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Treatment of a dye solophenyle 4GE by coupling electrocoagulation / nanofiltration

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Abstract. The study contributes in the treatment of waste generated by the textile complex cotton of Draa Ben Khedda, Algeria. The azo dye "Direct Red Solophenyle 4GE" represents the base particle of the discharges and electrocoagulation with nanofiltration are used as a means of treatment. The solar photovoltaic is suitable for electrocoagulation bach gives the best reduction 37% for a dye concentration of 7.21 mg/L ([NaCI]_{added} = 1 g/L; j = 25.2 mA/cm²). Coupling methods (electrocoagulation-nonofiltration) gives a complete discoloration rejecting concentration 52.4 mg/L (pHi = 7.6, [NaCI]_{added} = 3 g/L, j = 2.13 mA/cm²). The result shows the coupling efficiency with a reduced amount of resulting slurry at the end of treatment.

Keywords: rejection textile; aluminium electrodes; elecotrocoagulation; nanofiltration; organic membrane; solar photovoltaic

1. Introduction

The population growth has been very rapid development in recent years and in parallel textile demand can only increase in quantity and quality. Faced with this situation, the textile dyeing is a wide variety and discharges generated by the coloring operation pose serious problems to the environment. Our study is based on the processing solutions based on azo dyes used in the textile complex Draa Ben Khedda / Tizi Ouzou, Algeria. Azo dyes is currently the most widespread in terms of the application, they represent more than 50% of world production of dyestuffs (Øllgaard *et al.* 1998). They are characterized by the presence in the molecule of an azo group (-N = N-) linking the two benzene rings. Azo dyes are divided into several categories: direct basic dyes, acids, and soluble reactive water and disperse dyes and mordant non-ionic water-insoluble. It is estimated that 10-15% of the initial quantity is lost during the dyeing procedure and are discharged untreated into the effluent (Øllgaard *et al.* 1998, Christophe *et al.* 2001).

But these carcinogenic organic compounds are resistant to treatment processes usually

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implemented and are highly resistant to biodegradation (Pagga and Brown 1986, Brosillon *et al.* 2008). The dye is considered direct solophenyle 4GE red (R-SO₃ Na) (Direct Red 277) of anionic character and of which the solubility in water equal to 60 g/L. The solubility of these dyes in water is reduced by the addition of neutral salts of alkali metals (sodium sulfate, sodium chloride, etc.).

Textile industry effluents present two main problems: on one hand, they have high COD values and on the other hand, they contain organic compounds like phenols, dyes, tensioactives, some of them very toxic and prevent their direct treatment in a biological plant (Aouni *et al.* 2009). To solve this problem, some combinations of physical–chemical techniques have been tested, as chemical coagulation followed by sedimentation and absorption,ozonization and oxidation processes (Kobya *et al.* 2006, Canizares *et al.* 2006, Senthilkumar *et al.* 2012). Our work focus on the treatment of this wastewater by coupling electrocoagulation and nanofiltration.

The EC is an electrochemical method was used for the purification of many types of water and wastewater (Ün *et al.* 2006). This technique is characterized by its simple equipment, its ease of implementation and a small amount of sludge generated, the coagulant leads at an appropriate pH, the formation of insoluble metal hydroxide which is able of the removal of a wide variety of pollutants (Adhoum and Monser 2004). Previous studies conducted with textile dyes and real textile effluent have shown that electrocoagulation using aluminum, iron or stainless steel electrodes is a very effective means for color and COD removals (Kobya *et al.* 2003, Essadki *et al.* 2007, Mollah *et al.* 2001, Balla *et al.* 2010).

Other recent studies have focused on the treatment of wastewater using membrane processes. In fact, membrane technologies offer an important solution in environmental issues such as pollution reduction and water reuse, recycling of valuable components from waste streams (Marcucci *et al.* 2001). Untreated textile effluents cannot be used directly as an influent to nanofiltration or reverse osmosis membranes due to the high solids concentrations. Thus, it is necessary to carry out a very exhaustive pre-treatment in order to avoid fouling and membrane deterioration (Sostar-Turk *et al.* 2005, Fersi *et al.* 2009). Membrane technologies are frequently used for purification of textile wastewater (Voigt *et al.* 2001, Tang and Chen 2002, Sung-Pet *et al.* 2004, Akbari *et al.* 2002, Aouni *et al.* 2012). The use of membrane technology in combination with another treatment method was also demonstrated as a practical solution for treating textile wastewater (Wu *et al.* 1998, Dhale and Mahajani 1999, Aouni *et al.* 2009).

