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Formed Sludge in Tannery Wastewater: Modelling by the Experimental Design

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Abstract- This article is devoted to the experimental procedure followed for the treatment of industrial discharges of tannery by coagulation flocculation. These are industrial discharges from the tannery of the city of Mohammedia.

A screening study will be presented for the analysis of the volume of sludge formed, in order to identify the most influential factors and to minimize the number of experiments. The statistical characteristics and graphical methods will be presented.

Factors such as pH, the nature of the coagulating agent, the concentration of the coagulant, the concentration of the flocculating agent, the ash concentration and the type of flocculant are taken into account in determining their effects on the volume of Decanted sludge.

The results of the design of the mathematical model which has been established by the experimental design method are compatible with the experimental results.

Keywords- *Experimental Design, Screening, Sludge, Tannery Wastewater, Coagulation Flocculation, Tannery Wastewater, Sludges Production*

I. INTRODUCTION

The conventional physicochemical treatment of tannery effluents consists of pretreatment, coagulation, flocculation, sedimentation and treatment of sludge.

The treatment of these discharges by coagulation flocculation [1, 2] has shown that the pollution elimination efficiency and the volume of sludge generated depend on the characteristics of the raw effluent, the nature and the concentration of the coagulant and the Flocculant used.

In this study, two coagulants and seven flocculants were tested. We propose to carry out a screening study in order to highlight factors influencing the coagulation process such as pH, nature and concentration of coagulant, the nature and concentration of the flocculant and the addition or not of the ashes as absorbents. To minimize the time, cost and means used to carry out the experiments, the experimental design method is adopted.

The study of sludge formation during coagulation flocculation treatment using ferric chloride $FeCl_3$ showed that the amount of sludge produced increases with the concentration of coagulant and reaches its maximum at the optimal dose of coagulant. Beyond this dose, the amount of sludge formed does not undergo great variations due to the fact that the generation of sludge is linked to the elimination of pollution, which at this stage reaches its maximum and remains almost unchanged.

Aluminum sulfate has also been tested for the treatment of tannery wastewater. The jar tests were conducted at optimum pH. Processing performance was evaluated by quantifying the settled sludge.

The amount of sludge formed during treatment with $Al_2(SO_4)_3$ increased with the concentration of coagulant. Beyond the optimum coagulant dose, the amount of sludge increases in the same way, while the pollution elimination efficiency is not improved. It is therefore essential to optimize the dose of coagulant in order to obtain a better yield of pollution elimination while generating less sludge.

II. MATERIALS AND MRETHODS

A. Sampling process

The wastewater comes from an industrial tannery unit located in the city of Mohammedia [3]. The sampling is carried out at the level of the effluents of each stage of production and at the level of main collector where all the discharges of the plant lead.

Two types of samples were taken: medium and spot samples. The average samples were obtained by manual mixing of samples taken at the end of each hour throughout a day. We wanted to ensure that the average samples represent the greatest possible diversity depending on the variability of the production program. Several sampling campaigns have been carried out.

1) Reagents

As coagulants, ferric chloride $FeCl_3$ and aluminum sulfate $Al_2(SO_4)_3$, being the most widely used reagents for the treatment of waste water by coagulation flocculation, were selected for this study.

To improve the elimination efficiency of the flocculation coagulation pollution, flocculants are often used. The latter are organic or mineral, cationic, anionic or nonionic polymers and may be natural or synthetic. Seven flocculants of different natures were used in this study. These products have been provided free of charge by companies specializing in their production.

2) Analysis method

During the flocculation coagulation process and after the fast and slow agitation phases, the contents of the beakers of the Jar test apparatus are transferred into Imhoff cones. After a predetermined decantation time, the volume of sludge generated by the treatment of one liter of the wastewater is read directly on the graduated cones [4].

B. Experimental process

The flocculation coagulation tests were carried out under controlled laboratory conditions using a Jar test flocculator. For each test, four beakers of one liter each were used to examine the effect of the concentration of coagulant, flocculant, or coagulation pH. Before each test, the wastewater is mixed well and appropriate volumes are transferred to the corresponding beakers.

