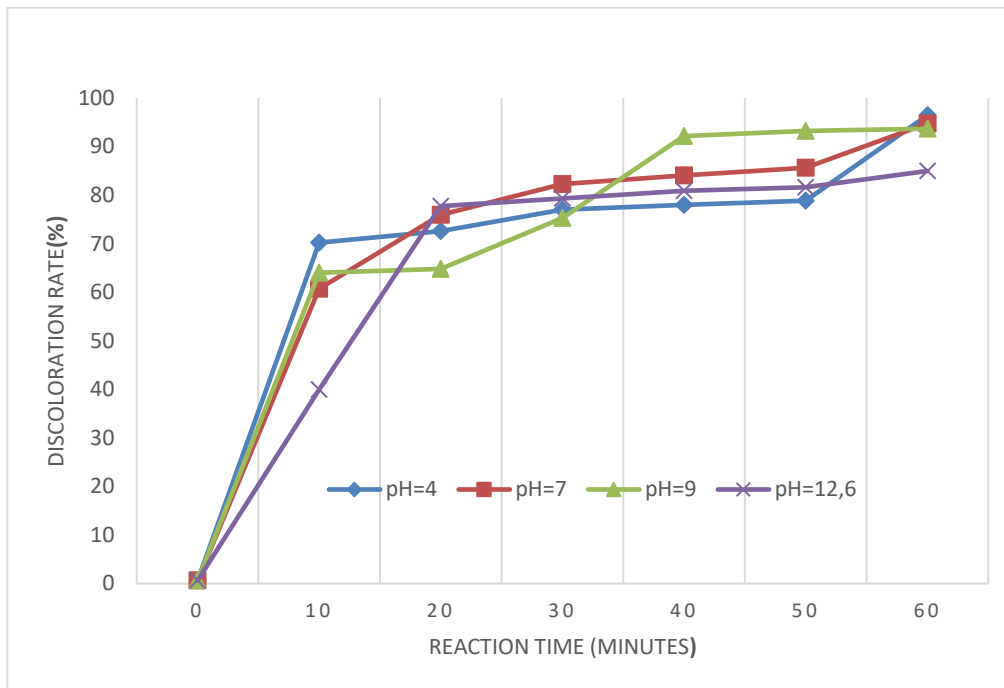


Figure 4: Effect of pH on decolorization of TEX-CI wastewater

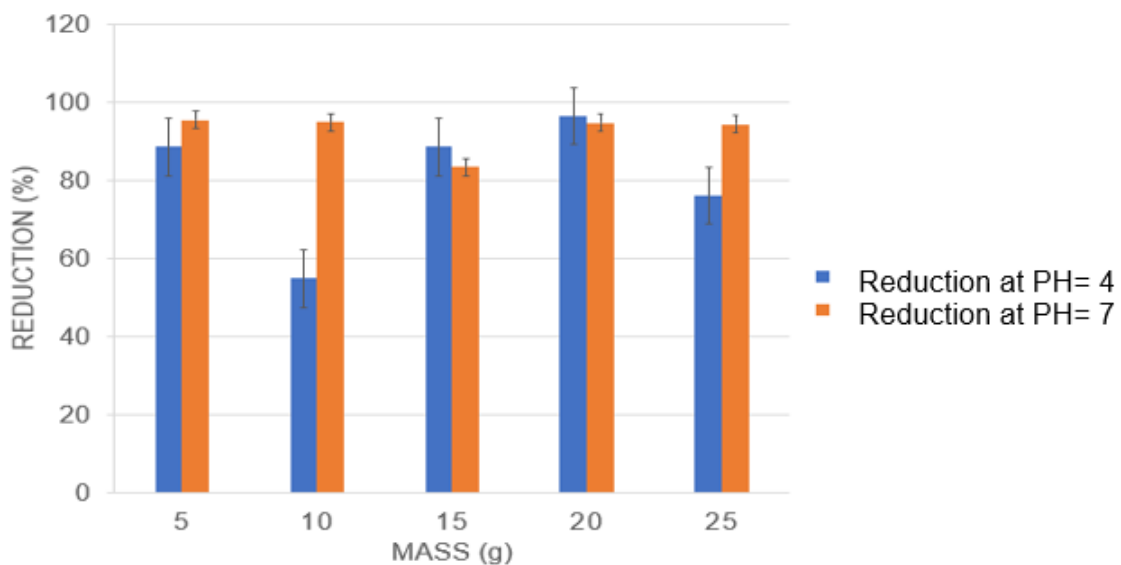


Figure 5: Laterite mass reductions

3.3.3. Study of the influence of hydrogen peroxide concentration

By varying the volumes of the oxidant ranging from 1 mL to 20 mL, the discoloration kinetics achieved gives the results presented in Figures 6 and 7. It emerges from the exploitation of Figure 6, that the greater the quantity of peroxide in the reaction medium increases, good discoloration is obtained. In addition, the best discoloration is obtained with 10 mL of hydrogen peroxide. Beyond this