CONDITIONING PROCESS AND CHARACTERIZATION OF FRESH ACTIVATED SLUDGE

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Abstract

Fresh activated sludge in many wastewater treatment plants may be considered unhealthy due to the large amount of organic and organism content. Due to the lack of research on municipal sludge, there is an apparent scarcity of actual data. Thus, this work will focus on the characterization of fresh activated sludge. The effect of dosage of different polyelectrolytes and coagulants has been investigated at pH level in a comparative fashion that is commonly associated with fresh activated sludge. The results indicated that the cationic polyelectrolytes had significant influence on the sludge properties, degree of flocculation and water quality. With respect to the optical analyses, it was observed that the floc sizes and densities were increased with rise concentrations of both types of cationic polyelectrolytes. It was found that the cationic CPAM-80 was the most effective chemical among other six used chemicals especially for solutions with pH near neutrality despite of the variations in feed properties of the fresh activated sludge. This polyelectrolyte gave lower turbidity, lower sludge volume index, faster zone settling rate and large floc density.

Keywords: Polyelectrolyte, Settling volume index, Zone settling rate, Floc density.

1. Introduction

Sludge is a multi-component mixture consisting of water mostly with 95%, organic content, micro-organism and colloids. It is a very unstable product depending on its origin and because of its characteristics, may pose some problems related to the daily operation such as cost related to its transportation, disposal treatment process [1-4].

One common challenge in the operations of the wastewater treatment are bulking,
Conditioning Process and Characterization of Fresh Activated Sludge

Abbreviations

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
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<tbody>
<tr>
<td>APAM</td>
<td>Anionic polyacrylamide</td>
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<tr>
<td>CPAM</td>
<td>Cationic polyacrylamide flocculants</td>
</tr>
<tr>
<td>FSS</td>
<td>Fixed suspended solid</td>
</tr>
<tr>
<td>HMW</td>
<td>Superfloc high molecular weight</td>
</tr>
<tr>
<td>NTU</td>
<td>Nephelometric Turbidity Unit</td>
</tr>
<tr>
<td>SVI</td>
<td>Sludge volume index</td>
</tr>
<tr>
<td>TSS</td>
<td>Total suspended solid</td>
</tr>
<tr>
<td>VSS</td>
<td>Volatile suspended solid</td>
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</tbody>
</table>

pin flocs and rising sludge [5]. For example, bulking could be caused by low sludge density or due to trapped water or gas between the solid flocs. Thus, bulking may be considered as a major operation problem which may lead to difficulties in separation of sludge water. Also, poor settling of waste sludge is caused by growth of filamentous bacteria and fungi [6]. Although the presence of bacteria is helpful for the removal of organic waste materials, presence filamentous bacteria responsible for weak flocs and thus leads to low sludge settling.

It is well known that the composition of the real activated sludge in wastewater plants continuously unstable. The constant changing characteristics vary depending on the origin of the feed and time at which enter the aeration section in the wastewater. As well, the differences in the design and operation of the treatment plants can also have an impact on the instability of activated sludge [7]. Additionally, the living micro-organisms in the sludge are complicated and also, unstable [8]. Consequently, many researchers have considered the use of synthetic sludge in order to carry out the experiment under ideal controlled conditions [8-9]. However, such sludge does not reflect the reality and complications of the treatment and does not provide a true picture of the mechanism of sludge conditioning by using chemical coagulants, especially under unstable characteristic of activated sludge.

Several researchers have applied many polyelectrolytes to simulated waste [10-13], but, only a few discussed its application on real waste [14-16]. Due to the complexity and nature of variations of the wastewater composition, the flocculation optimization process in municipal waste is currently based on trial and error [7].

Thus, this project will focus and study the conditioning of real samples of activated sludge collected from Nizwa municipal wastewater treatment plant within the Sultanate of Oman. The purpose of the research is to observe the effects of coagulant agents on the unstable and complex sludge, and then optimize dosage based on turbidity, sludge volume index, flocs density and settling rate. As the characteristics of the sludge are time dependent, testing is required to be performed in an expedient manner to prevent variations in the sludge properties that might occur contributing to erroneous results. Also, the overall objective of this study is to investigate the role of other coagulants such as aluminum sulfate, ferric chloride and different types of polyelectrolytes as flocculating and conditioning agents for sewage sludge at Nizwa municipal wastewater treatment plant.
1.1. Conditioning agents

Chemical coagulants such as polyelectrolytes are very important agents for the sludge conditioning of wastewater treatment. All coagulants are used in order to reduce the electrical charge of the particles breaking the stability of the colloids, and thus affecting the flocculated microbial aggregates [3]. The main applications of polyelectrolytes in waste water treatment are in coagulation and flocculation, and in the dewatering of treatment plant sludge. Polyelectrolytes have been utilized in coagulation/flocculation processes for wastewater treatment for at least four decades [17-19]. In comparison with alum, some of the advantages from the use of polymers in water treatment are; lower coagulant dose requirements; a smaller volume of sludge; a smaller increase in the ionic load of the treated water and reduced level of aluminum in treated water [20]. Polyelectrolytes are especially beneficial in dealing with the problems of slow-settling flocs such as waste water suspensions.

