

# Treatment characteristics of textile wastewater and removal of heavy metals using the electroflotation technique

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## Abstract

The present work experimentally studied the clarification of wastewater using the electroflotation technique (EF) with aluminum electrodes. In the first part, we have studied the principal parameters (feed tension  $U$ , distance between electrodes  $D$ , period of treatment  $t$ , pH and conductivity  $\kappa$ ) affecting the process using a simulated model of wastewater. The application of the optimized parameters ( $U = 20$  V,  $D = 1$  cm,  $t = 20$  min) on an industrial wastewater derived from an Algerian velvet manufacture showed a high removal of biological oxygen demand after 5 days ( $BOD_5 = 93.5\%$ ), chemical oxygen demand ( $COD = 90.3\%$ ), turbidity (78.7%), suspended solids ( $SS = 93.3\%$ ) and color (>93%). In the second part, we have studied the separation of some heavy metal ions such as iron, nickel, copper, zinc, lead and cadmium. This allowed us to show that the kinetics of electroflotation is very quick (<15 min), and the removal rate reaches 99%.

**Keywords:** Electroflotation; Aluminum electrodes; Turbidity; Textile wastewater; Heavy metals

## 1. Introduction

The industry in Béjaïa (a coastal town in Algeria) is growing quickly and currently discharges about 80,000 m<sup>3</sup> of wastewater each day. Metal finishing industries are severely polluting the environment by their disposals of bath and

rinse wastewater [1]. These disposals unfortunately contain toxic species such as heavy metals whose increased concentrations in the human body may cause some significant health-related problems in the long run [2,3]. Wastewater from dyeing and finishing processes in the textile manufacturing industry constitute a substantial source of pollution which exhibits intense color, high chemical oxygen demand, fluctuating pH

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and suspended particles. Indeed, the textile industry utilizes about 10,000 pigments or dyes, but most of them are toxic substances to human and aquatic life [4] and it has been reported that up to 15% of the dyes used are released into wastewaters [5]. These must therefore be treated before final discharge to achieve legal and aesthetic standards. This is a particularly critical problem in Algeria where the textile industry and metal finishing plants are highly developed. Conventional methods for removing dyes from industrial wastewater consist mainly of biological and physicochemical treatments and their various combinations [6–10]. Biological treatments are cheaper than other methods, but dye toxicity usually inhibits bacterial growth and limits therefore the efficiency of the decolorization [10]. Physicochemical methods include adsorption (e.g. on active carbon), coagulation-flocculation (using inorganic salts or polymers), chemical oxidation (chlorination, ozonisation) and photodegradation (UV/H<sub>2</sub>O<sub>2</sub>, UV/TiO<sub>2</sub>) [11–22]. However, these technologies usually need additional chemicals which sometimes produce a secondary pollution and a huge volume of sludge [5,23,24]. Water treatments based on the electroflotation technique have been recently proved to circumvent most of these problems, while being also economically attractive [4,5,24–28].

Electroflotation (EF)–electrocoagulation (EC) is an electrochemical method for treating polluted water which has been successfully applied for treatment of soluble or colloidal pollutants, such as wastewater containing heavy metals, emulsions, suspensions, etc., but also drinking water for lead or fluoride removal [29–31]. In EF, coagulants are delivered *in situ* using the corrosion of sacrificial anodes when a DC voltage is applied. Simultaneously, electrolytic gases (typically H<sub>2</sub>) are generated at the cathode. Aluminum and iron materials are usually used as anodes, the dissolution of which produces hydroxides, oxyhydroxides and polymeric hydroxides. These

are usually more effective coagulants than those used in chemical dosing: they are able to destabilize colloidal suspensions and emulsions, to adsorb, neutralize or precipitate dissolved polluting species, and finally to form flocs that can be removed either by settling/filtration or flotation. In EF, settling is the most common option, while flotation may be achieved by H<sub>2</sub> or assisted by air injection [31]. A typical EF unit includes therefore an EF cell/reactor, a separator for settling or flotation, and often a filtration step. EF was seen as a promising technology in the 19th century, but had nearly disappeared by the 1930s. However, EF has been experiencing a renaissance in the 1990s [31]. Indeed, the benefits of EF include simplicity, efficiency, environmental compatibility, safety, selectivity, flexibility and cost effectiveness [24–34]. In particular, the main points involve the reduction of sludge generation [4], the minimization of the addition of chemicals and little space requirements due to shorter residence time [4,23–28], especially when EF is compared to biological treatments.

