# Settling Characteristics of Wastewater from the Ceramic Tile Industry Treated with Different Coagulants

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**Abstract**— The settling characteristics of wastewater sludge emanating from a ceramic tiles factory were investigated. Settling characteristics were investigated by tracing the height of interface (h, cm) against time (t, hours). Three coagulants were used in different proportions, namely, carboxymethyl cellulose (CMC), alum, and polyacrylamide (PAM). It could be concluded that: CMC has to be discarded as for its possible use as deflocculating agent to assist precipitation of the solid matter in sedimentation tanks. The obtained results clearly play in favor of using PAM in sludge treatment and possible recovery. However, economic factors have to be taken into consideration owing to the large difference in its price compared to that of alum.

**Index Terms**— Wastewater treatment, Tile industry, settling rate, flocculating agents.

## 1 Introduction

WASTEWATER polluted with various contaminants from domestic, industrial, commercial and institutional sources needs to undergo efficient treatment. It is highly important to treat the wastewater before it is discharged or re-used to protect the environment and health of people [1].

Industrial wastewater treatment is a complex problem for a variety of highly polluting chemical industries such as fertilizer, distillery, dyes and pigment, textile and specialty chemical manufacturing. Generally, the industrial effluents are characterized in terms of broad parameters such as Chemical Oxygen Demand (COD), Ammoniacal Nitrogen (AN), Total Suspended Solids (TSS), Total Dissolved Solids (TDS) etc. [2].

The challenge is in the selection of most appropriate process(es) from physico-chemical methods such as adsorption/ ion exchange/ membranes, newer forms of advanced oxidation processes etc.; biological methods such as aerobic or anaerobic treatment or combination of both, for any specific industry sector/effluent stream. The volume of effluent, nature and concentration of pollutants and total cost of treatment usually dictates the selection [3].

Coagulation and flocculation is a coupled process used widely in numerous industries as a vital part of the overall treatment of wastewater. The principal aim of coagulation and flocculation is to decrease turbidity of wastewater. This refers to the cloudiness of water mainly due to the presence of suspended particles, namely colloids. The choice of a proper coagulant and its optimal dose is a challenging issue in many industries [4, 5].

Coagulation/flocculation is a straight forward and effective technique for wastewater treatment and has been broadly utilized for the treatment of different sorts of wastewater, for example, material wastewater, mash factory wastewater, sleek wastewater, clean landfill leachates and others [6-8].

Coagulation/flocculation is defined as the addition of inorganic salts or organic polymers that help destabilizing fine solids suspension. Combination of colloidal particles and suspensions with very fine solid particles can form large agglomerates in coagulation process. Polymeric flocculants show more benefits compared to inorganic flocculants since flocs are formed more rapidly are larger and stronger than inorganic flocs. Also, polymer dosage for polymeric flocculants is commonly less than inorganic flocculants (almost 1% and 20% respectively on a dry weight basis) [9, 10]. However their cost tends to be much higher than their inorganic counterparts.

Polymeric flocculants such as natural and synthetic polymers use bridging for flocculation. Natural polymers have less molecular weight compared to synthetic polymers besides being very expensive and occasionally toxic. Synthetic polymers, on the other hand are relatively cheaper and are capable of a chieving reasonable flocculation by modifying molecular weight or structure [11].

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Polyacrylamide and its derivative are the most commonly used synthetic flocculants. Polyacrylamide is generally nonionic and the ionic part is divided into anionic and cationic groups. Anionic polyacrylamide may be prepared by partial hydrolysis of polyacrylamide or by polymerization of acrylic acid and acrylamide together. Polymerization of acrylamide with some derivatives of it, forms cationic polyacrylamides. Depending on the application, the type of cationic material can be varied [12, 13]. Figure (1) illustrates a schematic of polyacrylamide polymerization.

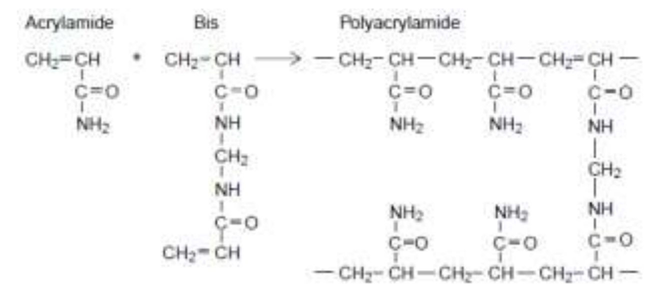


Fig. (1): Illustration of polyacrylamide polymer.