In order to improve the solid-liquid separation of the sludge, conditioning agents shall be used due to the large effects of these agents on the dispersed particles. There are two types of agents; inorganic and organic chemicals. Both are used to change the electrical potential of the suspended particles and reduce the repelling force [21].

Inorganic compounds such as calcium chloride and ferric chloride are commonly used for treatment [22]. These agents form polymeric charged hydroxide and are adsorbed on the dispersed particles, and thus, modifying their surface charge.

The organic agents such as polyelectrolytes with higher molecular weight are easily attached to the suspended particles having low electrical surface charge [23]. Some examples of these polyelectrolytes are; Superfloc C498 HMW, C946 HMW, CAPAM, and ANPAM.

1.2. Sludge volume index (SVI) and settling rate

The sludge volume index (SVI) is a general indicator for measuring the bulking properties of the sludge and process efficiency. Settling is highly related to the type of microorganisms present in the wastewater, and thus, serve as a viable an indicator for bulking and foaming [5]. Slow settling rates indicate bulking with a high presence of filamentous micro-organisms [24]. A healthier and faster settling rate indicates lower level of organic contents and filamentous. Therefore, the study of the settling properties of the sludge is considered as an ideal tool for analyzing and managing the activated sludge process. The SVI refers to the milliliters occupied by gram of suspension after 30 minutes settling [25]. Value of SVI of below 200 is an indication for a health process with optimum settling rate.

1.3. Electrical conductivity

Electrical conductivity of the activated sludge is an indicator for the amount of dissolved ions such as calcium, magnesium, and sodium; it also indicates the amount of nitrate in wastewater during the aeration process [26].
2. Materials and Methods

2.1. Sludge samples

The sludge samples were collected from the aeration tank in order to obtain the optimum solid concentration in the digester. However, each test was performed using a new fresh sludge solution. Thus, it can be seen that the initial point for all tests differ from each other in reference to all variables under considerations such as pH, turbidity, conductivity, and total suspended solid (TSS) as shown in Table 1.

<table>
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<tr>
<th>Test No.</th>
<th>Chemical Added</th>
<th>pH</th>
<th>Initial Conductivity (ms/cm)</th>
<th>Final Conductivity at last dose of chemicals added (ms/cm)</th>
<th>TSS (g)</th>
<th>FSS (g)</th>
<th>VSS (g)</th>
<th>VSS/TSS (%)</th>
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<td>7.6</td>
<td>1.166</td>
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<td>3.9</td>
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<td>1.7</td>
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<td>15</td>
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<td>16</td>
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<td>17</td>
<td>FeCl₃</td>
<td>9.4</td>
<td>2.49</td>
<td>2.43</td>
<td>8</td>
<td>2.1</td>
<td>5.9</td>
<td>73.8</td>
</tr>
<tr>
<td>18</td>
<td>FeCl₃</td>
<td>5.6</td>
<td>4</td>
<td>3.3</td>
<td>8</td>
<td>2.1</td>
<td>5.9</td>
<td>73.8</td>
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2.2. Activated sludge conditioning

Six types of chemicals were used for conditioning of the activated sludge. Three different types of polyacrylamides as flocculants such as CPAM-80, CPAM-10, and APAM-30 provided by Cytec Industries Ltd., UK. The remaining three types of coagulants such as; Aluminum sulfate, Calcium chloride and Ferric chloride were provided by the University of Nizwa laboratory.

All chemicals were prepared by mixing 1 g of each chemical with one liter of deionized water and stirred with a magnetic stirrer for at least 24 hours. Ten
different range of concentrations of each chemical used from 0.05 to 15 mg of chemical per gram of total suspended solid of the activated sludge. The pH for each solution was adjusted by adding sulfuric acid or sodium hydroxide solutions.

2.3. Coagulation and flocculation test
Flocculation was carried out on a unit with six multiple stirrers a stainless steel paddle (Jar test) using a rapid mixing rate of 200 rpm for a duration of 2 minutes, and then followed by a period of a slower mixing rate of 90 rpm for 30 minutes. The combination of short and fast mixing helped the binding of the polyelectrolyte molecules and dispersed particles. The slower mixing rate was used to promote flocculation [27]. The turbidity of the supernatant liquid was measured in NTU (Nephelometric Turbidity Unit) using a turbidity meter (CL 52D NEPHELOMETER).

