QUALITY IMPROVEMENT OF THE LOCALLY PRODUCED ALUM

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ABSTRACT

Locally produced alum has approximately 40 % of inactive solid materials (i.e., silt, clay and sand), that significantly damages the dosing pump systems and others fittings of the alum lines by erosion, and create important sediments deposits in pipe-lines, flash mixers and clarifiers. In order to reach the requirements of this investigation, a suitable solid separation process should be identified and classified to take in its consideration not less than 90 % reduction of inert materials with a maximum recovery of aluminum sulfate. A bench scale arrangement based on upward flow dissolving column incorporated with an artificial interface was suggested. Flow rate, concentration of stock, sand depth and quantity of rinsing water were regarded as the most promising variable to be considered through this investigation. The experimental data was fitted using a nonlinear regression analysis in order to estimate the coefficients of a second order polynomial.

INTRODUCTION

Aluminum sulfate (alum) is the most important of all salts of aluminum because of its industrial use. It is used in the pulp and paper industry for precipitating and fixing the sizing agents, wet-strength agents and basic dyes and as pH controller in papermaking. It is used in water treatment as a coagulant, and it helps in the removal of bacteria, and control taste and color of water. It is also used in the purification of by-product glycerin and as fire-retarding agent insulating materials [1].

The coagulation mechanism of the trivalent salt of aluminum have been widely studied and may be summarized in simplified forms as follows: (i) 'Free' trivalent aluminum is released in a solution of the respective salts. However, under coagulation conditions only relatively small concentration of Al3+ is present in solution and hence their influence on coagulation may not be as great as is sometimes suggested. (ii) Hydrated metal ions are hydrolyzed, producing complex hydrated metal ions.

$$Al(H_2O)_6^{3+} \Leftrightarrow Al(H_2O)_5OH^{2+} + H^+ \qquad (1)$$

$$Al(H_2O)_5(OH)^{2+} \Leftrightarrow Al(H_2O)_4(OH)_2^{+} + H^+ \qquad (2)$$

Polymerization may also occur:

$$2Al(H_2O)_5(OH)^{2+} \Leftrightarrow Al_2(H_2O)_8(OH)_2^{4+} + 2H_2O \qquad (3)$$

It is thought that the extent of polymerization increases with age. The dimer of Eq. (3) may undergo further hydrolytic reactions yielding higher hydroxide complexes, leading to formation of positively charged colloidal polymers and ultimately to hydroxide precipitates. Both the positively charged complex ions and the colloidal hydroxo polymers are considered to play an important role in the overall mechanism of coagulation. (iii) The anions of the trivalent salts also play a part in completing the coagulation process by promoting the coagulation of any excess of positively charged metal hydroxo colloids formed. In this respect the greater coagulating power of divalent anions such as sulfates over monovalent anions chlorides may be significant [2].

In General, the vast majority of settling basins is closed with chemicals capable of producing adsorbent bulky precipitate. There are many substances, which react suitably with water to produce such an effect and these are known collectively as coagulants. Most coagulant are salts of iron or aluminum on mixing with water they act as a process of a double decomposition involving the mutual interchange of groups, the end product being hydroxides in the form of gelatinous precipitates with sulfuric acid [3]. If the acid was left in water it would recombines with the hydroxide and reverts back to sulfate. This mutual interchange of groups was going to focus the attention for finding suitable engineering solution to improve the locally produced alum in water plant station. Hydroxide is the desired end product. It is insoluble, floc forming and heavier than water, and it carries the positive electric charge necessary to neutralize the negative charge of the colloidal particles. The reasons for the addition of some form of alkali to establish the optimum pH value at which coagulation can take place and to raise the final pH value after treatment to reduce corrosiveness. In coagulation, the pH of the water is important. Floc formed in any given water tends to be heaviest at specific pH value. Most coagulants are acidic and cause the pH value of water to fall on admixture. Coagulants should be added immediately downstream of any pre-settling basin, which may have been considered necessary. Their primary purpose is to assist in the removal of the more finely divided sediment and the colloids. Most of the larger and heavier particles settle unaided in the pre-settling basin, thus permitting the coagulant to work more efficiently on the finer particles. Thorough mixing is vitally important and flocculation may be equally desirable and therefore once the pre-settlement stage has been passed (if used at all) the coagulant must be introduced as soon as possible. Soft, coloured and acid waters with pH values 5 - 6.5 are often difficult to clarify, as they needed treatment with alkali and coagulant at dosages, which tend to be narrowly critical. Waters of pH 6.2 - 7 with a reasonable degree of alkalinity react well to aluminum sulfate. Alkaline waters with pH values of 7 - 7.8 may again be difficult and absorb high doses of alum. Floc forming tends to be satisfactory below pH value of 6.8 and above 7.9. Some waters have a dead area between 6.9 and 7.8 to be corrected. Each individual water has its peculiarities, which may vary seasonally and require frequent adjustment[4].