Although EF may be as cost-effective as chemical dosing [24,35], its main deficiency is the lack of dominant reactor design and modeling procedures. Mollah et al. [30] described six typical configurations for industrial EF cells and report their respective advantages and drawbacks. However, the literature reveals any systematic approach for these configurations for design and scale-up purpose. This situation stems mainly from the complex interactions between electrochemistry, colloidal forces and hydrodynamics that govern the behavior of EF reactors. While the recent literature focuses usually on the two first aspects (such as electrode design [29,30], electrode material [23–25]), the hydrodynamics of the three-phase gas–liquid–solid flow is still disregarded, the presence of gas being usually considered as an unnecessary complication [36]. This stems mainly from the fact that most papers use laboratory-scale EF cells in which magnetic

stirring is adjusted experimentally and the separation step by floatation/sedimentation is not studied.

In this study, experiments were conducted to examine the effects of the operating parameters, such as the applied voltage  $U$ , time of treatment  $t$ , pH, interelectrode distance  $D$  and conductivity  $\kappa$  on turbidity removal efficiency in the batch electroflotation process. The optimal operating conditions were determined and applied to a textile wastewater and separation of some heavy metal ions.

## 2. Materials and methods

The simulated wastewater was prepared with 1 L of drinking water and 500 mg/L of silica gel sieved to particle dimensions of 43  $\mu\text{m}$  to 500  $\mu\text{m}$  to determine the optimal of the operating process. The wastewater used in this research was taken from the effluent of an Algerian velvet manufacturer (AVM). A batch electroflotation reactor, illustrated in Fig. 1, with two aluminum electrodes (2.7 cm  $\times$  1.7 cm  $\times$  0.01 cm) installed in the middle of the reactor, was operated with 1 L sample volumes. The electrodes were connected to an external DC power supply providing 0 to 30 V. Samples were taken from the effluent port located 0.5 cm above the bottom of the reactor. The period of treatment in this experiment was fixed at 20 min, 17 min of contact time (the time during which current was applied), and 3 min of separation time (the separation time of flocs after the current was interrupted).

The measurements of chemical oxygen demand (COD), (biological oxygen demand after 5 days (BOD<sub>5</sub>), suspended solids (SS) and color followed the procedure of a standard method [37]. For the analysis of particle distribution, a Retsch apparatus was utilized. Turbidity was determined by a spectrophotometer (Shimadzu UVPC1601) and pH was measured by using EC30 pH meter. AAS (Atomic Absorption Spec-

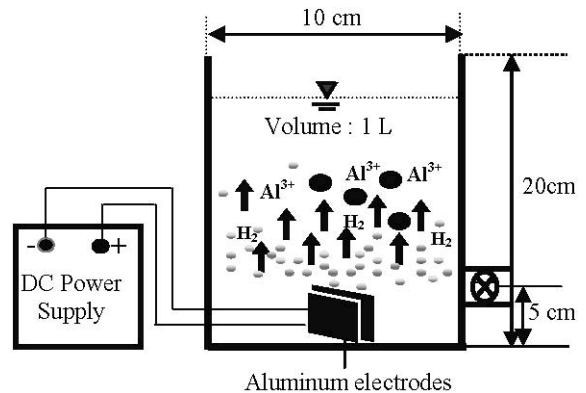


Fig. 1. Batch electroflotation reactor.

trometer, Shimadzu, Japan) was used to determine heavy metals.

## 3. Results and discussions

When current passes through an electrochemical reactor, it must overcome the equilibrium potential difference, anode overpotential, cathode overpotential and ohmic potential drop of the solution [38]. When aluminum is used as an electrode material, there are three major reactions in the electrochemical reactor as follows [39]:

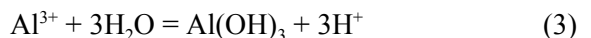
- oxidation reaction at the anode:



- reduction reaction at the cathode:



- hydrolysis reaction:



### 3.1. Effect of applied voltages on the efficiency of turbidity removal

To find the optimal electroflotation operating voltage, the influences of applied voltage on the