We note that the use of photovoltaic is not excluded from this study because it represents the energy supply in the electrochemical process.

The objective of this study is to educate the industry to enrol in a clean, economical and sustainable environmental policy.

2. Backgrounds study

The electrochemical coupling and membrane shows each use in the field of treatment of liquid waste, greater efficiency compared to chemical pretreatment (Mollah *et al.* 2001).

Electrocoagulation is an indirect electrochemical oxidation process wherein a redox reagent dissolved which exists, or is generated from the electrolyte or from the phase of the electrode in order to participate in the coagulation reaction. During the phenomenon of coagulation, the coagulant is usually added in the form of appropriate chemicals.

During the electrocoagulation process, the coagulant is generated in situ by electrolytic oxidation of an appropriate solid anode (Mollah *et al.* 2001) electrocoagulation (EC) is a technology that results from the interaction of three basic technologies that are: The electro-

chemistry, coagulation and flotation. (Holt et al. 2005).

It is based on the fact that the stability of colloids, suspensions and emulsions is influenced by the electric charges (Savaş Koparal and Öğütveren 2002). It refers to the electrochemical production of destabilizing agents that cause charge neutralization for the reduction of pollutants (Imen *et al.* 2011).

Adsorption phenomenon on the resulting complex ions of the anodic oxidation of the material phenomena can be added to the destabilization. When the electrode material used is aluminum, the reactions are as follow (Aouni *et al.* 2009)

• At the anode

$$\mathrm{Al}_{(\mathrm{s})} \to \mathrm{Al}_{(\mathrm{aq})}^{3+} + 3\mathrm{e}^{-} \tag{1}$$

• At the cathode

$$3H_2O + 3e^- \rightarrow (3/2)H_{2(g)} + 3OH_{(a0)}^- +$$
 (2)

• In the solution

$$Al_{3(aq)}^{+} + 3H_2O \rightarrow Al(OH)_3 + 3H_{(aq)}^{+}$$
(3)

Metal cations (Al^{3+}) form mono complexes with hydroxides ions (OH^{-}) such as Al $(OH)_{2}^{+}$, Al $(OH)_{2}^{4+}$ and Al $(OH)_{4}^{-}$ and poly complex such as Al₆ $(OH)_{15}^{3+}$, Al₇ $(OH)_{17}^{4+}$, AL₈ $(OH)_{20}^{4+}$, Al₁₃O₄ $(OH)_{24}^{7+}$, and Al₁₃ $(OH)_{34}^{5+}$, which transform finally into Al $(OH)_{3}$ according to complex precipitation kinetics (Can *et al.* 2003).

Formation rates of the different species play an important role in the decolorization process. Several interaction mechanisms are possible between dye molecules and hydrolysis products, and the rates of these depend on the pH of the medium and types of ions present. Two major interaction mechanisms have been considered in recent years: precipitation and adsorption, each one being proposed for a separate pH range. Flocculation in the low pH range is explained as precipitation, while that in the higher pH range (> 6.5) is explained as adsorption

Precipitation:

dye + monomeric Al
$$\rightarrow$$
 [dye monomeric Al](s)
pH 4.0 - 5.0 (4)

dye + monomeric Al
$$\rightarrow$$
 [dye monomeric Al](s)
pH 5.0 - 6.0 (5)

Adsorption:

 $dye + Al(OH)_3(s) \rightarrow \rightarrow [particle]$ (6)

$$[dye + monomeric Al](s) + Al(OH)_3(s) \rightarrow \rightarrow [particle]$$
(7)

At pH > 9, $Al(OH)_4^-$ is also present in the system.