The aim of this work was to find a suitable solution to improve the locally produced alum by reducing its inert materials with a maximum recovery of aluminum salfate.

EXPERIMENTAL WORK

Through the course of filtration two concentrations have been studied (25 wt % and 50 wt %), the filter medium was consisted of gravel gradually increase from 6 mm to 5 cm. This filtration technique was able to minimize the insoluble materials up to 33 % of the inactive materials. Back washing the filter medium is required to avoid any blockage could be happened, but in the other hand the back washing caused a big loss aluminum sulfate. Accordingly, this technique is not effective in reducing percent of the inactive materials to an acceptable limit besides loses in the aluminum sulfate. Another technique has been conducted using a horizontal flow-settling tank. Different concentrations (25 wt % and 50 wt %) have been used and different flow rates (1 lit/min, 3 lit/min and 5 lit/min). The results were underestimated the problem since only heavy particles were precipitated.

Experimental Design

In chemical industry experimental designs are practically applied to the study of process variables and how they affect the product. Their quantitative effect on yield are estimated by the regression coefficients [5]. Montgomery [6] had postulated that a mathematical model of second degree polynomial or higher is usually required. The representation of the response space takes into account the effect generated by each variable, as well as the interaction of the variables reflected on the response. So one must ensued a systematic method, which follow the response function that relates the dependent variables with the independent one with minimum number of experiments. Hence an experimental design technique was followed in order to determine the most important factors among the variables under study, how these variables influence the response of the simulation model, and why certain results occur as they do in the simulation experiments.

In lab scale investigations, the effects of process variables on purification of locally produced alum are studied. The experimental work was designed in following experimental ranges: -

1. Flow rate of the influent ranged from 0.4 to 4 liter/hr, coded as X1.
2. Concentration of alum ranged from 0.05 to 0.45 kg/liter, coded as X2.

3. Sand layer height ranged from 5 to 20 cm, coded as X3.
4. Quantity of rinsing water ranged from 1 to 9 liter, coded as X4.

The coded levels were related to real values of four variables as follows:

$$X_1 = \frac{F-2.2}{0.9}, \qquad (4)$$

$$X_2 = \frac{C-0.25}{0.1}, \qquad (5)$$

$$X_3 = \frac{H-0.4}{3.75}, \qquad (6)$$

$$X_4 = \frac{Q-1}{2} \qquad (7)$$

Where F is the flow rate of the influent in lit/hr, C is the alum concentration in kg/lit, H is the thickness of sand layer in cm and Q is the quantity of rinsing water in liter. The working ranges of coded and corresponding real variables are listed in Table (1). Accordingly, there are twenty seven experiments where the coded values +2, -2, and 0 represent the maximum, minimum and center values respectively.

Settling Experiments

Settling experiments as pictorial in Fig (1) were conducted in a column (7.5-cm inside diameter and 1 m depth) incorporated with six sampling ports located at different depths. The column was axially mounted with a stirrer of six blades configured at different depths to homogenize the admixture along the column.

