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AN EXPERIMENTAL STUDY ON THE RHEOLOGICAL PROPERTIES OF CONDITIONED MUNICIPAL ACTIVATED SLUDGE

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Abstract

This research work was focused on the rheological characteristics of conditioned fresh activated sludge using TA rheometer HR-2. The effect of cationic polyelectrolyte conditioner has been investigated for floc size, surface properties and yield stress at different pH values in a comparative fashion. Our approach was to reveal the effect of polymer on the municipal activated sludge with high organic contents up to 80%. The results indicated an improvement of 50% in settling properties by addition polyelectrolyte up to 4 mg/g solid/l. Rheological data analysis showed that responses of shear stress - shear rate were found to be closest to Bingham model and gave almost similar and smaller k values of average 6.2×10^{-3}. The results of shear creep indicated that all sludge samples have less rigid structures with no reconstruction behavior. The optical analyses of the samples indicated that the floc sizes were increased with successive addition of polyelectrolyte. The increase of floc sizes caused large stresses especially for solution with pH=9. As the flocculation accorded despite the negative zeta potential, this phenomenon can be referred to that inter-particles hydrogen bridging was governing flocculation rather than charge neutralization. Also, during the experiment, bacterial growth showed an adaption despite the conditioning with polyelectrolyte.

Keywords: Municipal sludge, Floc size, Rheology, Polyelectrolyte.

1. Introduction

Sludge is a multi-component mixture with high water content, organic, micro-organism and colloids. It is a very unstable product and depending on its origin. The treatment and management of waste activated sludge, especially its conditioning and processing, have been described as the most difficult and costly processes within the wastewater treatment plants.

The limitation of the current operation technology utilized in wastewater treatment plants requires the development of more efficient and optimized methods

Nomenclatures

k	Consistency index
n	Flow behavior index

Greek Symbols

τ	Shear stress, N/m^2
γ	Shear rate, , N/m^2

Abbreviations

CFU	Colony Forming Units
CPAM-80	Cationic Polyacrylamide
FSS	Fixed Suspended Solid
NTU	Nephelometric Turbidity Unit
SVI	Sludge Volume Index
TSS	Total Suspended Solid
VSS	Volatile Suspended Solid

for sludge management and disposal, which accounts for about 50% of the operating costs and 20-30% of the capital costs of wastewater treatment plants. In order to improve the settling and dewatering in wastewater treatment plant, sludge conditioning with polyelectrolytes have been proposed by Roult et al. [1] and Nguyen et al. [2]. The effect of conditioning on the rheological behavior of the activated sludge plays an important role in cost effective of equipment design, sizing, selection, and transportation as indicated by many researchers [3-8].

The main applications of the polyelectrolytes in wastewater treatment plants are in coagulation and flocculation processes and its role to enhance the solid-liquid separation and to reduce water content of the settled sludge. Many types of Polyelectrolytes have been utilized in coagulation and flocculation processes for wastewater treatment for at least four decades [9-11].

In sludge conditioning processes, polyelectrolyte is usually added to pre-treat sludge solution to increase its settling ability and the corresponding filterability. Sludge conditioning may cause sludge particles charge neutralization and inter-particle bridging and both are the two major mechanisms in flocculating the constituting particles into larger flocs. When the charge neutralization is dominating, a strong correlation may exist between the polyelectrolyte dose at which the sludge particles surface charges had neutralized leading to the maximum settling velocity and a minimum resistance to filtration [12]. When the inter-particle bridging controls the flocculation process, no such correlation would exist.

The selection of the type of coagulant and the determination of their optimum dosages are still based on laboratory level and depending on the operator's experience. This traditional procedure would lead to the polyelectrolyte over dosage and hence a high operational cost coupled with contaminated effluent. The understanding of conditioning process would provide a good guide in development and improved operation of the wastewater treatment plant [6].

Conditioning agents affect coagulation/flocculation processes and physical characteristics of flocs such as floc density, size and shape, strength and surface properties [13]. The flocs size and structure are an essential for floc removal efficiency and in the operation of the wastewater treatment plant [14]. For example, during settlement of low turbidity sludge solution, large compact flocs would have a high settling rate, but, large and porous flocs would help in filtration process due to their high permeability [15].