Amorphous flocs Al(OH)₃ freshly formed have large surface areas that are beneficial for rapid adsorption of soluble organic compounds and trapping of colloidal particles. These flocs polymerize as

$$nAl(OH)_3 \rightarrow nAl_n(OH)_{3n}$$
 (8)

and they are removed easily from the aqueous medium by sedimentation and by H₂ flotation.

This first treatment of the solution has an advantage for the membrane treatment. Indeed, the electrocoagulation process allows reduction of the pollution load thus preventing the problem of membrane fouling. (Taleb-Ahmed *et al.* 2012).

In addition, the membrane process is an effective method for the treatment of colored, (Taleb-Ahmed *et al.* 2002, Annarosa and Drioli 2013) solutions especially those that contain chromium (+3 or +6) and solutions containing azo particles textile field (Nadjib *et al.* 2012).

3. Experimental

In the experimental part, we discussed solutions based on solophenyle 4GE straight red (Direct Red 277), the recipe used at the complex is as follows:

- 20 g Red Solophenyle 4GE;
- 20 g NaCl;
- 1 L of fresh water at 100 °C;
- 1 mL of fixative (flo-fixed RD).

The analysis is performed on branded devices (pH meter: HANNA pH; condustivity: HANNA condutivimeter EC214 meter, Turbidity: Turbe 550, UV-Visible spectrometer: SHIMADZU / UV-1800.

The wavelength of maximum absorption (Fig. 1) is 514 nm for the Red Solophenyle 4GE (RS) at pH study, based on the absorption maxima of the dye, we proceeded to establish the calibration curve, in order to estimate their concentration in solution.

The calibration curve for this dye is given by the right side of Fig. 2. The selected concentrations are prepared from mere recipe solution and whose values are getting close discharge concentrations of the complex.

The fixing of the dyes varies between 60% and 90% in the field of the textile, for this reason the concentrations of the treated solutions were selected (Hawkins *et al.* 1984, 1985, Nour El Houda *et al.* 2013).

Solutions of 200 mL are processed in batch mode of 200 mL with a basic concentration of dye equal to 20 g/L.

The experimental device (Fig. 3) of the electrocoagulation consists of an installation comprising a reactor with two aluminum electrodes connected to DC power supply PS-305D, both electrodes are rectangular plates placed parallel and separated from each other with an inter-electrode distance of 2 cm, the width is 5.3 cm and the length is 4.5 cm (active area = 23.85 cm²). Stirring is provided by a magnetic type agitator N0690/1. The electrolysis time is 1 hour.

For the Continuous mode, we chose a cell shown in Fig. 4 it contains an open electrochemical reactor which is equipped with two electrodes, aluminium anode (external anode) and the cathode of stainless steel connected to a DC power supply ps-305D. The diameter of the anode is 3.1 cm and the height is 14.5 cm (active area = $141,143 \text{ cm}^2$). The volume of the colored solution treated in each experiment is 6 L and the amount of NaCl added equal to 3 g/L. The rate of percolation, dye concentration and current density are considered in this part of study.



Fig. 1 Absorption spectrum red visible solophenyle 4GE



Fig. 2 Calibration curve of red solophenyle 4GE maximum absorption



*Legend: (1) DC power; (2) electrolytic cell; (3) aluminum electrodes; (4) magnetic agitator; (5) solar photovoltaic system



Fig. 3 Electrolysis cell batch

*Legend: (1) tank; (2) pump; (3) reactor; (4) DC power; (5) electrodes

Fig. 4 Reactor electrochemical cell in continuous process

As energy is a challenge of great importance in the future. We though it essential to size a solar photovoltaic system to power the electric generator used for electrocoagulation. It is essential to know precisely the energy requirement of a standalone installation to design a photovoltaic system adapted. The photovoltaic power generator to be installed is deduced from the calculation of the consumption of the application. It takes into account the power, voltage electrical appliances as well as periods of use. In our study the system used is the following:

* Photovoltaic Module 12 V-50 Wc (solar ring SM55);