2.4. Settling property
The measurement of the settling properties was based on Sludge Volume Index (SVI) and was conducted by pouring one liter of well mixed activated sludge in an Imhoff cone. The position of the interface between the supernatant liquor and sludge zone was observed and recorded. Total suspended solids for each fresh sludge sample was analyzed using method 2540D in accordance with 1998 APHA standard. The settling properties of the sludge suspensions were characterized by the SVI as described by method 2710D in accordance with 1998 APHA standard [21]. The settling profile of each sample of activated sludge was determined by the height of the interface in the Imhoff cone. The SVI refers to the volume in milliliters occupied by 1 g of suspension after 30 minutes of settling.

2.5. Microscopic analysis
HORNET Micro Zoom 1280 was used for optical microscope photo of sludge samples; fresh and conditioned samples. The adopted magnification 6x-50x. The photos have been done with a digital camera MICROS CAM 320 "Advanced".

3. Results and Discussions
Several conditioning tests were carried out using fresh sludge with different polyelectrolytes and coagulants at various concentrations at different pH levels. All the results of turbidity and SVI are plotted versus mg of chemicals added per g of solid (TSS) per liter of activated sludge.

3.1. Turbidity
One of the most useful tools that indicated the effectiveness and clarified the efficiency of coagulants and flocculants materials on the sludge conditioning process was for measuring the turbidity of the solutions. Turbidity was measured for the supernatant liquor of all samples including samples with and without the addition of the polyelectrolytes. The results of turbidity with respect to the concentrations of coagulants and polyelectrolytes at different pH values are
shown in Figs. 1-3. These figures show that the effectiveness of the polyelectrolytes on the turbidity is much higher than that of the coagulants. Also, it can be observed that the APAM and calcium chloride had a negative effect on the turbidity despite the pH value, while no improvement of the settling properties was obtained using aluminum sulfate and concluded oscillatory results.

The results showed that the polyelectrolyte CPAM-80 had improved the turbidity more than other two types; CPAM-10 and APAM-30. The best turbidity was obtained for the sample with pH near neutrality as shown in Fig. 1. Also, it can be seen that the turbidity had improved immediately after the first dosage of CPAM-80 and reached 0.2 NTU at dosage of 0.35 mg CPAM-80/g solid/l. The further addition of CPAM-80 offered low turbidity level between 0–0.2 NTU. For sludge solution with pH=5.6 and pH=9.4, less improvement in turbidity were obtained compared to that under pH=7.6.

Similarly, CPAM-10 showed an improvement in turbidity especially at pH near neutrality as shown in Fig. 2. Also, it can be observed that the turbidity had improved immediately after the first dosage of CPAM-10 and reached 0.4 NTU at dosage of 1.32 mg CPAM-10/g solid/l. The further addition of CPAM-80 offered low turbidity level between 0.4 – 1.0 NTU. For sludge solution with pH=5.6 and pH=9.4, less improvement in turbidity were obtained compared to that under pH=7.6.

![Fig. 1. Effect of the CPAM-80 on turbidity at different pH.](image1)

![Fig. 2. Effect of the CPAM-10 on turbidity at different pH.](image2)

![Fig. 3. Effect of the APAM-30 on turbidity at different pH.](image3)
The results of using APAM-30 are shown in Fig. 3. A small improvement in turbidity was achieved with increase of dosage until reached the value of 1.2 NTU at dosage of 0.4 mg APAM-30/g solid/l. However, the increase of the APAM concentrations caused a sharp increase in turbidity as shown in Fig. 3 for the case of pH near neutrality. Similar result was obtained using the sample with pH=5.6. There was no improvement in turbidity at pH=9.4. It can be concluded that the effect of APAM-30 had a negative effect on turbidity compared to that of CPAM.

The use of coagulants (aluminum sulfate, calcium chloride and ferric chloride) for sludge characterization showed less effect on turbidity compared to that of using CPAM solution. The results are shown in Figs. 4–6. From these figures, it can be seen that that effect of aluminum sulfate on turbidity was much larger than that of other two coagulants; however, less improvement was obtained compared to that of the CPAM. The best turbidity results were, also, obtained for samples with pH near neutrality as shown in Fig. 4. The increase of aluminum sulfate concentration caused an increase in turbidity. Turbidity improvement for solutions with pH=5.6 and pH=9.4 had no effect. Similarly, there was minimal improvement in turbidity using ferric chloride as shown in Fig. 5. There was no improvement on turbidity with the use of calcium chloride, as shown in Fig. 6.