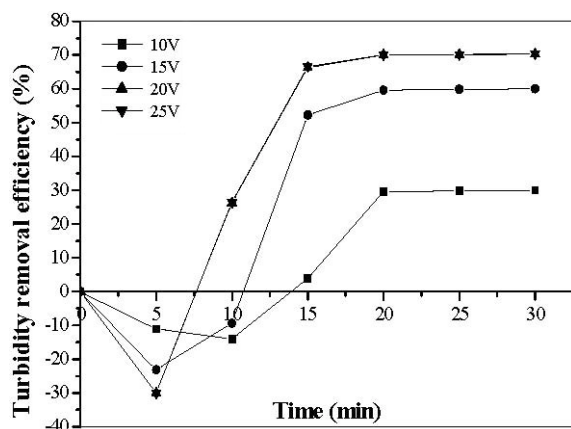


Fig. 2. Effect of applied voltage  $U$  on the turbidity removal efficiency:  $C_0 = 500$  mg/L, interelectrode distance  $d = 1$  cm, initial pH = 7.3, conductivity  $\kappa = 1.6$  mS/cm.

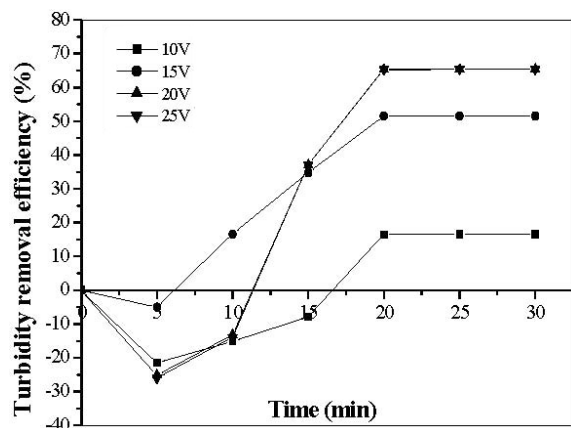


Fig. 3. Effect of applied voltage  $U$  on the turbidity removal efficiency:  $C_0 = 500$  mg/L, interelectrode distance  $D = 2$  cm, initial pH = 7.3, conductivity  $\kappa = 1.6$  mS/cm.

turbidity removal efficiency of simulated wastewater are represented in Figs. 2–4.

During the batch electroflotation experiments, the magnitude of the applied voltage (10, 15, 20 and 25 V), and the distance between electrodes (1, 2 and 3 cm) affected considerably the removal of turbidity, as shown in Figs. 2–4. The turbidity removal efficiency increased linearly with the

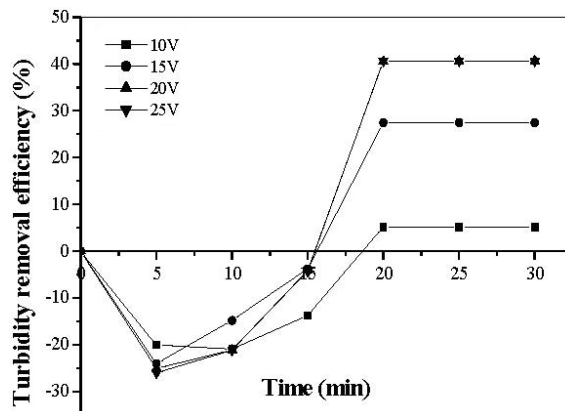


Fig. 4. Effect of applied voltage  $U$  on the turbidity removal efficiency:  $C_0 = 500$  mg/L, interelectrode distance  $D = 3$  cm, initial pH = 7.3, conductivity  $\kappa = 1.6$  mS/cm.

applied voltage; 70.8% turbidity removal efficiency was recorded at 20 V in a 20-min run time. However, a small decrease of current scarcely affected the rate of increase in removal efficiency. In Figs. 2–4, the negative removal efficiency of the turbidity at low times can be explained: during the flow of the electrical current, additional convection occurs. According to EDELIN [40,41], dissolved aluminum is polymerized in the shape of aluminium hydroxide and thus increases turbidity. Thus, the following investigation of removal efficiency for different items was accomplished only at the experimentally selected optimum parameters: applied voltage 20 V, electrode distance 1 cm, and period of treatment 20 min.