Another polymeric flocculant is carboxymethyl cellulose (CMC). It is a derivative of cellulose formed by its reaction with alkali and chloroacetic acid. CMC dissolves rapidly in cold water and is mainly used for controlling viscosity without gelling. As its viscosity drops during heating, it can be used to improve the volume yield during baking by encouraging gas-bubble formation. The controlled viscosity of CMC allows its use as a thickener, phase and emulsion stabilizer (e.g., with milk casein), and suspending agent. The more hydrophobic, lower substituted CMCs are thixotropic, but the more extended, higher substituted CMCs are shear thinning [14].

Some of the well-known and common inorganic coagulants used for water treatment are aluminum sulfate, iron (III) chloride and iron (II) sulfate. Aluminum sulfate is often referred to as alum, although this terminology is not an accurate description since chemically, the alum compound is a much more complex salt of aluminum usually consisting of aluminum sulfate, water of hydration and the sulfate of another element such as hydrated aluminum potassium sulfate [15].

When adding aluminum sulfate as a coagulant, it interacts with any natural alkalinity present in water, leading to a reduction of pH [16]. It is very effective in coagulating inorganic suspended solids, being less effective in case of organic solids. That is why its dosage largely depends on the wastewater composition [17]. Aluminum sulfate has been successfully used in removing phosphate, fluoride, and other harmful compounds such as arsenic along with the other coagulants such as ferric chloride or ferric sulfate, because of their low cost and relative ease of handling [18, 19].

In the present study, the settling characteristics of ceramic sludge suspension were investigated using three different coagulants, namely carboxymethyl cellulose, (CMC), Polyacrylamide (PAM) and Alum.

## 2 RAW MATERIALS AND EXPERIMENTAL TECHNIQUES

### 2.1 Raw Materials

#### 2.1.1 Sludge samples

The sludge samples employed for the experiments were kindly supplied by a ceramic tile plant located in Al Sharkia Governorate, Lower Egypt.

#### 2.1.2 Coagulants

All coagulants used in this study were diluted in distilled water before using. Table (1) summarizes the properties of the three coagulants as for their formula and molecular weight.

TABLE (1)
COAGULANTS CHARACTERISTICS

| Coagulant | Chemical | Formula | M.W. g.mol$^{-1}$ |
|---|---|---|---|
| **CMC** | Carboxymethyl Cellulose | $C_8H_{16}O_8$ | $3.9304\times10^5$ |
| **PAM** | Polyacrylamide, poly (2-propenamide) | $(C_3H_5NO)n$ | $4.1486\times10^4$ |
| **Alum** | Aluminum sulfate | $Al_2(SO_4)_3$ | 342 |



### 2.2 Settling Characteristics

The settling characteristics were investigated by plotting the height of interface (h, m) against time (t, hours). The main criterion for speed of settling has been taken as the initial settling rate since in most cases more than 80% of the solids will have settled though that period. It was calculated as the slope of the h – t line in the initial linear portion of the curve.

To study the effect of additive concentration a series of experiments were performed using different coagulant concentrations. Alum and CMC were diluted in concentrations ranging from 0.1 to 1% of ceramic sludge waste while PAM was diluted in concentrations ranging from 0.2 to 0.6 % of waste sludge.

## 3 RESULTS AND DISCUSSION

### 3.1 Settling of Untreated Sludge

Figure (2) illustrates settling curve obtained on performing the sedimentation experiment on an untreated specimen of sludge of solid concentration equals 152.8kg.m$^{-3}$.

This figure indicates a linear relation between interface height and time extending from the original suspension height of 0.5 m and extending to about 2 hours after which the slope of the *h – t* relation starts decreasing until a final height of about 0.29 m is reached after about 10 hours.

