Study on Ethylenediamine Removal from Textile Industry Wastewater

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Treating of the waste water from textile industry is an ongoing concern. The main problem is represented by dyes degradation leading to higher degrees of turbidity. Getting the textile by the new process in the recent years, change problems of wastewater treatment to eliminate compounds with toxicity and high concentrations. High consumption of water is reflected in the production of large quantities of wastewater. The present study proposes to remove ethylenediamine (EDA) from this type of water. Together with EDA has studied too, the possibility of reducing suspended solids (SS) and chemical oxygen demand(COD).The proposed process consist of the treat wastewater with an electrolyte solution (concentrations between 25 and 35 % by mass) and to addition of polyaluminiumsulphate (PAS). This method is applicable for removal EDA, COD and suspended solids in industrial practice.

Keywords: ethylenediamine, chemical oxygen demand, suspended solids, polyaluminium sulfate, textile wastewater industry

The existence of limited water sources and implicit water reuse, according to the best practices of the European Union regulations, require to finding new treatment solutions [1]. Using large amounts of water in the textile industry imposed decreased of cotton production which led to decrease of wastewater flow from about 140 L wastewater / kg cotton waste water to 30 L/ kg cotton [2-4]. But, this solution represent only a partial measure to reduce environmental impact. Unfortunately to the amount of water used for wash was added more water used in other manufacturing processes of the cotton compounds [5, 6]. But the replace of classical dye with non-sulfur compounds or contain a low sulfur, and use reducing agents, led to a decrease in the sulfur content in wastewater [7, 8]. But they remained the other components that make when was used the traditional treatment methods of the wastewater treatment, the treatment degree goes to low levels. Another feature that influence the quality of treated water is turbidity [9, 10]. The content of azo dyes in the wastewater will increase the turbidity over the permissible values.

One of the most important pollutants very difficult to remove from these waters is EDA. Water binary-EDA form azeotrope with maximum temperature of boiling.

For separating water from this mixture , is used fractionation process with a 6 bar pressure. At this pressure azeotrope is disappearing and EDA is separating 99 percent. This process is difficult to realize because of the other existent compounds in the waste waters and also because of the high cost investment.

As well, in this work is studied the possibility of eliminating EDA from the waste water through the application of well-known phenomenon *salt-effect*. Is actually the method by which is reduce the EDA solubility in the water through the introduction of electrolytes. The main idea comes from using electrolytes by changing the distribution coefficient of phenol between benzene and water. Using this process based on a previous embodiment led to results which simplified treatment process and also reduced the toxicity of waste water.

Treating of industrial wastewater with a high content of EDA maximum of 154 mg /L in two consecutive steps was the basis of this study.

Experimental part

The regents used in this study are Sigma Aldrich with advanced purity.

The waste water sample were collected from textile industry, from waste water treatment influent.

The experimental study consisted two phases:

-treating the industrial wastewater with an electrolyte solution

-wastewater treatment - electrolyte mixture with polialuminiumsulphate (PAS)

Preparation of the electrolyte NaOH + NaCl was made such a manner that respect the proportions shown in a previous study [11] .The amount of electrolyte added was 25% mass, 30% mass and 35% mass. It should be noted that sodium hydroxide in water is used in the textile industry because it is from the synthesis of artificial textile fibers. As such, initially was analyzed the amount of NaOH and then was completed to the required quantity of electrolyte in accordance with the proposed experimental plan.

The experiments were made in a glass reactor, thermostated with recirculation. The working temperature was 70°C. The vapor phase and also liquid phase were collected separately. The wastewater quality parameters analyzed and methods of analysis are presented in table 1.