### 3.2. Effect of initial pH on the efficiency of turbidity removal

It has been established that the influence pH is an important parameter influencing the performance of the electrochemical process [42,43]. To examine its effect, the sample was adjusted to a desired pH for each experiment by using sodium hydroxide or sulfuric acid. Fig. 5 demonstrates

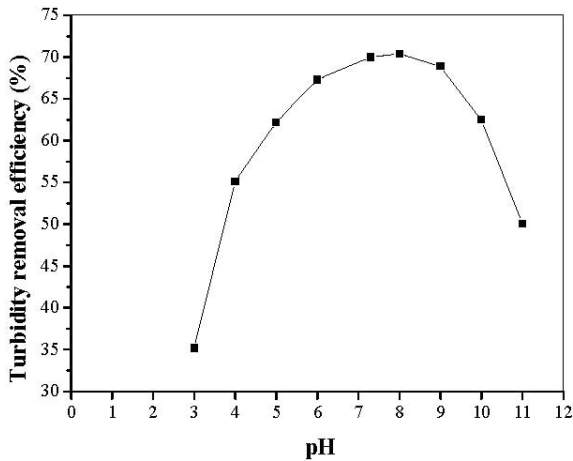


Fig. 5. Effect of initial pH on the turbidity removal efficiency:  $C_0 = 500$  mg/L, feed tension  $U = 20$  V, inter-electrode distance  $D = 1$  cm, retention time  $t = 20$  min, conductivity  $\kappa = 1.6$  mS/cm.

the removal efficiencies of turbidity as a function of the influent pH. The maximum removals of turbidity were observed at pH around 8. However, the pH changed during batch EF, as already mentioned in the above-mentioned papers. Its evolution depended on the initial pH. EF process exhibits some buffering capacity because of the balance between the production and the consumption of  $\text{OH}^-$  [33], which prevents a high change in pH (Fig. 6). The buffering pH seems just above 7: when the initial pH is above this value, pH decreases during EF; otherwise, the opposite behavior is observed.

The effect of pH can be explained as follows. The main reactions during EF are:

- Anode:



- Cathode:



At low pH, such as 2–3, cationic monomeric species  $\text{Al}^{3+}$  and  $\text{Al}(\text{OH})_2^+$  predominate. When pH

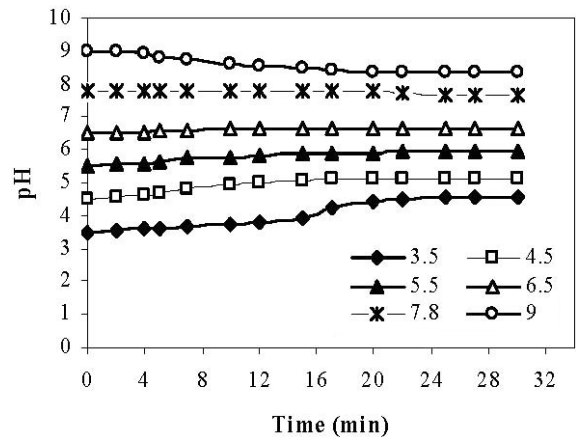
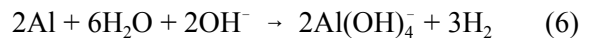


Fig. 6. Evolution of pH values during EF for different values of initial pH:  $C_0 = 500$  mg/L, feed tension  $U = 20$  V, inter-electrode distance  $D = 1$  cm, conductivity  $\kappa = 1.6$  mS/cm.

is between 4–9, the  $\text{Al}^{3+}$  and  $\text{OH}^-$  ions generated by the electrodes react to form various monomeric species such as  $\text{Al}(\text{OH})_2^+$ ,  $\text{Al}(\text{OH})_2^{2+}$ , and polymeric species such as  $\text{Al}_6(\text{OH})_{15}^{3+}$ ,  $\text{Al}_7(\text{OH})_{17}^{4+}$ , and  $\text{Al}_{13}(\text{OH})_{34}^{5+}$  that finally transform into insoluble amorphous  $\text{Al}(\text{OH})_{3(\text{s})}$  through complex polymerization/precipitation kinetics [26]. When pH is higher than 10, the monomeric  $\text{Al}(\text{OH})_4^-$  anion concentration increases at the expense of  $\text{Al}(\text{OH})_{3(\text{s})}$ . In addition, the cathode may be chemically attacked by  $\text{OH}^-$  ions generated together with  $\text{H}_2$  at high pH values [4]:



Two main mechanisms are generally considered: *precipitation* for pH lower than 4 and *adsorption* for higher pH. Adsorption may proceed on  $\text{Al}(\text{OH})_3$  or on the monomeric  $\text{Al}(\text{OH})_4^-$  anion depending on the pollutant chemical structure. The formation of  $\text{Al}(\text{OH})_{3(\text{s})}$  is therefore optimal in the 4–9 pH range, which corresponds to the optimum pH values investigated in this work.