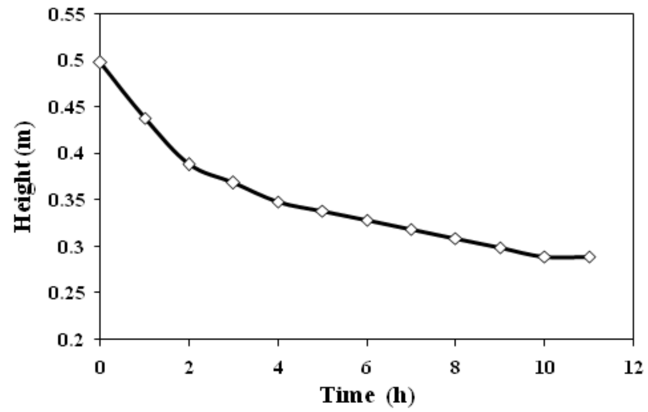


Fig. (2): Sedimentation curve for untreated sludge.

The initial rate of settling as calculated from the numerical value initial of the slope of the curve was found to equal 0.0547 $m.h^{-1}$. The final asymptotic height of interface was about 0.288 m.

### 3.2 Effect of Addition of CMC

As CMC was added the rate of settling suffered a radical decrease. This can be followed in Figure (3) where the combined $h - t$ plots are displayed for different addition levels of CMC reaching 1% by weight.



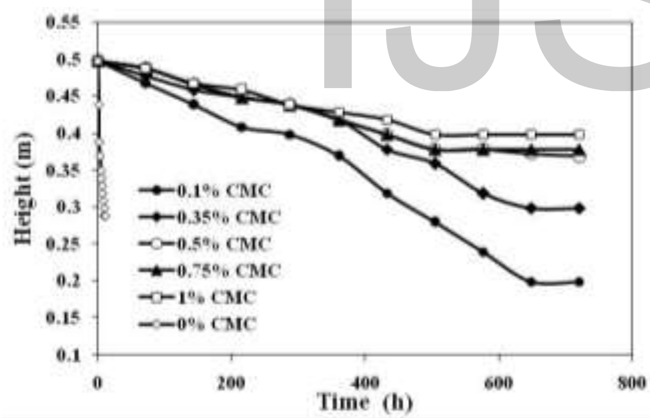


Fig. (3): Sedimentation curves for sludge treated with CMC.

First, these curves show a tendency of decreased initial slope following an increase in CMC addition. Also, the final height stabilizes at values that increase with increased CMC level. This implies that the presence of CMC simply hinders the settling process without any flocculating effect. This is probably due to the increase in viscosity associated with that addition. In Figure (4) this viscosity is seen to increase from about 2.6 cP for the untreated specimen to more than 50 cP in case of 1% CMC addition, as determined using a Brookfield viscometer. Another feature common to the sedimentation curves in case of specimens doped with CMC is that the rate of settling remains almost constant for long periods of time to stabilize after a period ranging from 500 to 650 hours.

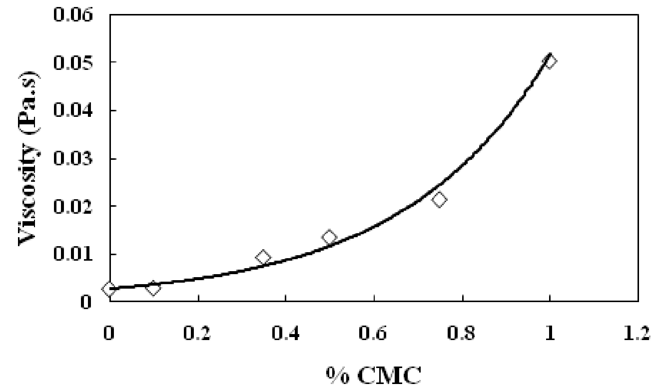


Fig. (4): Effect of CMC addition on apparent viscosity of sludge suspensions.

Figure (5) was drawn to show the effect of CMC addition level on the rate of settling.

On the other hand, Figure (6) illustrates the final height reached each time.

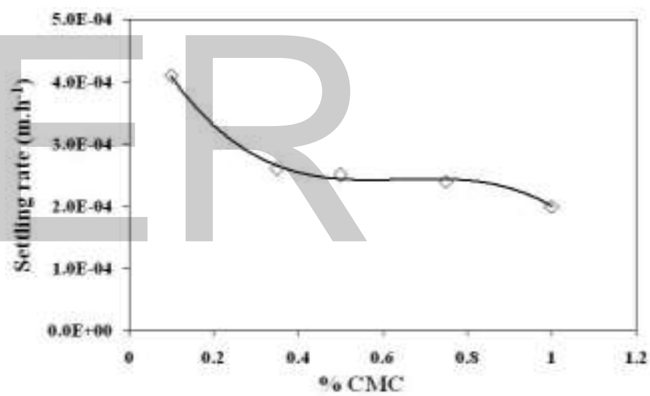


Fig. (5): Effect of CMC addition on sedimentation rate.