The procedure was that after dissolving the stock, the first course started by homogenizing the slurry along the column using stirrer action for at least 10 minutes. Then leaving the admixture in rest to let all the solid particles to be settled down at a level just beneath the second port from the bottom. Now the solution just above the interface is withdrawn and analyzed. The second course started by filling the column with fresh water and once more again the admixture was homogenized by stirring the contents for at least 10 minutes and letting the contents to settle down at a level just beneath the second port from the bottom. Then the solution just above the interface is withdrawn and analyzed.

Washing Experiment

This arrangement as pictorial in Fig (2) was consisted of 7.5-cm in diameter and 50 cm height column. At the upper end of the column sand layer was fixed on mesh support. At the lower end of the column there was a reducer incorporated with a flexible tube connected supplying the fresh water from the elevated storage tank. The supplier line was incorporated with calibrated rotameter and valve.

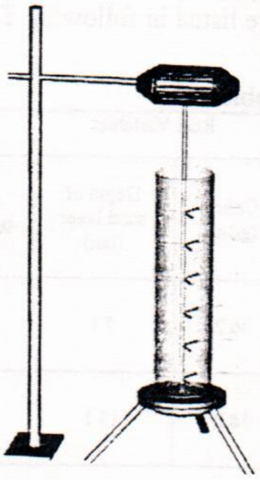

Fig. (1) Settling experiments layout

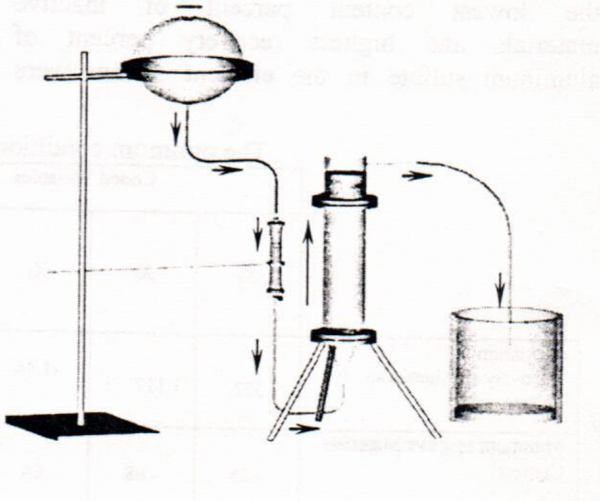

Fig. (2) Washing experimental equipment

The procedure was that the required concentration of locally alum is prepared by dissolving the required quantity of the stock in a separated vessel. Then purring the solution inside the column after pulling out the sand layer. Now, after replacing the sand layer, the desired quantity of water with the designed flow rate is introduced from the bottom of the column. The over flow is collected and analyzed.

RESULTS AND DISCUSSION

Through the course of this technique, many tests have been conducted, using different alum concentrations. It was noticed that stable suspension was generated at concentrations higher than 35 % w/v (i.e., long-term stability). Many trials have been conducted using fresh water enters from the lowest port of the column. Washing experiments have been conducted according to the conditions provided by the experimental design to obtain the influence of each variable on the recovery of aluminum sulfate and the content of inactive materials.

Experimental design is frequently performed between one or more measured responses and a number of variables. The recovery percent of aluminum sulfate and the percent of inactive materials in the effluent stream are correlated with four variables (i.e., flow rate, alum concentration, thickness of sand layer and quantity of rinsing water) by two-second order polynomials. A nonlinear analysis was carried out to estimate the coefficients of the correlation and optimum condition were obtained.

Using the coded data of the central composite rotatable Box Wilson design method, the coefficients of a second order polynomial were estimated and found as shown in Eqs (8) and (9). The number of iteration was terminated when the correlation coefficients were equal to 0.99.