volume, the discoloration remains more or less constant. A reddish color is noticed in the environment which can result in the color of the laterite which interferes with that of the treated water. In addition, the results show a reduction rate of more than 94% at 10 mL of hydrogen peroxide and a stability of at least 90% when the volume exceeds 10 mL. These different reductions are illustrated in Figure 7.

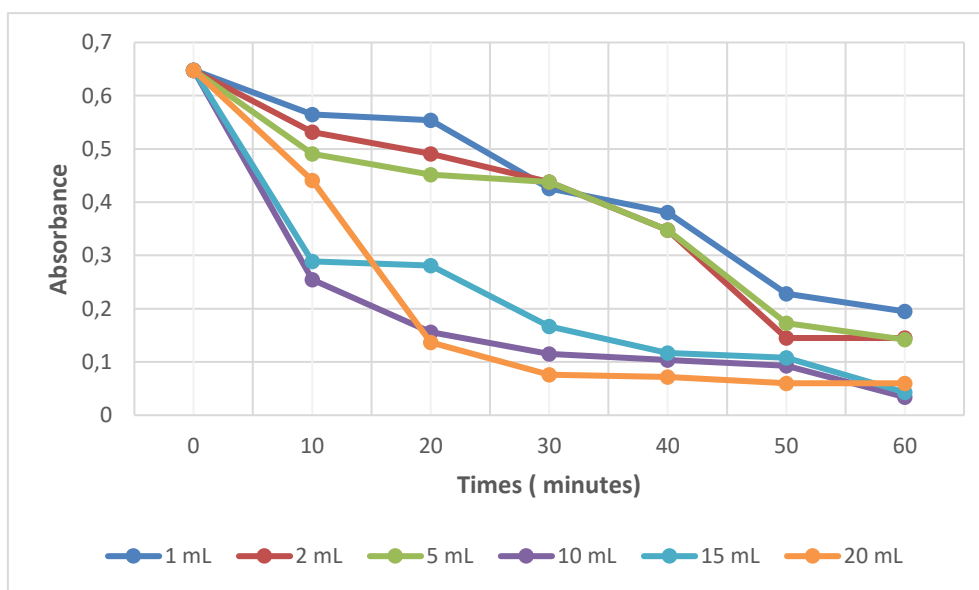


Figure 6: Effect of peroxide volume on wastewater discoloration

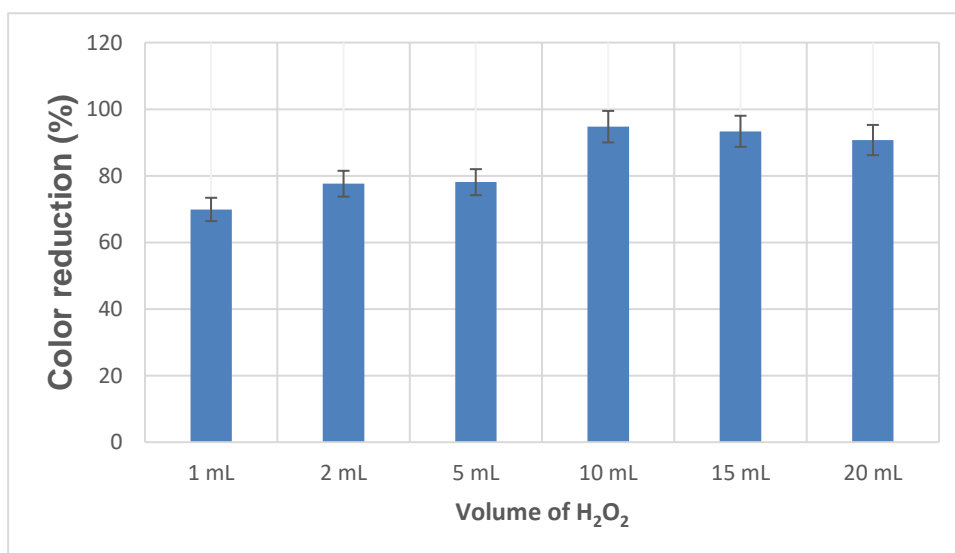


Figure 7: Reductions depending on the quantity of H₂O₂ added to the reaction medium