The surface properties of dispersed sludge particles in the solutions of activated sludge affect coagulation and flocculation processes. The surface charges of the sludge particles have direct effect on sludge natural flocculation properties. As a result of bio-chemical and physic-chemical interactions between inorganic components and bacteria present in wastewater, activated sludge particles are negatively charged [16]. The surface charges of the sludge particles influence the rheology and stability of many colloids. These particles charges can be modified by changing pH or by changing the ionic species of the sludge solution by using polyelectrolytes that adsorbed on the particles surfaces changing their charge characteristics [17]. The result of these measurements is zeta potential to measure an electrophoretic mobility of the solution. The high values of zeta potential prevent flocculation process, on the other hand low values of zeta potential would allow the particles to approach each other and flocculate. Faster flocculation occurs when zeta potential values approach zero. This mechanism would be interpreted by an attraction force termed Van der Waals attraction [18].

Sludge particles with high negative or positive zeta potential are electrically stabilized while sludge particles with low zeta values tend to flocculate. The addition of polyelectrolyte is used to reduce the energy barriers between sludge particles leading to efficient settling and agglomeration [19]. The improvement of separation process in wastewater treatment can be performed by controlling the sludge properties as suggested by Bennoit and Schuster [20].

This study intended to understand the basic rheological characteristics of the conditioned activated sludge using cationic polyacrylamide (CPAM-80) as a conditioning agent. Samples from Nizwa municipal sludge has been selected, this possessing a high organic content and continuously unstable composition [21]. Other objectives were to determine the particles surface charges of the treated sludge samples. This provides fundamental and comprehensive information on the relationship between flocs structures and strength. Bacteria present in the solution of the conditioned sludge were examined. The rheological behavior of the sludge samples; viscosity, shear stress-shear rate and yield were investigated.

2. Rheological Theory

Due to the non-uniform of sludge operation conditions, wastewater shows different sludge rheological characteristics. Many researchers are stated that wastewater activated sludge has a non- Newtonian fluid behavior and possesses both viscous and elastic properties [22-24]. Mostly, activated sludge is rheologically characterized using pseudo-plastic or Bingham plastic models [22, 25]. But, the behavior of thickened sewage sludge has been considered as thixotropic and modeled using Herschel-Bulkly model [26].

Rheological properties of the activated sludge are very important design criteria for wastewater plant in respect to pumping, storage, operation procedure of controlling stabilization and dewatering and sludge management [27]. The most important rheological parameters are yield point, viscosity, and solid concentration [4]. Due to the non-uniformity sludge operation conditions, wastewater shows different rheological characteristics especially in empirical correlation [28]. According to Lee et al. [29], conditioned sludge shifts its rheological properties from viscoelastic liquid-like to solid-like behavior.

Yield stress is defined as the minimum shear stress required for initiating the flow of sludge solution [29]. An elastic solid behavior sludge sample would be shown during rheological tests of shear stress ramp measurements; this act forces the viscosity to increase as more stress is applied. At point of reaching yield stress, sludge sample starts to flow and hence the viscosity starts falling accordingly [30]. Measurement of the yield stress provides valuable information for the sludge handling and transportation ability of the filter and thickener output as a function of the solid concentration of flocculated sludge. There are many ways to evaluate the yield stress for fluid-like substances and no single "best" technique can be defined [31].

Yield stress of fluid-like material is the most popular technique for fluid viscoelasticity measurement constructed from the extrapolation of shear stress versus shear rate. Most commonly applied models are [3, 28]; Bingham Plastic Model, Eq. (1) that assumes a linear relationship between shear stress and shear rate; Herschel-Buckley Model, Eq. (2) and Casson Model, Eq. (3) are designed to determine the yield stress of non-linear plastic behavior; Ostwald de Waele (power law) Model, Eq. (4) is developed for a purely shear thinning depending if the flow model is considered to start from zero stress; and Sisko model, Eq. (5) which is designed for high-shear rate that containing infinite-shear-viscosity.

$$\tau = \tau_o + k\gamma \tag{1}$$

$$\tau = \tau_o + k\gamma^n \tag{2}$$

$$\tau^{1/2} = \tau_o^{1/2} + (k\gamma)^{1/2} \tag{3}$$

$$\tau = k\gamma^n \tag{4}$$

$$\tau = \mu_B\gamma + k\gamma^n \tag{5}$$

where: τ is the shear stress, γ (s^{-1}) is the shear rate, k is the consistency index represents the cohesiveness of the solution, the higher value reflect higher viscosities, n is the flow behavior index (n = 1 is for Newtonian fluids and value far from 1 indicates deviation from Newtonian behavior), τ_o, indicates the resistance of the sludge to the deformation until sufficient stress is applied, and μ_B is the high shear limiting viscosity where the shear rate imposed on the fluid tends to an infinite value [3].