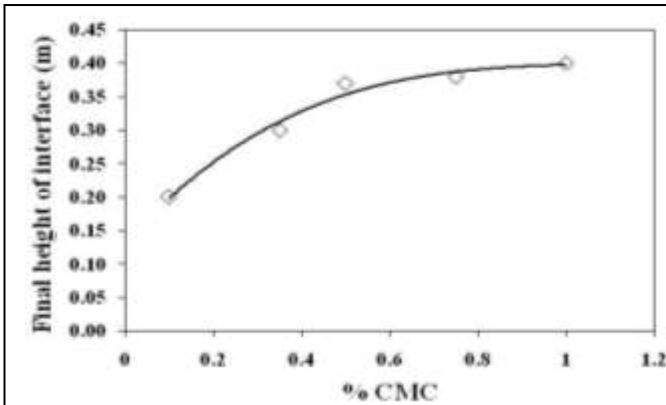


Fig. (6): Effect of CMC addition on final height.

In conclusion, the addition of CMC was found to be extremely notorious on the sedimentation properties of the suspension tending to keep its particles in deflocculated form and drastically decreasing the rate of sedimentation. Effectively, a minor addition of 0.1% CMC had for effect to reduce the sedimentation rate from 0.0547 m.h$^{-1}$ down to 0.00041 m.h$^{-1}$, a 133 fold decrease.

This has led to totally discard CMC from any consideration as for its possible use as deflocculating agent to assist precipitation of the solid matter in sedimentation tanks.

### 3.3 Effect of Addition of Alum

The effect of adding alum with different percentages to ceramic sludge on the sedimentation curves is displayed in Figure (7). It indicates that the time required to reach a practically constant height of interface is about 2 hours for suspensions containing alum regardless of its content, compared to more than 10 hours in case of untreated sludge.

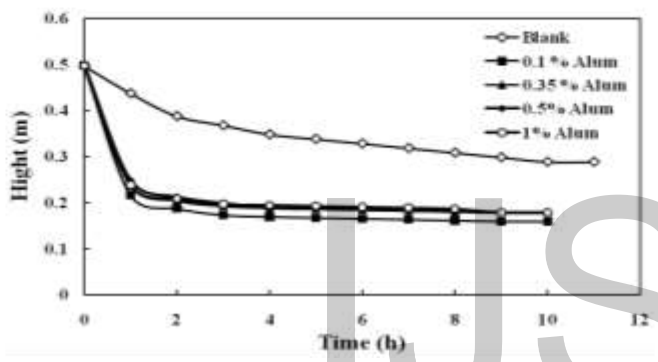


Fig. (7): Sedimentation curves for sludge treated with alum.

In that figure the sedimentation curves obtained on adding alum were compared to that of pure sludge. The following features can be observed: First, there is an appreciable settling effect as evidenced by the decrease in final height of suspension. Second, there is a marked increase in the rate of settling as proven from the increase in slope of the h – t linear portion on adding alum. Third, the addition of alum has for effect to rapidly reaching the final compressed height. These features are displayed in Figures (8) and (9) respectively.

Figure (8), also reveals that the addition of alum has decreased the final height of suspension from about 0.29 m for untreated sludge to a nearly constant value of about 0.18 m regardless of the amount of alum added.

### 3.4 Effect of Addition of PAM

When PAM polymer was added to ceramic sludge and sedimentation experiments performed, the results shown in Figure (10) were obtained. In these curves, a common behavior can be noticed, namely, the presence of two sedimentation zones with different slopes. The predominant feature, however, is the extremely rapid reaching of steady state compression. The time required to reach that final stage is a few minutes compared to about 10 hours in case of untreated sludge.