The equations of the weight percent of inactive materials and the percent of aluminum sulfate recovery in the effluent stream are respectively as follows:

$$\begin{aligned} Y_{\text{inactive}} &= 6.6043 + 2.3187\ X_1 + 4.0588\ X_2 - 1.2455\ X_3 - 2.1732\ X_4 + 0.0167\ X_1^2 \\ &+ 0.6639\ X_2^2 + 0.3847\ X_3^2 + 0.1132\ X_4^2 + 0.5600\ X_1X_2 - 0.1160\ X_1X_3 \\ &- 0.2258\ X_1X_4 - 0.3881\ X_2X_3 - 1.0311\ X_2X_4 - 0.4459\ X_3X_4 \end{aligned} \tag{8}$$

$$\begin{aligned} Y_{Al_2(SO_4)_3} &= 83.7765 + 0.4442X_1 + 0.3685X_2 - 0.5465X_3 + 15.4754X_4 - 0.1723X_1^2 \\ &726X_2^2 + 0.3847X_3^2 + 0.1132X_4^2 + 0.5600X_1X_2 + 0.1160X_1X_3 \\ &+ 0.2258X_1X_4 + 0.3881X_2X_3 + 1.0311X_2X_4 + 0.4459X_3X_4 \end{aligned} \tag{9}$$

Optimum values of the studied variable at the lowest content percent of inactive materials and highest recovery percent of aluminum sulfate in the effluent stream were determined by optimizing the proposed two correlation via Hooks and Jeeves constrained method. The optimum conditions in coded and real values are listed in following Table.

The optimum conditions in coded and real variables

	Coded Variables				Real Variables			
	X_1	X_2	X_3	X_4	Flow Rate (Lit/h)	Conc. (g/lit)	Depth of sand layer (cm)	Rinsing Water (liter)
Maximum Recovery of Aluminum Sulfate	1.322	1.117	-1.44	-.0044	3.4	36.2	7.1	5
Minimum Inactive Materials Content	-.68	-.68	.68	1.68	1.6	18.2	15.1	8.4

Figures from (3) to (16) shows the effect of the variables on recovery percent of aluminum sulfate and content percent of inactive materials.

Figures (3), (4) and (5) shows the effect of flow rate, alum concentration and sand layer height on recovery of aluminum sulfate. It was obviously that insignificant effects had observed from these variables on the recovery of aluminum sulfate in comparison with the quantity of rinsing water as shown in Fig. (6), which represents the most dominate parameter in controlling the recovery of aluminum sulfate. This was ascertained from statistical analysis for the significance where only (X4) was found significant.

Figures (7) to (10) shows the individual effect of flow rate, alum concentration, sand layer thickness and the quantity of rinsing water on the content of inactive material. Figure (7) and (8) shows, increasing the flow rate and alum concentration will increase the content of inactive materials in the effluent streams respectively, and these would bring a serious limitations in confining larger quantities of raw alum within a specified period. Customarily, increasing the flow rate would resulted higher degree of turbulence of the admixture, besides increasing the channeling in the sand layer that leads larger quantity of inactive materials to passerby the sand layer and consequently increasing its content in effluent streams. Whereas, increasing the alum concentration inside the column would increase the content of the inactive materials that makes the suspension beneath the sand layer to endue long-term stability and resulted in higher content of inactive materials in effluent streams.

Figure (9) and (10) shows that increasing sand layer thickness and quantity of rinsing water will decrease the content of inactive materials in effluent streams. Increasing the sand layer height and its contributions to lesser solid content in effluent streams was simply attributed to larger resistance for the solid materials to passerby the sand layer. Whereas, increasing the quantity of rinsing water would result in decreasing the concentration of aluminum sulfate beneath the sand layer that consequently resulting in floc formation and increasing the settling velocity of solid materials. Also the four figures, Figs (7) and (10) had shown linear dependence of the content of inactive materials on flow rate X1 and quantity of rinsing water X4 while nonlinearly behavior had observed in studying the effect of alum concentration X2 and sand layer height X3.

Figures (11) to (16) monitored the interaction effects of two variables where individual recurrence of the four studied variables on the content of inactive materials were demonstrated. Examining these figures had postulated same findings as previously outlined from studying the effect of each term on the content of inactive materials individually.