Sludge creep happens on what is called biological sludge which is difficult to dewater. In the creep test, an instantaneous stress is applied to the sludge sample and the change in the strain (called the creep) is observed over time. When the stress is released, some recovery of the properties may be observed as the material attempts to return to the original shape [4, 5, 32, 33]. For example, creep testing is the polymer relaxations and a fundamental form of polymer behavior.

3. Materials and Methods

Many sludge samples were collected and conditioned with polyacrylamide CPAM-80 and characterized using TA-Rheometer.

3.1. Sludge sample

The sludge samples were collected from the aeration tank of Nizwa city's municipal wastewater treatment plant in the Sultanate of Oman. The average values of each characterization parameter of the collected sludge sample are presented in Table 1. These values indicated that most of the samples had an organic content above 75%.

Table 1. characterization parameters of the collected sludge sample.

	Value
Temperature (^oC)	22
pH	6.8
Conductivity (ms/cm)	2.67
Total suspended solid - TSS (g/l)	7.31
Volatile suspended solid - VSS (g/l)	5.51
Fixed suspended solid - FSS (g/l)	1.8

Polyacrylamide CPAM-80 used for sludge conditioning was provided by Cytec industries Ltd. Conditioning solution was prepared by mixing 1 g of CPAM-80 with one liter of deionized water, then stirred using magnetic stirrer for at least 24 hours. Six different concentrations of conditioning solution were used to cover range from 0.5 to about 14 mg of polyelectrolyte per one gram of total suspended solid. The process of sludge conditioning was performed at three levels of pH viz.; pH =5.6, pH=6.8 and pH=9. pH were adjusted using NaOH and H_2SO_4.

In order to determine the type and growth of bacteria present in the activated sludge, samples were analyzed for colony forming units (CFU) in the microbiology lab at the University of Nizwa. Firstly, a serial of dilutions from the sample (10^{-1}, 10^{-2}, 10^{-3}) was prepared followed by incubation for 24 hours. After incubation period, the CFU was calculated using the following formula:

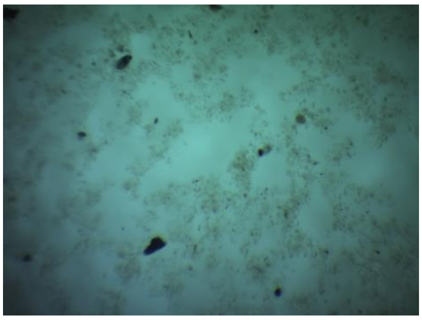
cfu/mL=(#colonies)*(dilution factor)/volume taken in mL

3.2. Microscope analysis

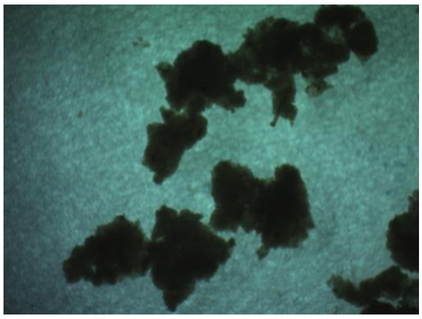
HORNET Micro Zoom 1280 supplied by Micros Austria company, was used for optical microscope photo of sludge samples; fresh and conditioned samples. The adopted magnifications were between 6× - 50×.The imaging was done using a digital camera MICROS CAM 320 "Advanced".

Sample photos of conditioned and non-conditioned sludge are presented in Fig. 1. It can be observed that the fresh sludge sample was almost homogeneous while the sludge sample conditioned with polyelectrolyte was non-homogeneous due to separation of solid particles. Also, these photos showed the complexity of the sludge and most of the sludge particles are heterogeneous in shapes.