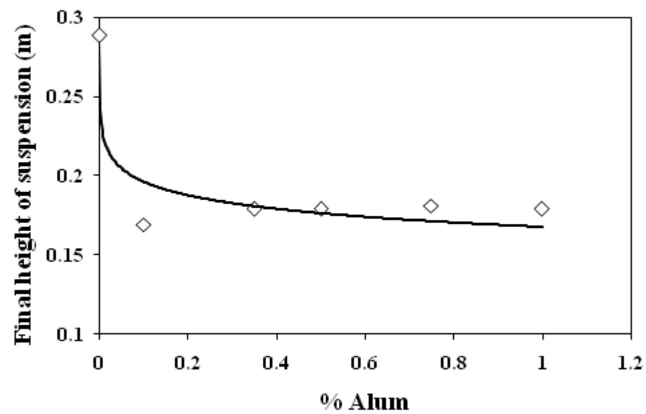


Fig. (8): Effect of alum addition of final height of suspension.

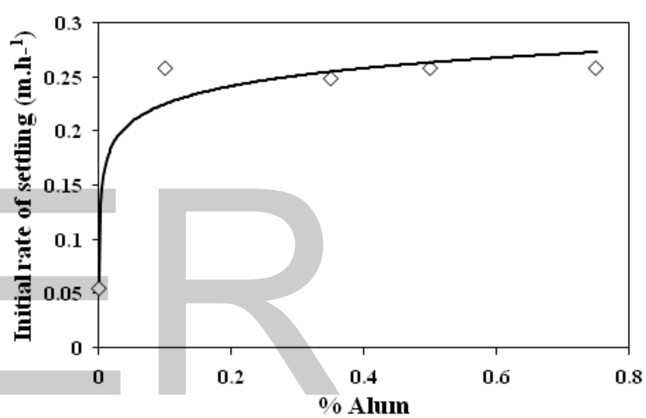


Fig. (9): Effect of alum addition of initial rate of settling.

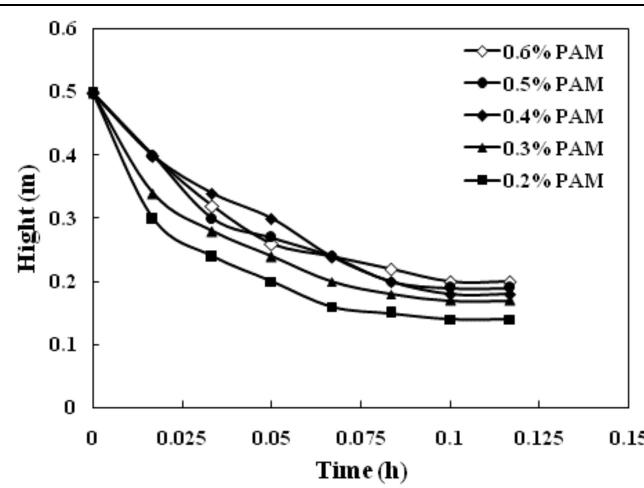


Fig. (10): Sedimentation curves for sludge treated with PAM.

As for the final height of suspension, the addition of PAM drastically reduces that height, therefore increasing the amount of settled solids. As can be followed in Figure (11), adding PAM has for effect to reduce the limiting height of suspension from about 0.29 m in case of untreated sludge to less than half that value upon adding 0.2% PAM. Increasing the polymer addition is accompanied by a slight increase in that height.

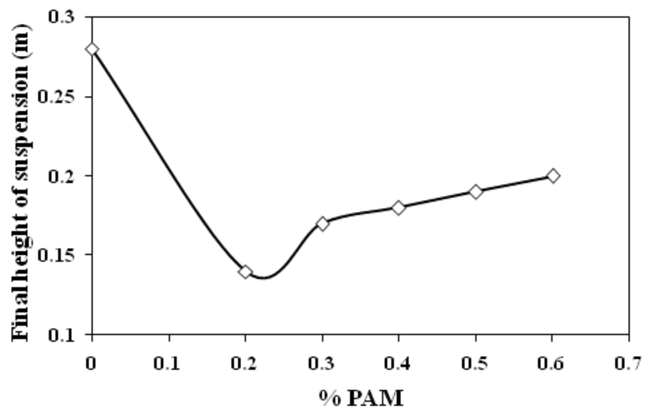


Fig. (11): Effect of PAM addition of final height of suspension.