Table 1
WASTEWATER INDICATORS AND ANALYSIS METHODS USED IN EXPERIMENTAL STUDY

Parameters	Analysis methods
EDA	UV detection
SS [12]	SR EN 872:2005
COD [13]	SR EN 6060:1996

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The concentration of the electrolyte, % mass	The amount of PAS, mg
25	0.1
	0.2
	0.3
	0.4
	0.5
30	0.1
	0.2
	0.3
	0.4
	0.5
35	0.1
	0.2
	0.3
	0.4
	0.5

Table 2
PAS AND ELECTROLITE QUANTITY USED IN THE EXPERIMENTAL STUDY

Method of analysis for EDA is based on UV detection at 254 Nm wavelength. The device used was SPECORD® PLUS. The double beam with a real device measures the sample and reference simultaneously.

Method for the determination of suspended solids in water is based on filtering under pressure or under vacuum.

The standard for determining the content of COD is based on determining the mass concentration of oxygen who is equivalent with the amount of potassium dichromate consumed by dissolved materials in suspension.

It should be noted that after the addition of electrolyte and PAS , the *p*H was kept within the limits of 7.0-7.5 . Because of this variation it's never been presented in this paper.

Due to the large amount of suspended solids contained in wastewater after treating wastewater with the electrolyte solution and liquid phase separation it was necessary to add reactive polymer, namely Polyaluminum Sulfate (PAS) (chemical formula:$[Al_2(OH)n(SO_4)_3\text{-}n/2]m$ (m ≤10, 1≤ n≤16), solution 10%).

The addition of this reagent has led to lower value of the *p*H (7.0), SS and COD .Reagent test was meant cancellation of electrostatic charges created and generating bridges that led to appear the gelatinous nature of agglomerate particle.

This particles were separated by filtering from wastewater. The optimal dose of the reagent required was determined using the Jar –test method [14].Important in the experiments was to mix the added reagent. It should be made within five minutes, at stirring speed of 300 rev / min .The quantities of reagents used in the experimental study are shown in table 2.

The removal degree of EDA, COD and suspended solids from wastewater is shown in equation 1 [15]:

$$ED = \frac{c_i - c_f}{c_i} * 100 \quad (1)$$

where:

ED represent the removal degree for EDA, COD and SS;
c_i – initial concentration of pollutant, mg/L;
c_f – final concentration of pollutant, mg/L.

Results and discussions

Quality indicators of waste water from textile industry, used in the experimental study are presented in table 3.

Analyzing the results obtained is observed that EDA has a concentration well above to the treatment plant (154 mg/L) , COD has values higher than 100 mg/L and the amount of suspended solids is too large for applying a conventional treatment process.

Treating the industrial wastewater by 25, 30 and 35 % electrolyte and different amounts of PAS between 0.1 mg and 0.5 mg, decreased the concentration EDA, SS and COD (figs. 1-3).

QUALITY INDICATORS	Value
pH, pH units	8.2
EDA, mg/L	154
SS, mg/L	532
COD, mg/L	1846

Table 3
QUALITY INDICATORS OF WASTE WATER FROM TEXTILE INDUSTRY

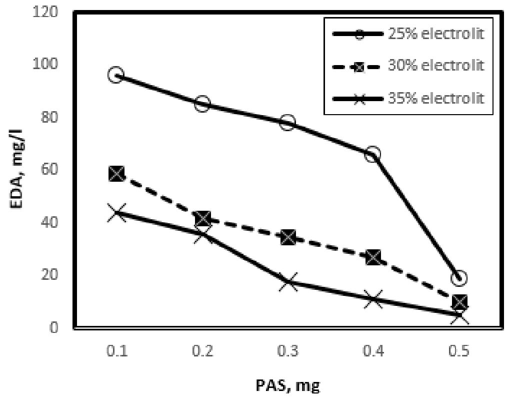

Fig.1. EDA concentration from wastewater after treatment with different amount of electrolyte and PAS

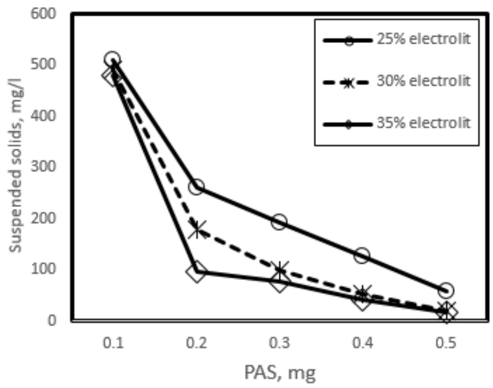

Fig.2. SS concentration from wastewater after treatment with different amount of electrolyte and PAS