Images were analyzed for determining flocs sizes and shapes using MICROVISIBLE image analyzer software combined with HORNET microscope. As the sludge particles has irregular shape, the area of the particles were evaluated based on their perimeters [34].



(a) Non-conditioned sludge.



(b) Conditioned sludge.

Fig. 1. Image of conditioned and non-conditioned sludge samples diluted 50 times with water. Magnification: 30x.

3.3. Rheological test

TA-Rheometer type HR-2 Discovery Hybrid was used for Rheological measurements of sludge samples. Peltite plate geometry was selected which is suitable for low viscosity liquids. During all rheological tests, no water was rejected even when imposing a high shear rate. The procedure was started by selecting geometry and choosing the DIN concentric cylinder and then set the system to the zero gap. Then, sludge sample was loaded into the geometry. All tests conditions work were selected as: sample intervals 10 point per decades; range of shear rate 0-1200 1/s; temperature $25^{o}C$ [35].

3.4. Coagulation and flocculation test

Flocculation was carried out on a unit of 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 allowed the binding between the polyelectrolyte molecules and dispersed sludge particles. The slower mixing rate helped to promote flocculation [11, 36]. The turbidity of the supernatant liquid was measured in NTU (Nephelometric Turbidity Unit) using a turbidity meter (CL 52D NEPHELOMETER).

3.5. Zeta potential

The type and strength of the surface electrical charges of the particles of the activated sludge were measured using Zetasizer model nano Z supplied by

Malvern instruments, UK. The zeta analysis was performed three times with each sample and average value was calculated.

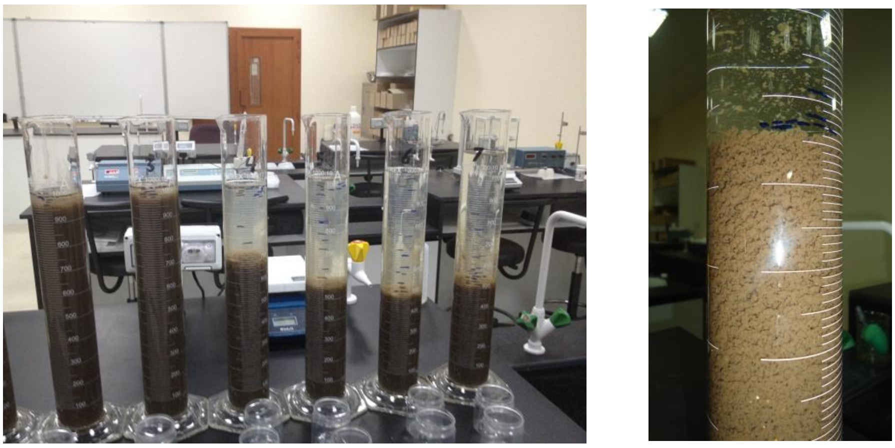
3.6. Settling property

Settling properties of sludge samples were characterized using Sludge Volume Index (SVI), which was prepared by pouring one liter of each sludge materials in 1 liter cylindrical glass tube. The position of the interface between the supernatant liquid and settled sludge was observed and recorded. Total suspended solids for each 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 [37]. The effect of conditioning on the wastewater sludge with respect to settling and turbidity for one set of experiment as illustrated in Fig. 2.

4. Results and Discussion

The addition of the polyelectrolyte to the wastewater sludge was found to have a good impact on its turbidity and settling properties as pictured in Fig. 2.

The results of turbidity and zeta potential measurements at different concentrations of polyelectrolytes and different acidities were presented in Fig. 3. The results showed a sudden improvement in turbidity immediately after the first dosage of polyelectrolyte and kept the turbidity at low values even with successive addition of the polyelectrolyte. This settling improvement in fresh activated sludge can be referred to the presence of filamentous microorganisms coupled with high organic contents; this has an effect on the bridging networks and provided a backbone for flocs build up with the polyelectrolyte assistance. These findings agreed with that reported by Nguyen et al. [2].



(a) Settling. **(b) Less compressed sludge.**

Fig. 2. Picture of one set of experiment using different concentration of polyelectrolytes.