For tests involving the addition of coagulant and flocculant, after the addition of coagulant, a known amount of flocculant is added and rapid stirring is continued for a further one minute. After rapid stirring, the mixture was slowly stirred for 20 min at 30 rpm. The coagulated wastewater is then transferred to Imhoff cones and allowed to settle for one hour.

C. Design of experiment method [5, 6, 7]

The response observed in this study is the volume of sludge formed (ml/L)

The measurements of the volume of sludge produced during the experiments are shown in figure 1.

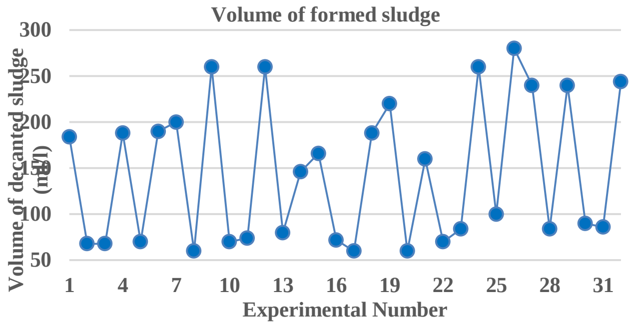

Figure 1. Plots of Volume of formed sludge versus experimental number

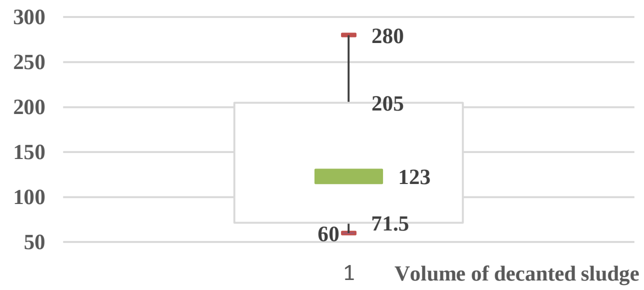

Figure 2. Boxplot of volume of formed sludge

The mustache box is a graphical representation which allows representing schematically the distribution of the answer called volume of sludge formed.

1) Factors definition

The factor definition stage, carried out with the help of the Laboratory team, identified 6 potentially influential factors on the volume of sludge formed, 5 factors at 2 levels and 1 factor at 7 levels:

TABLE I. DESIGN VARIABLES AND THEIR LEVELS

Factor	*Factor Name*	*Level 1*	*Level 2*	*Level 3*	*Level 4*	*Level 5*	*Level 6*	*Level 7*
X1	Coagulant	Ferricchloride $FeCl_3$	Aluminun sulfate $Al_2(SO_4)_3$					
X2	Coagulant concentration	250 mg/L	500 mg/L					
X3	Flocculantconcentration	0 mg/L	10 mg/L					
X4	Ashes concentration	0 mg/L	100 mg/L					
X5	pH	5	6					
X6	Flocculant	P3000	Polyacrylamide	Astral Flocculant	Chimec 2063	Chimec 5161	Chimec 5264	Alginate

- Polysep 3000 (P3000), Cationic Organic Polymer Vegetable Origin
- Chimec 2063, Polyamine in liquid form
- Chimec 5161, Anionic polyelectrolyte powder
- Chimec 5264, Cationic polyelectrolyte powder
- Superfloc A-1820 (SU), Anionic polyacrylamide
- Praestol 2515 TR, Copolymer of acrylamide and sodium acrylate
- Alginate, Alginate, natural polymer

2) Empirical model and its coefficients

In our case, we used an asymmetric screening matrix to study the weights of the main factors. We note that the model we are looking for is additive and has 12 unknowns to be determined. It is in the form:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_5X_5 + b_6X_6 + b_7X_7 + b_8X_8 + b_9X_9 + b_{10}F_5X_{10} + b_{11}X_{11}$$

Where Y is the response function (Sludge volume in NTU). X_1, X_2, X_3, X_4 and X_5 are the independent variables. b_0 is the average theoretical value of the response. b_1, b_2, b_3, b_4 and b_5 are the factor effects of X_1, X_2, X_3, X_4 and X_5 respectively.