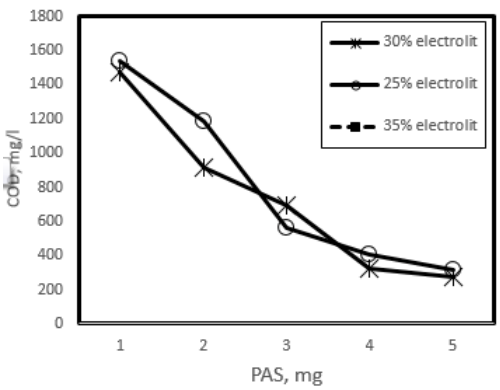

Fig.3. COD concentration from wastewater after treatment with different amount of electrolyte and PAS

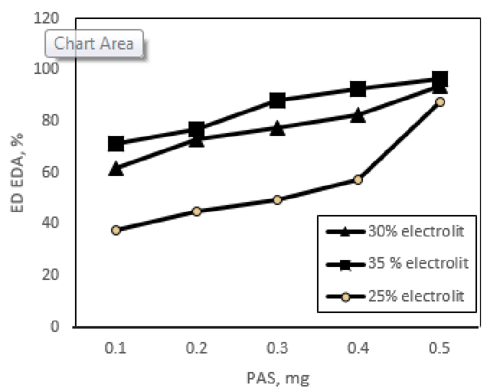

Fig. 4. Degree removal of EDA

From figure 1 it is observed that adding an amount of 25 % mass led to the decrease of electrolyte concentration EDA in wastewater to the value of 96 mg/L. The subsequent treatment with PAS to EUR 0.5 mg/L decrease EDA concentration in the wastewater to the amount of 19

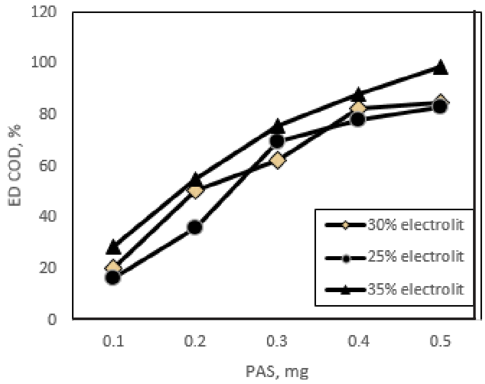

Fig. 5. Degree removal of COD

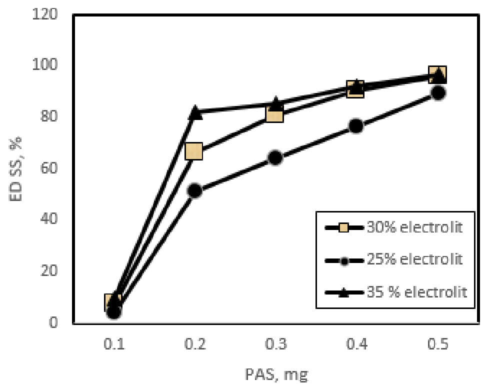

Fig. 6. Degree removal of SS

mg/L. A higher concentration of electrolyte initially added, namely 30% by mass removes a greater amount of EDA in the wastewater, the value obtained being 59 mg/L. The dosage of more than 0.5 mg PAS, namely decreases the EDA concentration up to the 10 mg/L. This is explained by training of EDA in flocks precipitated of adding the reagent.

The concentration of suspended solids present in the wastewater does not change significantly after adding the electrolyte. However, in the wastewater treatment with PAS, there is a sharp decrease in their concentration up to 58 mg/l (for wastewater by 25 % electrolyte) and up to 18 mg/L (for wastewater by 35 % electrolyte) (fig. 2).