- * Stationary Battery 12 V-12 Ah (VRLA Battery SUNLIGHT);
- Support module * aluminum frame;
- * Inverter 500 w (X-Power Inverter 150, 300 and 500);
- * Charge controller (Steca Solsum 6.6 c).
- Sample analysis is followed by:
- * pH meter (HANNA pH210);
- * Conductivity (HANNA / EC214);
- * Turbidity (turbe 550 type);
- * UV-visible spectrophotometry (Shimadzu / UV-1800)



*Legend: (1) Paper tray; (2) displacement pump; (3) and (6) pressure; (4) module nanofiltration; (5) recirculation of the permeate; (7) recirculation of retentate; (D) flow regulator; (P) pressure sensor; (T) temperature sensor; (8) heat exchanger

Fig. 5 Installation of nanofiltration process

The Installation of nanofiltration is illustrated in Fig. 5, it consists of a stainless steel cell, wherein the membrane is held in place by gaskets. The effluent is introduced into the input tray, cooled by a cryo-thermostat and circulating in the system by means of a volumetric pump. Filtration is carried out under the effect of a pressure difference created on both sides of the membrane and which is controlled by two pressure regulators placed before and after the cell. The permeate and the retentate solutions are recycled in the tray (closed circuit). The membrane used is spiral organic negatively charged the surface equal to 0.37 m^2 (Nanomax50) and permeability of water is equal to $27.5 \ 10^{-6} \text{ m/s.bar}$.



Fig. 6 Evolution of abatement rate of the dye according to variation of dye concentration ($pH_{solution} = 6.5-8; j = 25.2; mA/cm^2, [NaCl] = 1 g/L;$ time of electrolysis = 1 h)

Effects of dye concentration, NaCl concentration and flow percolation have been studied to determine the optimal conditions for dye removal during the treatment of wastewater by electrocoagulation and nanofiltration process.

4. Results and discussion

In order to assess the role played by the initial concentration of the dye in the electrocoagulation treatment, we performed experiments (batch processing) keeping all other parameters constant, namely the salt ([NaCl]_{added} = 1g/L, current density = 25.2 mA/cm² and initial pH of the solutions. we varied concentration of the dyes from 3.7 to 13.7 mg/L.

The results are expressed as the abatement of the colour given by the final concentration ratio relative to the initial dye concentration (Fig. 6).

The best discoloration (37.4%) was observed in dye concentration of 7.21 mg/L, increasing the dye concentration in excess of 7.21 mg/L decreased the rate of reduction is noted. This may be due to insufficient formation of complex metal hydroxide excess to coagulate dye molecules present at high concentrations (Mollah *et al.* 2004).

The effect of NaCl was studied on the efficiency of the process (Fig. 7).

The increase of the conductivity by the addition of sodium chloride is known to reduce the voltage between electrodes due to the decreased resistance of polluted water and to reduce the energy consumption. The choice of sodium chloride results from the fact that the chloride ions significantly reduce the adverse effects of other anions.

In the study by electrocoagulation in a continuous mode, to get closer to a real effluent we selected a concentration of 52.4 mg/l dye. we can see from Fig. 8 the results obtained for the evolution of abatement rate as function of flow percolation for an initial concentration of the solution of 52.4 mg/L, $[\text{NaCl}]_{\text{added}} = 3 \text{ g/L}$ and a current density of 2.13 mA/cm².



Fig. 7 Abatement rate of the dye according to variation of the NaCl concentration



Fig. 8 Evolution of the rate reduction of the dye according to the flow percolating $C_0 = 52.37$ mg/L; [NaCl]_{added} = 3 g/L; j = 2.13 mA/cm²; time of electrolysis = 1 h



Fig. 9 Permeate flux of RS dye solution versus transmembrane pressure using NF membrane (C_0 = 4.88 mg/L; pHs = 7.82, conductivity = 5 mS/cm, turbidity = 0.23 NTU)

We note that when the rate of percolation increases from 0.332 mL/s to 3.981 mL/s, the reduction rate decreased from 92% to 83%, which is due to the short contact time between the electrode and the electrolyte. The low flow allowed the coagulant generated by electrochemical oxidation of the anode and to the pollutant to be mixed which allows improving the coagulation rate (Salim *et al.* 2013).

At the end of this study, the supernatant resulting from the continuous process was taken that been treated by nanofiltration.