The Effects table is :

TABLE II. CALCULATION OF THE ESTIMATION OF BI EFFECTS

Designation factor	*Value*
b0	219,19
b1	-22,62
b2	-19,37
b3	10,13
b4	0,88
b5	-139,37
b6	21
b7	17,5
b8	16,5
b9	14
b10	3,5
b11	-10

III. RESULTS AND DISCUSSION

A. Statistical analysis of the model

The Lenth method [8] consists in estimating a pseudo-standard error to implement a statistical test, the result of which is the plotting of the significance limits on the bar graph. Coefficients whose estimated values are outside the boundaries correspond to active effects. Coefficients whose estimated values are within the limits correspond to non-active effects. Between these two limits, it is necessary to have additional information to arrive at a conclusion and to pronounce on the average effects of the factors.

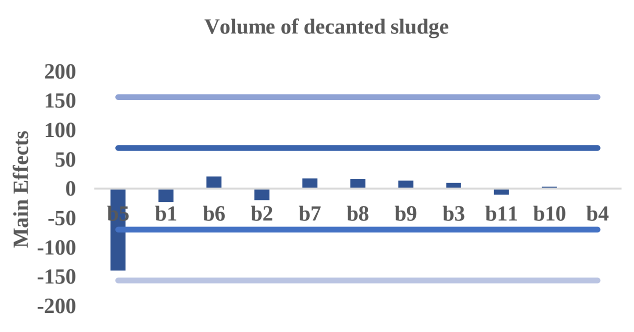

Figure 3. Significant effects identified by Lenth's method

This graph shows that no factor is active except pH. This does not mean that the results of mathematical analysis can not be exploited. It is preferable to analyze these results using another method such analysis of variance.

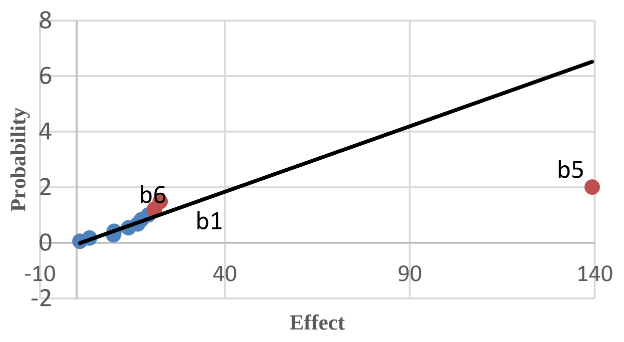

Figure 4. Half normal plot

The points aligned on the straight line passing through the origin represent the non-statistically influential variables. On the contrary, the positioning on the graph of the points associated with pH (coefficient b5), coagulant (coefficient b1) and flocuculant (coefficient b6) tends to show that these variables would be statistically significant on the overall variance of the model.

Polysep 3000 (P3000) is a natural organic flocculant based on acidified vegetable tannins and can replace the inorganic salts for the treatment of wastewater.

Flocculation coagulation tests were conducted to evaluate the effect of P3000 on the quantification of sludge generated during treatment. It has been shown that the volume of settled sludge increases almost linearly with the concentration of P3000.

B. Analysis of coefficients (Student test)

This analysis was carried out in order to know the parameters that are really significant on the response.

TABLE III. ANALYSE STATISTIQUE DES COEFFICIENTS

Coefficient		*VAR*	*Standard Error*	*t Stat*	*Significance P-Value %*
b0	219,19	332,300	18,229	12,024	0,000<0,01
b1	-22,62	102,259	10,112	-2,237	3,684<5
b2	-19,37	102,259	10,112	-1,915	6,984
b3	10,13	102,259	10,112	1,002	32,843
b4	0,88	102,259	10,112	0,087	93,152
b5	-139,37	102,259	10,112	-13,782	0,000<0,01
b6	21	306,776	17,515	1,199	24,455
b7	17,5	409,034	20,225	0,865	39,714
b8	16,5	409,034	20,225	0,816	42,420
b9	14	409,034	20,225	0,692	49,675
b10	3,5	409,034	20,225	0,173	86,435
b11	-10	409,034	20,225	-0,494	62,638

The factors whose coefficients are b_1 and b_5 appear to influence the volume of sludge generated. Their influential character is confirmed by a new analysis of variance, presented in Table 3.