![Fig. 4. Effect of the Aluminum sulfate on turbidity at different pH.](image1)

![Fig. 5. Effect of Calcium Chloride on turbidity at different pH.](image2)

![Fig. 6. Effect of the Ferric Chloride on turbidity at different pH.](image3)
3.2. Settling behavior

As mentioned in Section 3.4, the optimum performance of activated sludge settling process would be achieved at SVI of 200 ml/g solid/l. However, all samples collected within the three month period at the Nizwa wastewater treatment plant had a very high SVI. These unhealthy biomass contents may be as a result of fungi, algae, worms and viruses organisms found in the wastewater feed. Sludge bulking may be due to the high organic contents in the wastewater as shown in Table 1. This Table shows that most waste water samples consisted of significant amounts of Volatile Suspended Solid (VSS). The organic contents caused settlement problems due to the formation of colloidal particles that had low settling rates because of extracellular polymer generated from organic material that had increased the amount of bound water in the sludge [6, 28]. This behavior indicates that the particles of sludge under consideration were negatively charged. The effect of high organic contents decreased the efficiency of the conditioning by polyelectrolyte, which caused an amorphous appearance, non-compact and non-dense flocs [29]. The non-compact, non-dense flocs, and non-regular particles formation are photographically illustrated in Fig. 7. Fig. 7(a) shows a sample of sludge without polyelectrolyte and Fig. 7(b) shows the positive effect of the polyelectrolyte for separating solids particles of sludge in comparison with that of fresh sludge without conditioning. These photos show the complexity of the sludge and most of the sludge particles are irregular and heterogeneous in shapes.

Fig. 7. Digital photo of fresh activated sludge diluted 50 times.

The contents of the samples regarding the total suspended solid (TSS) and volatile suspended solid (VSS) are presented in Table 1. The results indicated that there were large values of TSS and VSS in the aeration tank at the Nizwa wastewater treatment plant. The values of TSS seems to be much higher than recommended by the OECD [30], that provides official guidelines of optimum TSS contents of healthy activated sludge process. Also, the contents of the samples showed high organic matter caused in slow settling and bulking behavior. The high organic contents of most of the samples were in the average range of more than the 80 percentile.
In order to observe the effects of coagulants and polyelectrolytes on sedimentation of the fresh activated sludge, several tests were conducted using different concentrations of the chemicals at different pH values similar to that of the turbidity tests. The settling volume index was considered as an indicator for the settling properties. The results of these tests are shown in Table 2 which showed an improvement of 50% was achieved by using CPAM-80 and CPAM-10 for the samples with pH near neutrality. The lowest SVI of 1100 was achieved at dosage 2.8 mg/g solid/l of CPAM-80 and at dosage 3.3 mg/g solid/l of CPAM-10. However, there was no improvement observed for samples using APAM.

Table 2. SVI of Fresh and conditioned sludge using different concentrations of coagulant of range 0 to 12 mg coagulant/g solid.

<table>
<thead>
<tr>
<th>polyelectrolyte</th>
<th>Initial SVI</th>
<th>Final SVI</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>pH=5.6</td>
<td>pH=7.6</td>
</tr>
<tr>
<td>CPAM-80</td>
<td>3869.56</td>
<td>1831.57</td>
</tr>
<tr>
<td>CPAM-10</td>
<td>3869.56</td>
<td>1960.13</td>
</tr>
<tr>
<td>APAM-30</td>
<td>3800</td>
<td>2464.73</td>
</tr>
<tr>
<td>Aluminum Sulfate</td>
<td>3930.38</td>
<td>7755</td>
</tr>
<tr>
<td>Ferric Chloride</td>
<td>3735.3</td>
<td>4425.31</td>
</tr>
<tr>
<td>Calcium Chloride</td>
<td>3693.73</td>
<td>3600</td>
</tr>
</tbody>
</table>

The use of coagulants had a negative effect on the SVI as indicated in Table 2 revealed that the higher the concentration of coagulants the higher the SVI. The effects of pH on both turbidity and SVI on the conditioning of activated sludge displayed a negative profile in the case of using polyelectrolyte and coagulants.