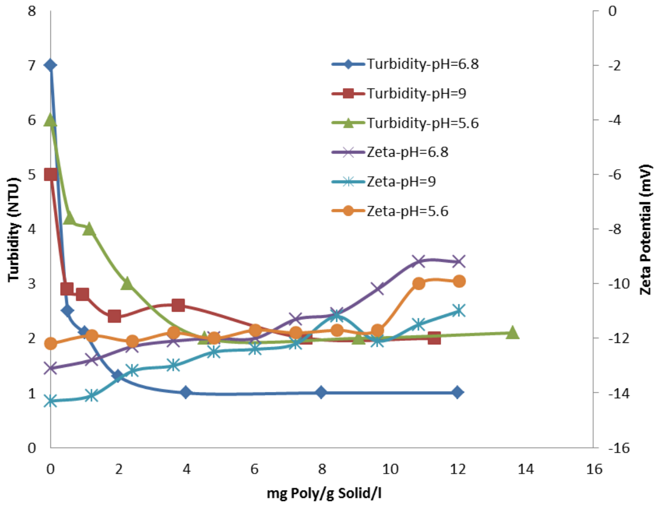


Fig. 3. Turbidity and zeta potential at different coagulant concentrations and pH.

The results revealed that setting properties and turbidity were improved despite the negative measurement of zeta potential. Moreover, the results indicated that the optimum conditioning did not necessarily happen at zero zeta potential. This behavior explained that the dominant mechanism of flocculation into larger flocs was governed by the interparticles bridging rather than by the charge neutralization. This bridge mechanism produced a less compressed sludge as shown in Fig. 2(b). The reason for the weaker flocs strength refers to the strong backbone structures of the flocs created by filamentous microorganism present in the fresh activated sludge. These findings are in accordance with the findings of Lee et al., [29]. In addition, there is no much effect of the acidity on the zeta potential measurements. However, higher zeta potential was obtained with solution at neutral pH.

All samples collected from Nizwa wastewater treatment plant had a high SVI as shown in Fig. 4. The high SVI was caused by filamentous and high organic contents which caused a significant settling problem due to the formation of colloidal particles that have low settling rate and due to the extracellular polymer generated from organic material, which increased the amount of bound water in the sludge [38-40]. However, the results indicated that setting properties improved by almost 50% by the addition of polyelectrolyte up to 4 mg/ g TSS, and no improvement was observed with successive addition of polymers. This improvement in SVI can be considered as a good indicator for sludge dewatering as stated by Nguyen et al. [2].

The effect of polyelectrolyte concentrations on the floc size is shown in Fig. 5. The results showed that floc average diameters were strongly dependent on the coagulant concentration despite the negative measurements of zeta potential of the sludge particles. For higher coagulant concentration, larger flocs were formed. These results supported the results obtained from sludge turbidity tests confirming that the colloidal particles were joined together in larger flocs. It can be observed that the floc sizes with higher alkalinity conditions (i.e., pH=9), were larger than those with pH=5.6 and 6.8 and produced higher yield stress as shown in the rheology results. This may be attributed to the hydration forces acting on the flocs [41].

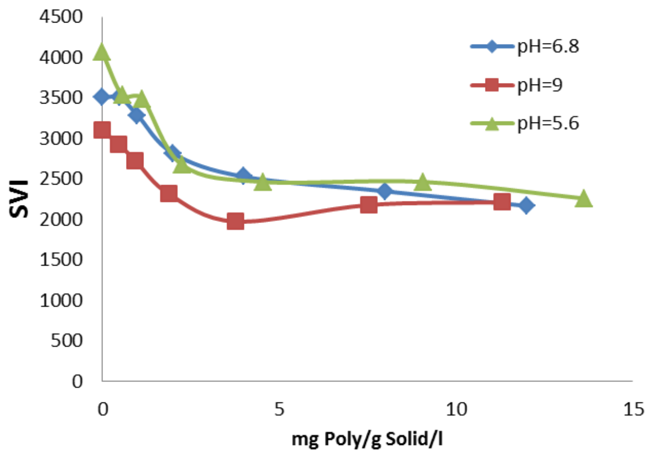


Fig. 4. SVI at different coagulant concentrations and different pH values.