C. Analysis of variance (Ficher-Snedecor test)

TABLE IV. ANOVA TABLE FOR THE LINEAR REGRESSION

Source of variation	*Sum of squares*	*Degrees of Freedom*	*Mean Squares*	*F Ratio*	*Significance F*
Regression (Model)	1.66917E+05	11	1.51742E+04	18.5488	< 0.01 %
Residual	0.63614E+04	20	8.18069E+02		
Total	1.83278E+05	31			

The statistical analysis [9, 10] of the model therefore leads to the analysis table of the following variance. It consists of comparing the experimental variances, the variances of the answers and the variances of the deviations. Table 4 mainly indicates that the model of the volume of sludge produced is well adjusted since the response has a very low probability F (<0.01%).

D. Graphical method

The effects of the factors on the volume of sludge formed can be represented graphically as mentioned in figure 5. The plot of the mean effects facilitates the restitution of the information. It is an indisputable asset of the methodological approach associated with the plans of experiments. This figure 5 clairly shows that pH factor is significant.

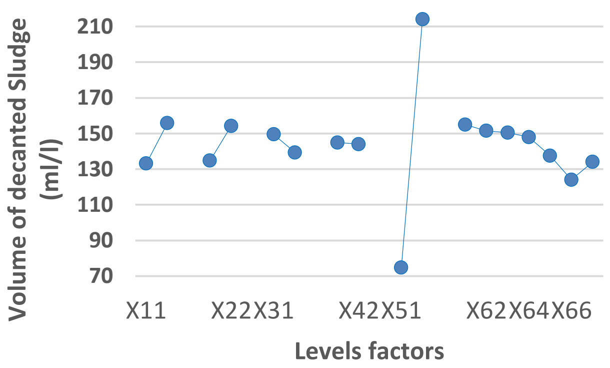

Figure 5. Classical diagram to represent factor effect

The Pareto diagram shows the influence of each of the six factors analyzed on the volume of sludge decanted. The most influential factor is the pH (coefficient b5) followed by the Coagulant (coefficient b1), the flocculant (coefficient b6) and the Coagulant concentration (coefficient b2). The figure 6 summarizes this analysis.

In this example, we see that the effect of the factors X5, X1, X2 and X6 is important and contributes to explaining nearly 80% of the variations in the "sludge formed" response. On the other hand, the other factors do not seem significant. It is then necessary to verify that these factors are not involved in an interaction, if not, these factors can be removed from the study.

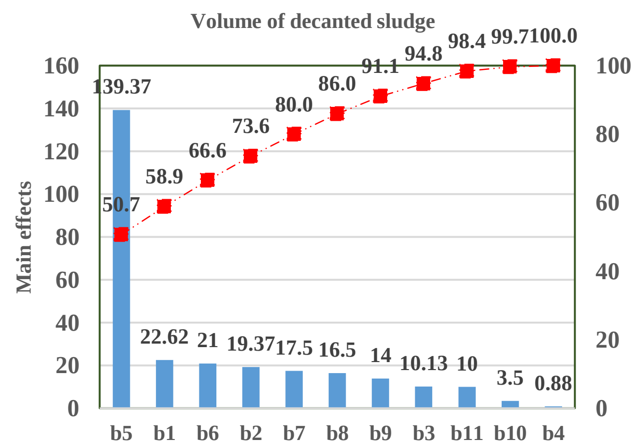

Figure 6. PARETO chart of factors effects

E. Model validation

The descriptive quality is observed as shown in figure 7 in the case of the volume of sludge formed. The plot of the first bisector promotes visualization and shows a good agreement between the computation and the measurements.

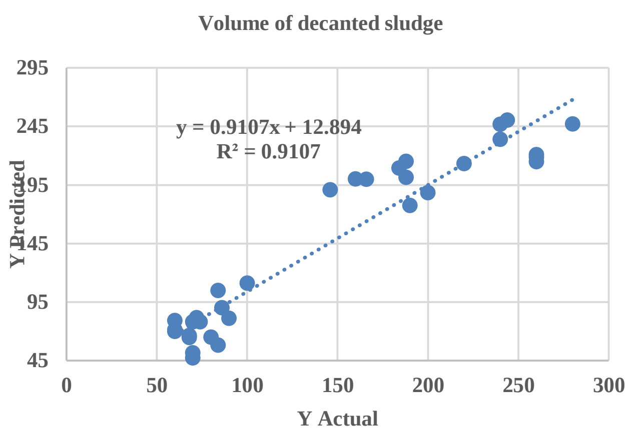

Figure 7. Correlation between theoretical and estimated yield.