Eliminating the wastewater COD is made significantly by adding PAS. The waste water having a concentration of 25% electrolyte with an amount of 0.2 mg PAS , COD decrease to the value of 1183 mg/L. Increasing the amount of PAS to a maximum of 0.5 mg will have good results up to the value of 312 mg / L. PAS treatment of wastewater containing 35% electrolyte will led to the obtaining a minimum COD of 25 mg / L to 0.5 mg PAS added .

Degree removal (ED) for EDA, COD and SS from wastewater are represented in figures 4, 5 and 6.

The most efficient method of removal of EDA in the wastewater is that in which is added in a 35 % mass electrolyte and 0.5 mg PAS. In this case, the degree of removal of EDA is 96.75 %. The similar results for this value (93.5 %) are obtained in the case of treatment with an electrolyte solution of 30 % and 0.5 mg PAS.

SS can be removed from wastewater from the textile industry in the proportion of 96.61 % by adding 35 mass% and 0.5 mg PAS electrolyte.

Elimination of COD from wastewater was done in a rate of 98.64 % for an treatment with electrolyte - 35% mass and 0.5 mg PAS .

Based on data from experimental study it was studying the possibility of estimating effects of concentrations of EDA , suspended solids and COD when adding quantities of electrolyte between 25 and 35 % mass and some known reactive type PAS.

Concentration removed from wastewater	Equation		
	25% electrolite	30% electrolite	30% electrolite
EDA	$y = -1.2917x^4 + 16.417x^3 - 71.208x^2 + 110.08x - 10$	$y = 0.125x^4 - 3.0833x^3 + 20.375x^2 - 58.417x + 100$	$y = -0.875x^4 + 7.25x^3 - 19.625x^2 + 10.25x + 99$
COD	$y = -1.3549x^4 + 19.189x^3 - 98.457x^2 + 228.56x - 143.98$	$y = -1.4881x^4 + 21.241x^3 - 112.14x^2 + 268.7x - 168.8$	$y = -3.2503x^4 + 44.565x^3 - 220.62x^2 + 471.04x - 282.14$
SS	$y = 2.5822x^4 - 32.458x^3 + 137.37x^2 - 204.19x + 113.16$	$y = -2.1849x^4 + 26.246x^3 - 111.9x^2 + 214.76x - 106.5$	$y = 0.4943x^4 - 5.6203x^3 + 18.818x^2 + 1.6928x + 13.326$

Table 4
EQUATION OBTAIN FROM REGRESSION ANALYSIS BY EXPERIMENTAL DATA

Selection of the independent variables and their simultaneous action are presented in table 4.

After the regression analysis performed corresponding equations were established EDA recovery, COD and suspended solids from wastewater (table 5). In equations obtained was noted by x the amount of PAS and y the amount of EDA, COD or SS removed from wastewater.

Conclusions

The remove pollutants from wastewater alkyleneamines type from textile industry is a problem that requires finding solutions to eliminate simultaneous EDA, COD and materials in suspension. The objective of the proposed study was achieved by using the electrolyte like solution NaOH and NaCl, in order to decrease the solubility of EDA in wastewater and its recovery by adding PAS. Finding an optimal physico-chemical treatment process involved testing several working versions namely electrolyte was added at a temperature of 70 ° C in concentrations ranging from 25 mass% to 35% mass. The solutions thus obtained were added different amounts of PAS between 0.1 and 0.5 mg.

Eliminating advanced EDA to the 10 mg/L, COD up to the value of 25 mg/L and SS to the value of 18 mg/L was obtained for a 35% working version of electrolyte and 0.5 mg PAS. The elimination grades were: 96.75 % EDA, 98.64 % and 96.61 % COD SS.

The paper established relationship by regression analysis of experimental data, allowing estimation of the EDA , COD and SS removed. It is necessary to achieve a design calculation of future industrial installation of physico-chemical treatment.

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