However, pH affects also bubble size [44]. Typical bubble sizes in EF always fall in the

range of 20–70  $\mu\text{m}$  [45], far smaller than those observed in conventional air-assisted flotation, which provides both sufficient surface area for gas–liquid–solid interfaces and mixing efficiency to favour the aggregation of tiny destabilized particles. Hydrogen bubbles, which obey usually to a lognormal size distribution, are known to be the smallest about neutral pH [46].

As a conclusion, pH may be adjusted in the optimum range in order to achieve a compromise between best coagulation and best flotation. The optimum range may however vary as a function of electrode material and silica gel structure. In the following sections, initial pH will be fixed at about 7.6 to maximize turbidity removal efficiency.

### 3.3. Effect of conductivity on the efficiency of turbidity removal

The increase of the conductivity by the addition of sodium chloride is known to reduce the cell voltage  $U$  at constant current density due to the decrease of the ohmic resistance of wastewater [5,26,28]. Energy consumption, which is proportional to  $UI$ , will therefore decrease. Chloride ions could significantly reduce the adverse effects of other anions, such as  $\text{HCO}_3^-$  and  $\text{SO}_4^{2-}$ , for instance by avoiding the precipitation of calcium carbonate in hard water that could form an insulating layer on the surface of the electrodes and increase the ohmic resistance of the electrochemical cell [5,47]. Chloride anions can also be oxidized and give active chlorine forms, such as hypochlorite anions, that can oxidize dyes.

The conductivity of the simulated wastewater was adjusted to the desired levels by adding an appropriate amount of 1 N NaCl solution. The experimental conditions were: initial pH = 7.6, applied voltage  $U = 20$  V, and retention time  $t = 20$  min. The effect of conductivity on performance of the EF process is shown in Fig. 7.

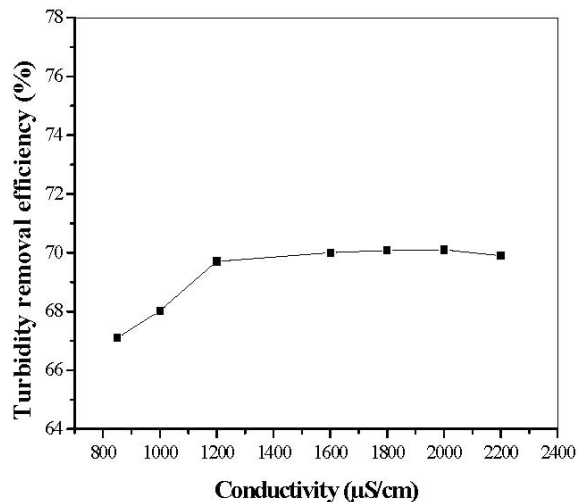


Fig. 7. Effect of conductivity on the turbidity removal efficiency:  $C_0 = 500$  mg/L, feed tension  $U = 20$  V, initial pH = 7.6, interelectrode distance  $D = 1$  cm, retention time  $t = 20$  min.

In Fig. 7, turbidity removal efficiency remains almost unchanged between the conductivity range of 1200 and 2200  $\mu\text{S/cm}$ .

### 3.4. Case study: Algerian velvet manufacture wastewater

The textile wastewater used in this research was obtained from a textile industrial complex located in Béjaia, Algeria. The initial characteristics of this textile wastewater are shown in Table 1.

Under the optimal operating conditions (feed tension  $U = 20$  V, retention time  $t = 20$  min, interelectrode distance  $D = 1$  cm, initial pH = 7.98, conductivity  $\kappa = 3.12$  mS/cm), the final characteristics of the wastewater used after treatment by EF are shown in Fig. 8.