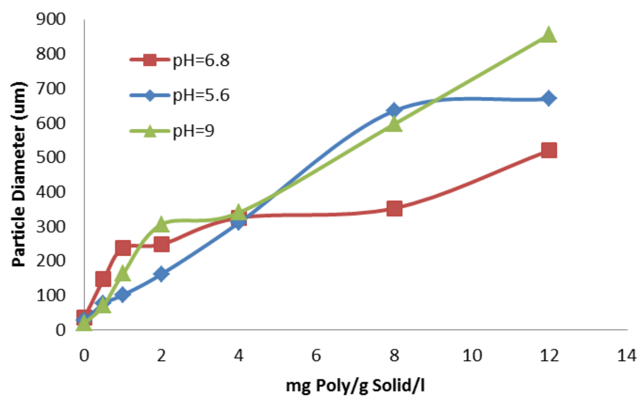
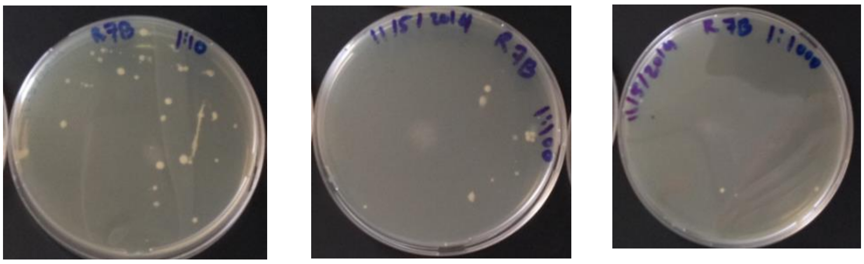


Fig. 5. Sludge particle sizes at different polyelectrolyte concentrations and pH.

Bacterial growth was monitored during the experimental work and pictured as shown in Fig. 6. Figure 6(a) shows large bacterial contents within the freshly collected sludge material that was brought from conditioning tank at Nizwa wastewater plant. Figure 6(b) shows that the bacteria adapted themselves to the new conditions (acid, base and neutral) and kept growing despite the concentrations of polyelectrolyte added. For all experiment, the CFU values were ranging between 43-75×10^3. The analyzed samples showed that the Gram positive Bacillus species were present.

(a) Fresh activated sludge.



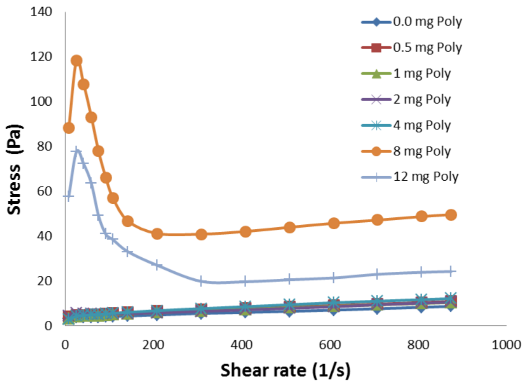
(b) Conditioned activated sludge.

Fig. 6. Image of bacterial growth during experiment.

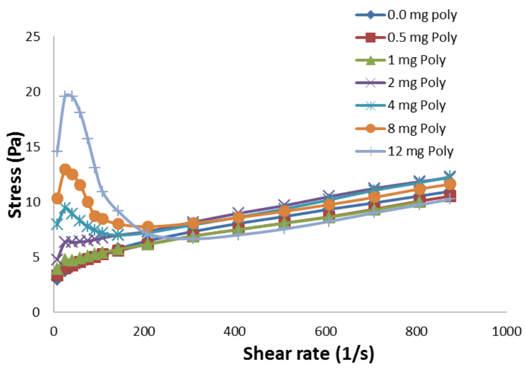
In order to analyze the rheological behavior of sludge with and without conditioning and at different pH values, typical test, the ramp shear stress was conducted and the results are shown in Fig. 7. The non-Newtonian behaviors were clearly shown. It can be seen that increasing the concentration of polyelectrolyte caused strong bonds between particles and water and resulting aggregation of particles which required a higher shear stress in order to flow. The formation of these aggregates with certain strength of bonding causes stress overshoot due to the broken structure. The result showed that the larger concentration of the polyelectrolyte produced a higher overshoot size.