✓ Optimal study conditions retained

After the study carried out by varying the different parameters influencing wet oxidation, the following parameters were therefore retained for the continuation of the TEX-CI wastewater discoloration tests. Thus, the temperature remains fixed at 150 °C, the pressure assumed always greater than 2 bars since the medium remains hermetically closed throughout the heating time and because the

more the internal temperature increases, the more the pressure increases. The quantity of laterite in the reaction medium is now 5 g and the volume of peroxide is 10 mL. These conditions were therefore used to carry out other tests over hours in order to judge the effectiveness of the process.

3.3.4. Evaluation of parameters after treatments

The previous optimal conditions, tested on the real textile effluent, made it possible to follow the evolution of the pollution reduction (Turbidity, COD and SS) of this effluent (table 3). According to the results, the COD reduction rate is very low despite the fact that the effluent is loaded and composed of several components. The maximum turbidity reduction rate is 82.79%. For SS, a reduction of 86.89% was recorded. These average values for turbidity and SS are due to the fact that the discharges from this industrial complex are very colorful and concentrated.

Table 3: Changes in the reduction of certain physical and chemical parameters after treatment

Times (min)	Turbidity (UNT)	DCO (mgO ₂ /L)	SS (mg/L)
0	87.2	3784	14.5
60	15	3306	1.9
Reduction Rate (%)	82.79	12.63	86.89

3.3.5. Reuse rate of granitic laterite in the treatment of textile industry wastewater

Under the optimal conditions retained and predefined previously, we carried out an evaluation of the reuse of laterite over seven (7) hours of grinding while using the same laterite. The results obtained are illustrated in Figure 8. It appears from the exploitation of the latter on seven tests carried out that the rate of reduction of discoloration obtained was greater than 90% with a minimum reduction of discoloration obtained at 95.1% in the last test. This reduction rate increases from the first trial to the fifth trial, reaching a maximum of 97.99% before dropping slightly towards 95.1%. Thus, we can say that the laterite used is effective in the treatment and can be reused more than 6 times to bleach textile industry water with a reduction of at least 90% for a treatment duration of 1 hour.