In order to study the variation of permeate flux as a function of transmembrane pressure using pure water (distilled water) and a colored solution of RS with initial concentration of 4.88 mg/L, pHs = 7.82, a conductivity 5 mS/cm and turbidity = 0.23 NTU (It's the supernatant recovered after electrocoagulation of a RS solution with initial concentration of 52.37 mg/L, $Q_M = 1.296$ mL/s and j = 2.13 mA/cm²) we have represented the following figure (Fig. 9). The equations of obtained plots are shown in the corresponding figure for pure water and studied dye solution.

Permeate flux increases from $2.06 \ 10^{-6}$ to $14.15 \ 10^{-6}$ m/s and $2.33.10^{-6}$ to $12.44 \ 10^{-6}$ m/s for RS and pure water, respectively (a linear increase of the permeate flux versus transmembrane pressure).

The permeate flow from the dye solution is below that of pure water, this phenomenon can be related, not only, to the concentration polarization but also to the presence of an important osmotic pressure near the membrane-effluent interface which leads to a decrease in the effective driving force (Petrini *et al.* 2007).

In fact, the retention of the ionic species results in an osmotic pressure build-up across the membrane. The osmotic pressure causes flux to decline, but this is due to reduced driving force rather than an increase in the resistance to mass transfer (Aouni *et al.* 2012).

Fig. 10 shows the retention rates for colour, conductivity and turbidity for dye solution ($C_0 = 4.88 \text{ mg/L}$; pHs = 7.82; conductivity = 5 mS/cm and turbidity = 0.23 NTU).

From Fig. 10, we find that:

- The retention rate of the maximum color (38.8%) and does not vary with increasing pressure.
- The retention rate of the conductivity increases from 6% to 24% with the increase in the transmembrane pressure of 1 to 4 bars.
- The retention rate of the turbidity decreases to 86.95% 43.47% with the increase in transmembrane pressure of 1 to 4 bars.



Fig. 10 Retention rates as a function of transmembrane pressure after red solophenyle solution treatment using NF membrane ($C_0 = 4.88 \text{ mg/L}$; pHs = 7.82; conductivity = 5 mS/cm; turbidity = 0.23 NTU)



Fig. 11 Change in colouring retention as a function of the change in concentration of the recycling solution ($C_0 = 52.4 \text{ mg/L}$)

These results can be explained by the fact that the negatively charged membrane increases the electrostatic repulsion of anionic compounds. The studied direct Red Solophényle 4GE dye is characterized by the sulfonate group which highly interact with the negative charge membrane. This phenomenon justifies the high colour retention and the improvement of chloride retention which affect the conductivity retention rate.

At the end of this study, the supernatant resulting from the continuous process was taken that been treated by nanofiltration.

Pressure applied was fixed to 3bar and the solution was allowed to be recycled during 30minute for each experiment and we recover the permeate and retentate (Fig. 11).

We find that increasing of the dye concentration from 3.86 to 7.5 mg/L leads to a higher retention (ranging from 22% to 60%), which can only be explained by the fact that the burden of the negative membrane Beyond its isoelectric point (pH = 4.5) and taking into account groups that constitute our dye gives a greater repulsion.

5. Conclusions

This work was aimed at the treatment of a red azo dye solophenyle 4GE by coupling two processes. A reduction of 37.4% was obtained by electrocoagulation in batch for a pH of 8 and a dye concentration of 7.21 mg/L, the concentration of added salt (NaCl) is 1g/L and density current is 25.16 mA/cm².

The coupling processes (electro-nanofiltration) has allowed us to reach a total discoloration (100%) of the solution (pH of the solution is approximately 7.6, $[NaCl]_{added} = 3 \text{ g/L}$; density applied current was 2.13 mA/cm²; and the initial dye concentration is 52.4 mg/L).

Overall, the study is promising to reduce the sludge generated after electrocoagulation and dye concentration for possible use. The photovoltaic significantly reduces energy consumption in the

expectation of modeling towards the membrane process to put together the whole system whose elements work within the framework of sustainable development (Taleb-Ahmed *et al.* 2012, Nadjib *et al.* 2012, Benhadji *et al.* 2011).

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