Fig. 8 shows the removal efficiency for various items under an applied voltage of 20 V, 1 cm distance electrodes and 20 min run time, which turned out to be effective values found in Fig. 2. It showed that most of the SS that

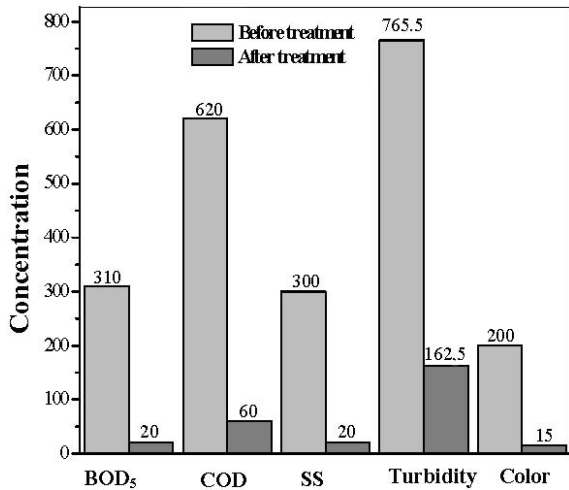


Fig. 8. Removal efficiency for various items: feed tension  $U = 20$  V, retention time  $t = 20$  min, interelectrode distance  $D = 1$  cm, initial pH = 7.98, conductivity  $\kappa = 3.12$  mS/cm.

Table 1  
Characteristics of textile wastewater used

Characteristics	Value
pH	8.7
BOD <sub>5</sub> (mg O <sub>2</sub> /L)	310
COD (mg O <sub>2</sub> /L)	620
SS (mg/L)	300
Turbidity (NTU)	765.5
Color (Co-Pt)	>200
Conductivity $\kappa$ (mS/cm)	3120

generated turbidity in AVM wastewater were removed by 93.3%, forming a floating scum layer. Moreover, the removal efficiencies of COD and BOD<sub>5</sub> showed values of 90.3% and 93.5%, respectively. This remarkable reduction of COD and BOD<sub>5</sub> can be explained by the fact that electroflotation has some effects on non-biodegradable organics in AVM wastewater. The great reduction in the color (>93%) of the sample shows that the electroflotation is a technique of choice for the clarification of wastewater.

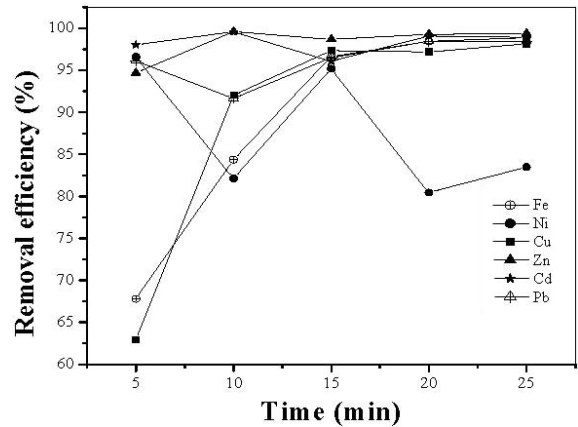


Fig. 9. Removal efficiency of various metals — presence of sulfates:  $C_0 = 100$  mg/L, feed tension  $U = 20$  V, interelectrode distance  $D = 1$  cm, initial pH = 8, conductivity  $\kappa = 2.7$  mS/cm.

### 3.5. Separation of heavy metals

#### 3.5.1. In the presence of sulfates

A model solution of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ,  $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ ,  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ ,  $\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$ ,  $\text{PbSO}_4$  and  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  was used for conducting the experiments. All solutions were prepared using distilled water with the following characteristics:  $C_0 = 100$  mg/L, initial pH = 8, and conductivity  $\kappa = 2.7$  mS/cm. The experimental results are presented in Fig. 9.

According to the results obtained, we note that the EF process functions for all studied metals. Comparing the results obtained in this study with similar ones [48–50], it can be observed that the EF process has an average removal efficiency of 93%, the kinetics of electroflotation is fast and does not exceed 15 min (effectiveness = 95–99%), except for nickel.

It should be noted that the pH drops: this is due to a neutralization of the  $\text{Al}^{3+}$  by  $\text{OH}^-$  [51].