Also, it can be seen that the higher the concentration of polyelectrolyte the larger the yield stresses were obtained as shown in Fig. 8 due to increased floc strength resulting from bridging formation. In addition, solutions with higher pH value caused larger yield stress. After this disruption, the effect of the polyelectrolyte vanished and shear stress rapidly decreased and when reaching shear rate of 150 1/s, all samples behaved like samples without conditioning and showed the same trend.

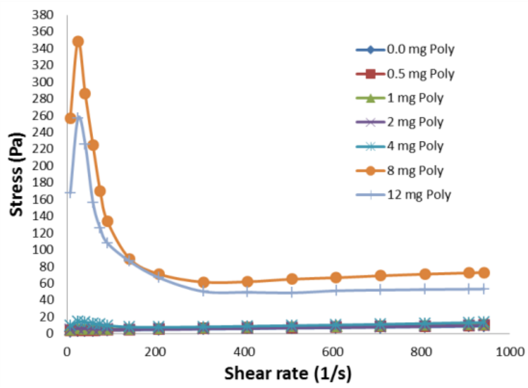
All samples of sludge under stress followed Bingham model. For each test, k values were calculated at different pH values as shown in Table 2. However, the differences between these k values were small, but it can be seen that the higher the pH the smaller the k value.



(a) Shear at pH= 6.8.



(b) Shear at pH= 5.6.



(c) Shear at pH= 9.

Fig.7. Shear stress vs. shear rate at different concentration of polyelectrolyte.

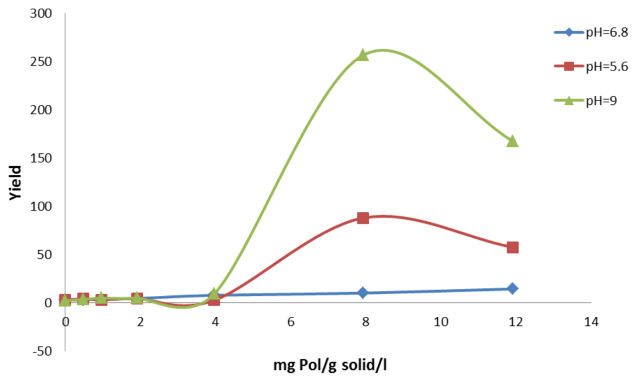


Fig. 8. Yield at different concentration of coagulant and pH value.

Table 2. k values for Bingham model.

	pH
$\boldsymbol{k = 6.9\times10^{-3}}$	6.8
$\boldsymbol{k = 6.2\times10^{-3}}$	5.6
$\boldsymbol{k = 5.5\times10^{-3}}$	9.0

Shear stress of a single shear rate was studied in order to understand shear stress-shear rate relation at different concentration of polyelectrolyte added. For stress analysis, the shear rate value of 300 s^{-1} was selected, and the results were shown in Fig. 9. The results indicated that the addition of polyelectrolyte up to 4 mg/ g TSS has no much effect on shear stress of all samples despite the formation of large flocs sizes. The reason of no change in yield stress of conditioned samples is due to lower floc strength and weaker interparticles forces. Further increase in polyelectrolyte concentration caused an increase in shear stress for samples with acidic and basic conditions only and there was no variation found in that with pH=6.8. Sludge with pH=9 showed a higher stress, this result confirmed that with an increase in concentration of coagulant, floc strength was increased especially for sludge with high alkalinity.

The effect of SVI on the shear stress at a shear rate of 300 s^{-1} is presented in Table 3. The results indicated that the low SVI had a large effect on the shear stress especially at higher concentration of the polyelectrolyte.

Creep test was performed and the results were presented in Fig. 10. A higher strain % value was observed at the higher pH value, which confirms the high floc strength of alkalinity conditioned sludge. The results of creep test indicated continuous changes in the behavior of all samples regardless of the effect of the addition of polyelectrolyte. The results show that the creep started steadily during hardening of sample materials and thus caused reduction in reducing flow rate. After overcoming the hardening period, the stain% increased rapidly following power law. The power index was higher than one indicating that shear rate was increasing with time as there was no steady state. The experiment proved that the sludge samples did not have restructuring behavior.