In addition, table 4 also confirms the graph of suitability of the model.

IV. CONCLUSION

The study of the effect of the coagulants and of the flocculant used on the efficiency of treatment of the tannery discharges showed that the effluent removal efficiency in terms of the amount of sludge formed depends on the reagent used.

The study showed that aluminum sulfate is more efficient for the treatment of tannery effluents. However, the use of this coagulant in the presence of the chimec 5161 flocculant (anionic flocculant) improves the quality of the treated water, further reduces the residual chromium content and reduces the volume of settled sludge compared to the use of Aluminum sulfate alone.

It can be concluded that aluminum sulfate produces less sludge for the same pollution elimination efficiencies than ferric chloride $FeCl_3$ and P3000.

The $FeCl_3$ allows higher pollution removal efficiencies, generates quantities of non-excessive sludge and the cost of its use is relatively average.

To compare the results obtained with each coagulant, the ratios between the volume of settled sludge and the COD removal rate [11] and the color removal efficiency [12] Were estimated taking into account the removal performance of the organic matter and the color.

The treatment and disposal of settled sludge during the flocculation coagulation process are important aspects to consider when selecting the product to be used as a coagulant or flocculant. Considering the results obtained, taking into account the volume of sludge to be treated, the most suitable coagulant will be aluminum sulfate. In fact, the latter, and for the same yields of COD and color elimination produces less sludge than $FeCl_3$ and P3000.

The results show that the pH and the nature of the coagulant used have a significant effect on the volume of sludge formed. Coagulation flocculation generates less sludge at pH = 5 and using aluminum sulfate. However, the removal of organic matter and the color of the wastewater is effective at pH = 6.

REFERENCES

[1] M. Assou, L. El Fels, A. El Asli, H. Fakidi, S. Souabi& M. Hafidi Landfill leachate treatment by a coagulation–flocculation process: effect of the introduction order of the reagents Desalination and Water Treatment 2016.

[2] M. Assou, A. Madinzi, A.Anouzla, M.A. Aboulhassan, S. Souabi, M. Hafidi, Reducing Pollution of Stabilized Landfill Leachate by Mixing of Coagulants and Flocculants: a Comparative Study, International Journal of Engineering and Innovative Technology (IJEIT) Volume 4, Issue 1, July 2014.

[3] M. Assou, A. Madinzi, M. A. Aboulhassan and S. Souabi, Removal of Turbidity in Tannery Waste Water: Modelling by the Experimental Design International Journal of Civil and Environmental Engineering, ISSN:1701-8285, Vol.36, Issue.2 september 2014.

[4] APHA (1999). Standard methods for the examination of water and wastewater, 20th ed. American Public Health Association, Washington, DC.

[5] F. Rabier, Modélisation par la méthode des plans d'expériences du comportement dynamique d'un module IGBT utilisé en traction ferroviaire, Thèse de Doctorat de l'Institut National Polytechnique de Toulouse, 2007.

[6] S. Karam, Application de la méthodologie des plans d'expériences et de l'analyse de données à l'optimisation des processus de dépôt, thèse de Doctorat Faculté des Sciences et Techniques, Université de Limoges, 2004.

[7] S. Vivier, "Stratégie d'optimisation par la méthode des plans d'expériences et application aux dispositifs électrotechniques modélisés par éléments finis", thèse, 2002.

[8] R. V. Lenth, "Quick and easy analysis of unreplicated factorials", Technometrics, Vol. 31, N°4, 469-473, 1989.

[9] H. Toutenburg, "Statistical Analysis of Designed Experiments", Springer, seconde édition, 2002.

[10] S.S. Shapiro, M.B. Wilk, "An analysis of variance test for normality (complete samples)", Biometrika, Vol. 52, pp. 591-611, 1965.

[11] M. I. Aguilar, J. Saez, M. Lioréns, A. Soler and J. F. Ortuno, " Nutrient removal and sludge production in the coagulation-floculation process", Water res. 36, pp. 2910-2919, 2002.

[12] Aboulhassan M. A., Souabi S., Yaacoubi A., Baudu M., "Treatment of textile wastewater using a natural flocculant", Environ. Tech., 26, pp. 705-711, 2005.