#### 3.5.2. In the presence of nitrates

A model solution of  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  was used for conducting the

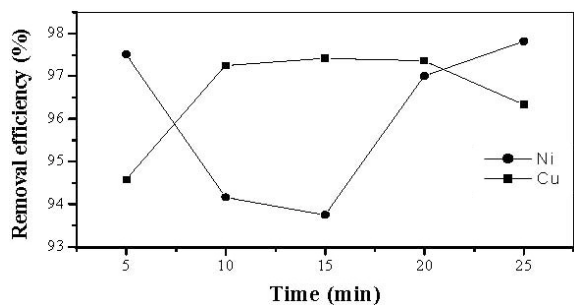


Fig. 10. Removal efficiency of various metals — presence of nitrates:  $C_0 = 100$  mg/L, feed tension  $U = 20$  V, interelectrode distance  $D = 1$  cm, initial pH = 8, conductivity  $\kappa = 2.7$  mS/cm.

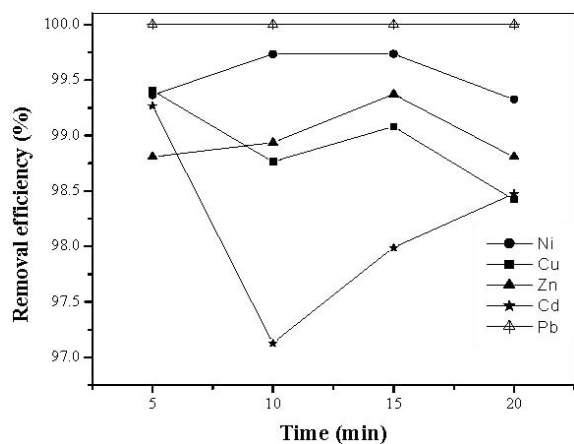


Fig. 11. Removal efficiency of various metals — polymetallic solutions:  $C_0 = 100$  mg/L, feed tension  $U = 20$  V, interelectrode distance  $D = 1$  cm, initial pH = 8, conductivity  $\kappa = 2.7$  mS/cm.

experiments. The concentration of each model solution was 100 mg/L, initial pH was 8 and conductivity was 2.7 mS/cm. The experimental results are presented in Fig. 10.

Fig. 10 shows that the kinetics of EF process is fast. We also note that the effectiveness of treatment of  $\text{Ni}(\text{NO}_3)_2$  is 97.5% after 5 min, then it decreases to around 94%, to take again its initial value at 25 min.

### 3.5.3. Kinetics of electroflotation of poly-metallic solutions

For the polymetallic solutions, we prepared a solution containing the ions of metals — Pb, Zn, Ni, Cu and Cd — in the presence of sulfates. The concentration of each model solution was 100 mg/L, initial pH was 8 and conductivity was 2.7 mS/cm. The experimental results are presented in Fig. 11.

Fig. 11 shows that the efficiency of treatment of the polymetallic solutions containing sulfate is faster than the monometallic solutions (Figs. 9 and 10). It should be noted that the efficiency of treatment is about 99% for all metals. For  $\text{PbSO}_4$ , we did not record any remaining traces.

## 4. Conclusions

The results obtained in this study suggest an opportunity for the application of electroflotation technology. It can be concluded that the treatment of wastewater by electroflotation is effective based on the following:

1. Based on the characteristics of particles in simulated wastewater ( $\phi_{50} < 25 \mu\text{m}$ ), electroflotation is applicable to the treatment of industrial wastewater.

2. In the absence of additional coagulants and electrolytes, the applied voltage had an effect on the efficiency of turbidity removal.

3. At an applied voltage of 20 V, 1 cm electrode distance and 20 min of run time, removal efficiencies for various items were as follows:  $\text{BOD}_5$  (93.5%), COD (90.3%), turbidity (78.7%), SS (93.3%) and color (>93%).

4. For a concentration of 100 mg/L and initial pH of 8, the EF process functions for all studied metals.

5. We were able to show that the kinetics of EF process is very quick (<15 min), and the removal rate reaches 99%.

6. Anions  $\text{SO}_4^-$  and  $\text{NO}_3^-$  contained in wastewater influence neither the kinetics nor the effectiveness of purification of different metal solutions.

7. The efficiency of treatment of the poly-metallic solutions containing sulfates is faster than the monometallic solutions.

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