3.3. Floc density

The effect of high organic content decreases the efficiency of the conditioning by polyelectrolytes causing amorphous appearance, open structure and non-dense flocs. The open structure and non-dense flocs and irregular particle formation were photographically illustrated in Figs. 8-10. In these figures, images 1N to 6N show flocs density of sludge solution at pH near neutral conditions, images 1A to 6A show flocs density at pH=5.6 and images 1B to 6B show flocs density at pH=9. With respect to these optical analyses, it can be observed that the flocs densities, compactness and sizes were increased with rise concentrations of both polyelectrolytes; CPAM-80 and CPAM-10. However, there were no changes in flocs densities and compactness using APAM-30. This indicated that the fresh activated sludge contained higher.
3.4. Electrical conductivity

Table 1 consists of the profile obtained from the conductivity measurements carried out during three months of sample collection at the Nizwa wastewater treatment plant. The profile obtained showed high variability of the electrical conductivity ranging from 1000 µs/cm to over 4000 µs/cm due to the continuous changes of the feed compositions as illustrated in Fig. 11. However, it can be seen that all polyelectrolytes and coagulants used in the tests caused a reduction in electrical conductivities of the samples, except for calcium chloride which increased the electrical conductivity, especially when pH=5.6 [31]. The largest
reduction in the electrical conductivity was observed through the use of aluminum sulfate.

![Graph of electrical conductivity over time in the aeration tank.](image)

**Fig. 11.** Electrical conductivity as function of time measured in the aeration tank.

### 3.5. Zone settling rate

The position of the interface between sediment and supernatant liquid for all the experimental tests were determined and the results are shown in Figs. 12-17 as a function of time depending on different concentrations of polyelectrolytes, coagulants, and pH. In all the tests, a decrease in the sediment thickness over time indicated that the particles had settled in flocculated form [32].

Regarding zone settling rate and final value of sediment thickness, it can be seen from Figs. 12-14 that both CPAM-80 and CPAM-10 had a larger effect on the reduction of the sediment thickness compared to the effect of all other chemicals used; APAM-30, aluminum sulfate, calcium chloride and ferric chloride at all different dose of concentrations and different pH levels. Both CPAM-80 and CPAM-10 provided almost a 50% reduction in the sediment thickness and had a larger zone of settling rate compared to others that offered a maximum reduction of 20% and slower zone of settling.

Although results of CPAM-10 proved a 50% reduction in sediment at all the three pH values compared to that of CPAM-80, results of CPAM-80 caused reduction at a lower dose range compared to that of CPAM-10. These results are in agreement with that achieved from turbidity and SVI results in which CPAM-80 had a significant effect on the characteristics of the activated sludge.

Comparing results of coagulants, see Figs. 15-17, it is clear that the results of samples treated with aluminum sulfate has better settling than the other two coagulants as it is forced particle settlement fast compared to others which took an average 20-30 minutes extra to start the sediment process with respects to pH values. Also, from these results, it can be seen that the decrease in sediment thickness was higher at smaller dosages of coagulants.
(a) Settling volume at pH=7.6.

(b) Settling volume at pH=9.4.

(c) Settling volume at pH=5.6.

Fig. 12. Sedimentation of sludge at different concentration of CPAM-80 and different pH.
(a) Settling volume at pH=7.6.

(b) Settling volume at pH=9.4.

(c) Settling volume at pH=5.6.

Fig. 13. Sedimentation of sludge at different concentration of CPAM-10 and different pH.
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Fig. 14. Sedimentation of sludge at different concentration of APAM-30 and different pH.

(a) Settling volume at pH=7.6.

(b) Settling volume at pH=9.4.

(c) Settling volume at pH=5.6.
Fig. 15. Sedimentation of sludge at different concentration of \( \text{Al}_2(\text{SO}_4)_3 \) and different pH.
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(a) Settling volume at pH=7.6.

(b) Settling volume at pH=9.4.

(c) Settling volume at pH=5.6.

Fig. 16. Sedimentation of sludge at different concentration of CaCl₂ and different pH.
(a) Settling volume at pH=7.6.

(b) Settling volume at pH=9.4.

(c) Settling volume at pH=5.6.

Fig. 17. Sedimentation of sludge at different concentration of Fe$_2$Cl$_3$ and different pH.
4. Conclusions

The following conclusion can be drawn:

- Samples of fresh activated sludge collected from the Nizwa wastewater treatment plant showed unhealthy biomass content which caused a slow settling rate and a high SVI.
- Cationic polyelectrolytes provided almost a 50% reduction in the sediment thickness and a faster settling rate compared to that of anionic polyelectrolyte which gave a maximum reduction of 20% and a significantly slower zone of settling.
- Larger floc sizes were produced using cationic polyelectrolytes compared to that obtained from using anionic polyelectrolyte and other three coagulants.
- Effects of coagulants; aluminum sulfate, calcium chloride and ferric chloride on turbidity, SVI, floc size were, in general, much less than that obtained by using polyelectrolytes.

Acknowledgement

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References