CONCLUSIONS

- Using settling tank to obligate the objectives of this study, will increase the active material losses and may need auxiliary process, so it is not reasonable to use such technique.
- Upward flow clarifier has proved to be adequate treatment to fulfill the requirements of this study for purification of alum from inactive materials.
- Two mathematical models of the objective functions (active material recovery and inactive material content) in terms of four variables (i.e., flow rate, raw alum concentration, thickness of sand layer and quantity of rinsing water) were developed; both equations describe the behavior of the process within the studied range in bench scale arrangements.
- Optimum conditions were predicted as shown in the Table above.

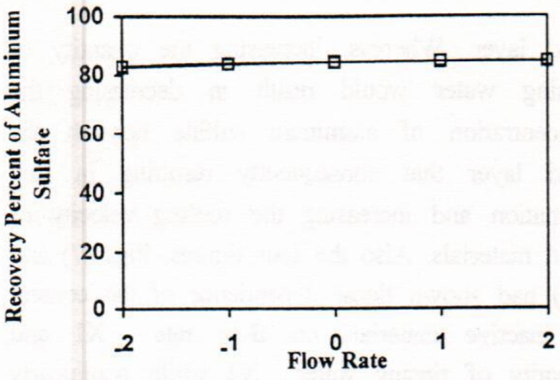

Fig (3) The influence of flow rate on recovery percent of aluminum sulfate in effluent at optimum alum concentration, sand layer thickness and quantity of rinsing water

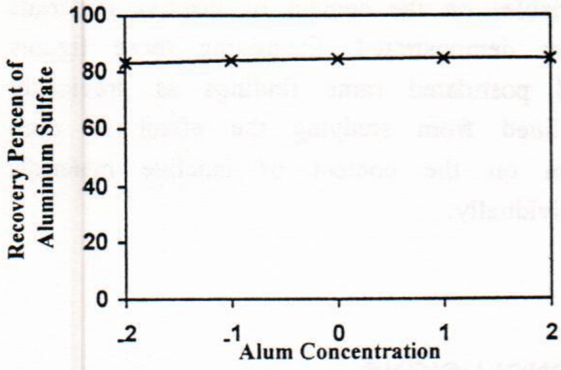

Fig (4) The influence of alum concentration on percent content of inactive materials in effluent at optimum flow rate, sand layer thickness and quantity of rinsing water

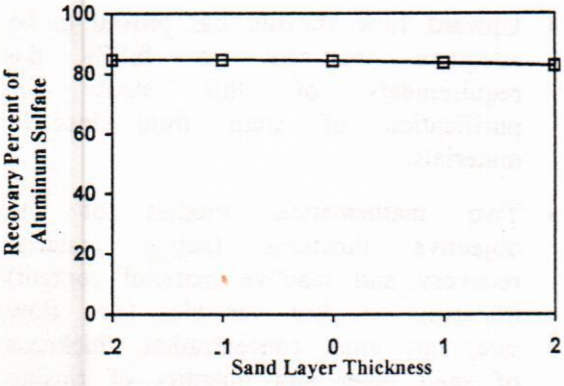

Fig (5) The influence of sand layer thickness on percent content of inactive materials in effluent at optimum flow rate, alum concentration and quantity of rinsing water

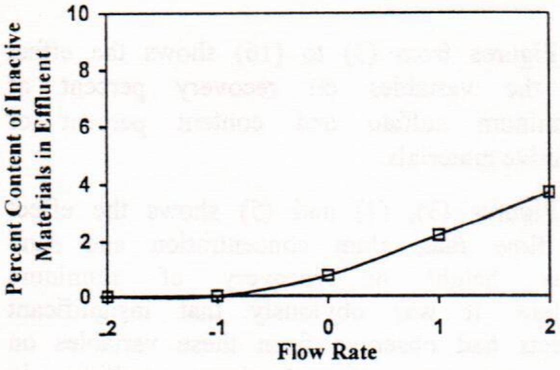

Fig (6) The influence of flow rate on percent content of inactive materials in effluent at optimum alum concentration, sand layer thickness and quantity of rinsing water