It is worth mentioning that the solid concentration of the untreated sludge = 152.8 kg.m$^{-3}$. Taking into consideration that the specific gravity of the solids is about 2500 kg.m$^{-3}$, this corresponds to a volumetric concentration of approximately 0.061 m$^3$ solids per m$^3$ water. Since the total volume of solid is constant, the following equality can be set between the initial conditions (0) of the suspension and its final conditions (f):

$$C_0 V_0 = C_f V_f \quad (1)$$

Where:

$C_0$: is the initial volumetric concentration of solids, in m$^3$ solids per m$^3$ water.
$C_f$: is the final volumetric concentration of solids in compressed zone, in m$^3$ solids per m$^3$ water.
$V_0$: is the initial volume of suspension, in m$^3$ water.
$V_f$: is the final volume of the compression zone, in m$^3$ water.

Since the cross – sectional area of the graduated cylinder is constant then this expression can be modified to read:

$$C_0 h_0 = C_f h_f \quad (2)$$

Where:

$h_0$: intial height of the interface, in m
$h_f$: final height of the interface, in m

Actually, the initial volumetric concentration of the sludge suspension = 0.061 m$^3$ solids per m$^3$ water. Consequently, substituting with $h_f$ = 0.14 m in equation (2) will result in a maximum solid volumetric concentration in compression zone = 0.218 m$^3$ solids per m$^3$ water.

Also, the rate of settling in the initial linear period was found to increase sharply upon adding PAM as can be seen from Figure (12). Actually the rate increases so rapidly that it was necessary to plot the logarithm of that rate rather than its value against PAM addition. The initial sedimentation rate increased from about 0.0547 m.h$^{-1}$ in case of untreated sludge to about 4.5 m.h$^{-1}$ for treated sludge, regardless of the percentage of PAM added.

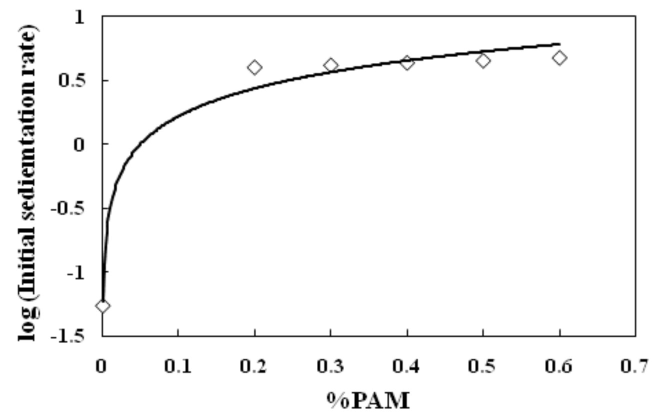


Fig. (12): Effect of PAM addition of initial rate of settling.



### 3.5 Comparison between Different Additions

The aforementioned results clearly play in favor of using PAM in sludge treatment and possible recovery. However, economic factors have to be taken into consideration owing to the large difference in its price compared to that of alum. Current prices of alum powder range from $ 120 to 200 per ton compared to $ 1000 to 2500 for PAM. The choice of any particular addition will be dictated by the benefit of having a much faster sedimentation rate in case of the much more costly PAM compared to using alum.

## 4 CONCLUSION

The following results could be drawn from the present research:

1. The initial rate of settling of untreated sludge as calculated from the numerical value of the initial slope of the curve was found to equal 0.0547 m.h$^{-1}$. The final asymptotic height of interface was about 0.288 m.
2. The addition of CMC was found to be extremely notorious on the sedimentation properties a minor.
3. Addition of 0.1% CMC had for effect to reduce the sedimentation rate from 0.0547 m.h$^{-1}$ down to 0.00041 m.h$^{-1}$, a 133 fold decrease.
4. Addition of alum decreases the final height of suspension, increases the settling rate and fastens reaching the final compressed height.
5. The addition of PAM decreases the time required to reach that final stage to a few minutes compared to about 10 hours in case of untreated sludge. Adding PAM has for effect to reduce the limiting height of suspension from about 0.29 m in case of untreated sludge to less than half that value upon adding 0.2% PAM.

6. It could be concluded that: CMC has to be discarded as for its possible use as deflocculating agent to assist precipitation of the solid matter in sedimentation tanks. The obtained results clearly play in favor of using PAM in sludge treatment and possible recovery. However, economic factors have to be taken into consideration owing to the large difference in its price compared to that of alum.

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