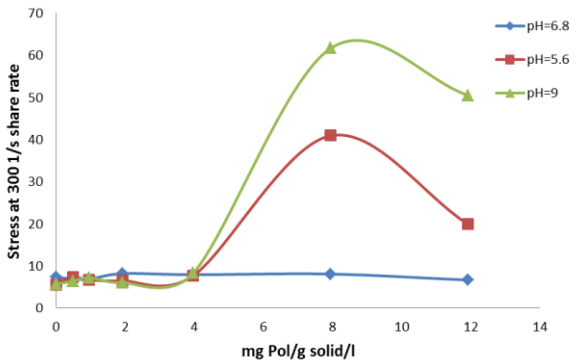
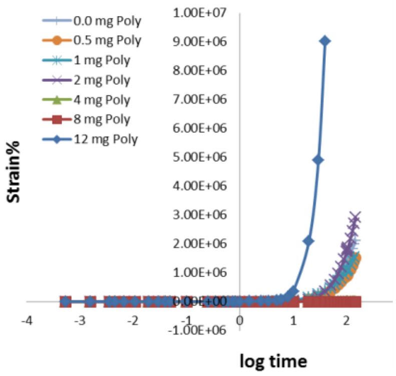
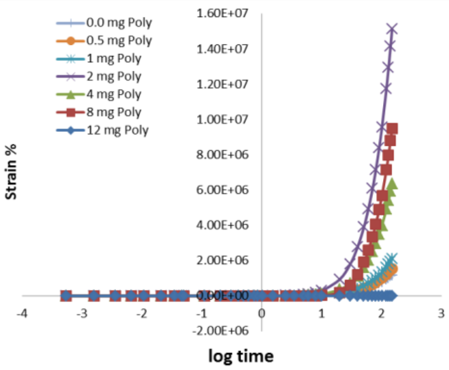


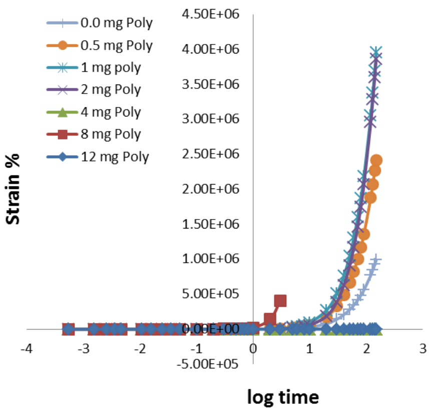
Fig. 9. Shear stress at 300 s^{-1} shear rate at different poly concentration and pH.



(a) Creep test at pH=6.8.



(b) Creep test at pH=5.6.



(c) Creep test at pH=9.

Fig. 10. Creep experiment at different concentrations of coagulant (mg poly/g solid/l) and pH.

Table 3. SVI versus shear stress at 300 (s^{-1}) shear rate at different pH.

	pH=6.8		pH=5.6		pH=9.0	
Polyelectrolyte (mg /g solid/l)	**SVI**	**Shear stress**	**SVI**	**Shear stress**	**SVI**	**Shear stress**
0.0	3502.2	7.337	4063.4	5.579	3096.6	5.72
0.5	3502.2	6.928	3529.9	7.333	2926.4	6.388
1	3285.6	6.905	3283.6	6.723	2722.3	7.194
2	2816.2	8.192	2667.9	6.507	2313.9	6.08
4	2527.4	7.9	2462.7	7.72	1973.6	8.318
8	2346.8	8.091	2462.7	40.921	2177.8	61.662
12	2130.2	6.669	2216.4	19.894	2211.9	50.495

5. Conclusions

Some concluding observations from the investigation are given below.

- The following conclusions can be arrived at from experimental results and their analysis:
- Samples of fresh activated sludge collected from the Nizwa wastewater treatment plant exhibited high organic contents and showed unhealthy biomass content which caused higher SVI.
- Addition of polyelectrolyte up to 4 mg/ g TSS reduced turbidity improved settling by 50%. This improvement in SVI can be considered as a good indicator for sludge dewatering.
- Low turbidities and large floc sizes were produced at lower concentration of cationic polyelectrolyte despite the negative measurements of zeta potential, and thus, optimum coagulant dose can be achieved without the need of reaching the isoelectric point.
- The results indicated that the addition of polyelectrolyte up to 4 mg/g TSS has no much effect on shear stress of all samples despite the formation of large flocs sizes due to lower floc strength and weaker interparticles forces.
- Creep test indicated that the sludge samples have no reconstruction behaviors.

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