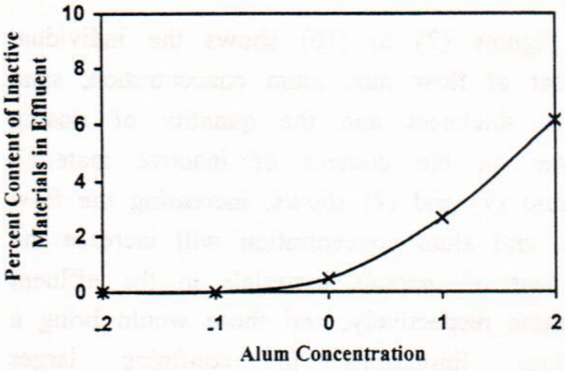

Fig (7) The influence of alum concentration on percent content of inactive materials in effluent at optimum flow rate, sand layer thickness and quantity of rinsing water

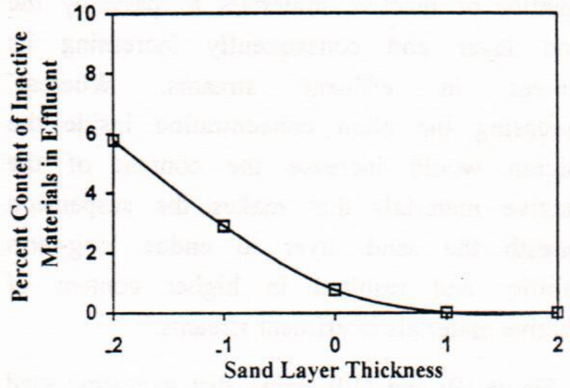

Fig (8) The influence of sand layer thickness on percent content of inactive materials in effluent at optimum flow rate, alum concentration and quantity of rinsing water

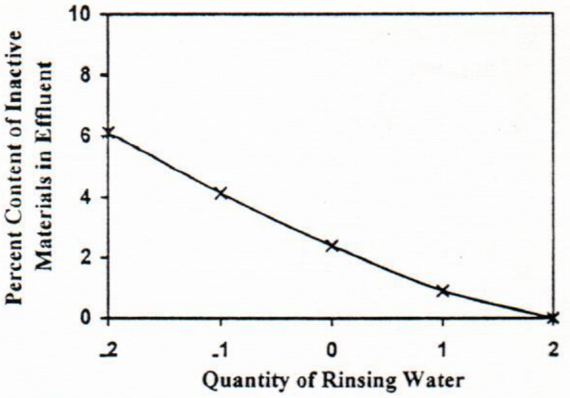

Fig (9) The influence of quantity of rinsing water on percent content of inactive materials in effluent at optimum flow rate, alum concentration and sand layer thickness

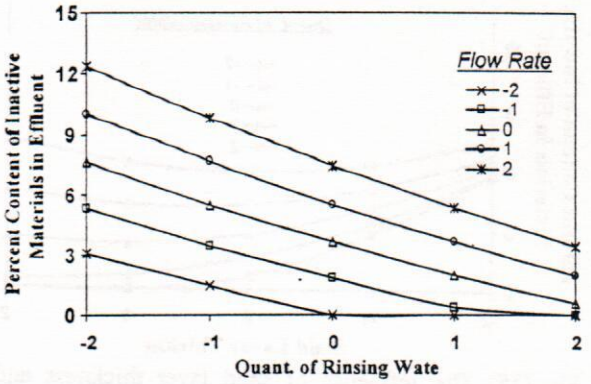

Fig (12) The influence of flow rate and quantity of rinsing water on the percent content of inactive materials in effluent at optimum sand layer thickness and alum concentration

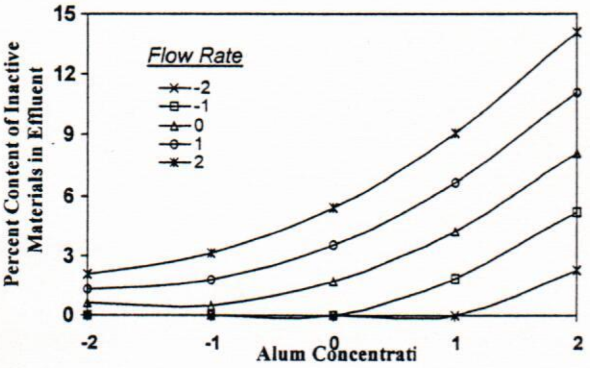

Fig (10) The influence of alum concentration and flow rate on the percent content of inactive materials in effluent at optimum sand layer thickness and quantity of rinsing water