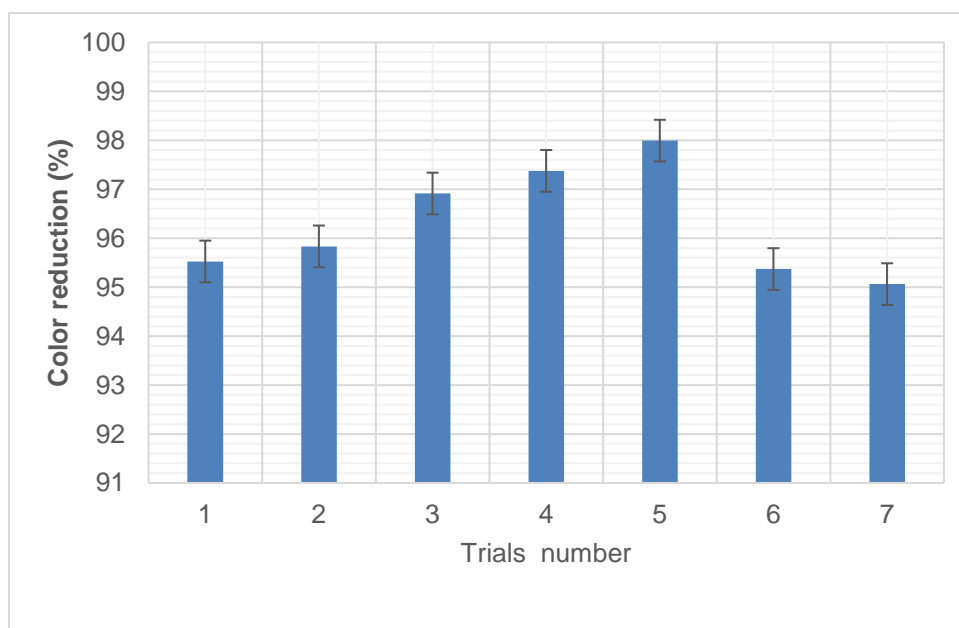


Figure 8: Evaluation of the laterite reuse rate

4. Discussion

The ratio $R = \text{SiO}_2 / (\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3)$ gives a value of $R = 0.67$, well below 1.33, which allows us to say that the rocks used are indeed granitic laterite (Autret and Tran, 1982). This high iron content is essential for a good reaction with hydrogen peroxide to break down the dyes. Once in solution by heating, the Fe^{3+} ions will react with hydrogen peroxide to produce $\text{OH}\cdot$ radicals which in turn degrade the dyes.

Zirconium is not found in large quantities in our samples. Previous work by Bensouilah and colleagues, using Zirconium as a catalyst, showed very effective results with 90% degradation of bisphenol A at 100°C with a pressure of 20 bars for 3 h (Bensouilah *et al.*, 2020). This element could also be effective for good wet oxidation if its content was high in our samples. It is the same for Manganese coupled with Iron and copper used by Falcon (2005) for the degradation of organic effluents from the chemical industry with reduction rates of 91% in TOC in the following conditions: $\text{pH} = 3.5$; $T = 98^\circ\text{C}$; $P = 1 \text{ atm}$; $t_s = 1 \text{ h}$, using Fenton's reagents, but at elevated temperature. Furthermore, the average value of chromium content of 437.03 ppm is higher than the limit value which is 150 ppm. This high concentration could be explained by the difficulty of alteration, because chromium minerals are minerals fusing at high temperatures generally found in basic, ultrabasic and metamorphic eruptive rocks. This element can be considered a metallic pollutant.

The wastewater analyzed is very basic. These high values of Hydrogen potential are justified by the use of soda in almost the entire production chain from boiling to washing after printing the designs in the fabric. These high values are also characteristic of the textile industries. The recorded COD values are high. High COD values of between 1600 and 3200 mgO_2/L were also observed during the work of Hussain *et al.* (2020). This high chemical oxygen demand of wastewater is believed to be due to the presence of oxidizable compounds, which are used in various stages of the process. Suspended solids contents are relatively low while water turbidity is high. This state of affairs could be justified by the essential use of dyes that are difficult to decant and which remain in the column.

Regarding the reduction in the reduction both in acidic and neutral environments when the mass of laterite increases is due to the quantity of high residual iron in the reaction medium after treatment. This slight drop in the reduction beyond 10 mL H_2O_2 could result in the interference of the color of the laterite with that of the treated water. It may also be due to the fact that the laterite particles collide, which weakens their structures, also causing the release into the environment of a fairly large quantity of soluble residual iron. On the actual effluent when the volume of hydrogen peroxide increases from 1 mL to 10 mL, we obtain a reduction of 12.63%. This could be because the H_2O_2 present in the industrial wastewater either increased the COD values of the wastewater sample or hindered the COD measurement process because the H_2O_2 forms a complex with the dichromate potassium (Berner *et al.*, 2020).

Conclusion

Improving wastewater management in the textile industry is a major challenge for manufacturers in developing countries, who need to reconcile industrial development with environmental protection. Several methods are used to treat textile industry wastewater. In the present work, we have proposed and tested a treatment method called wet catalytic oxidation. This method uses hydrogen peroxide as the oxidant and granite laterite, containing around 48% iron as the major element required for the catalytic reaction, as the catalyst. This catalytic reaction, which is highly effective in an acidic environment, has been carried out in a neutral environment for economic reasons, as acidifying the effluent with an acid solution represents a significant treatment cost. With a mixture of 5g of laterite

and 10mL of oxidant, an abatement of at least 95% is achieved. It should also be noted that the catalyst can be reused while retaining its catalytic potential at around 90% decolorization of the textile effluent. This treatment reduced turbidity to 82%. Last but not least, this catalytic oxidation method was highly effective in treating wastewater.

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Compliance With Ethical Standards: This article does not contain any studies involving human or animal subjects

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