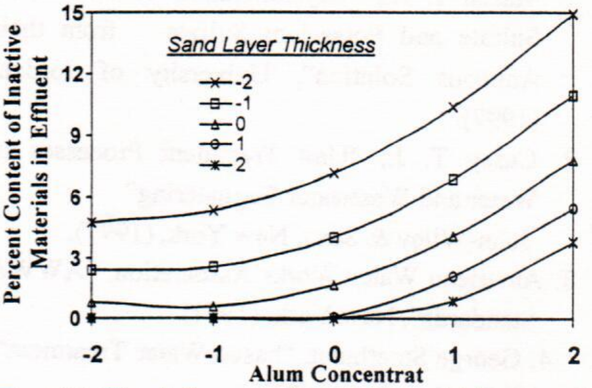

Fig (13) The influence of sand layer thickness and alum concentration on the percent content of inactive materials in effluent at optimum flow rate and quantity of rinsing water

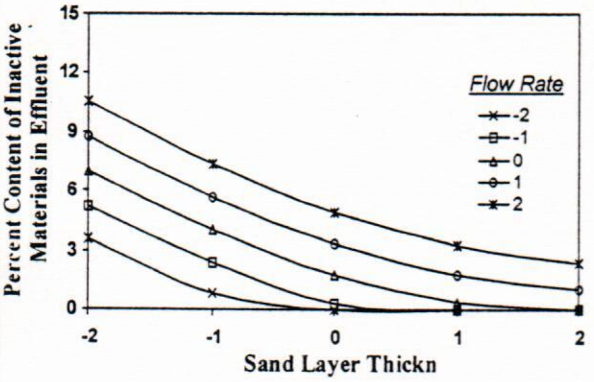

Fig (11) The influence of sand layer thickness and flow rate on the percent content of inactive materials in Effluent at optimum alum concentration and quantity of rinsing water

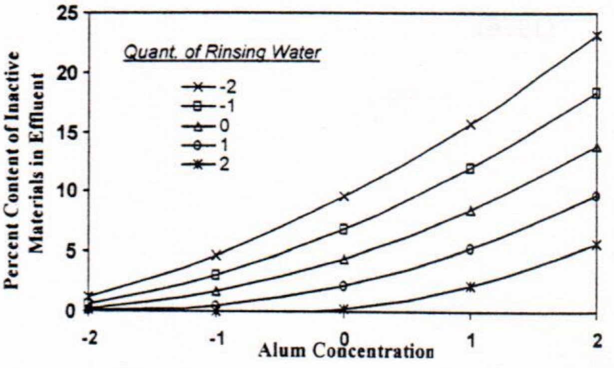

Fig (14) The influence of alum concentration and quantity of rinsing water on the percent content of inactive materials in effluent at optimum sand layer thickness and flow rate

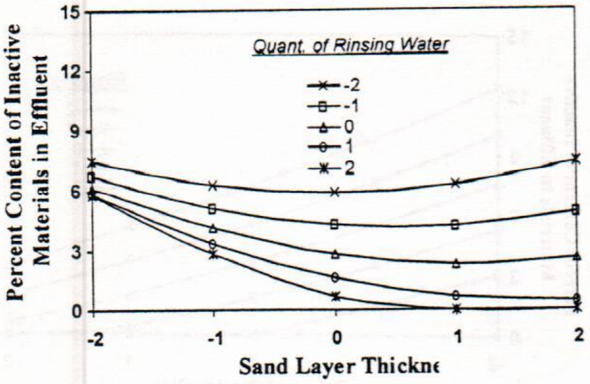

Fig (15) The influence of sand layer thickness and quantity of rinsing water on the percent content of inactive materials in effluent at optimum flow rate and alum concentration

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