Chemical Engineering Journal 166 (2011) 529–535

Contents lists available at ScienceDirect

Chemical Engineering Journal

journal homepage: www.elsevier.com/locate/cej

Treatment of pulp and paper mill wastewater with various molecular weight of polyDADMAC induced flocculation

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ARTICLE INFO

Article history:
Received 1 September 2010
Received in revised form 2 November 2010
Accepted 2 November 2010

Keywords:
Flocculation
PolyDADMAC
Polyelectrolyte
Zeta potential

ABSTRACT

The flocculation performances of different molecular weight of polydiallyldimethylammonium chloride (polyDADMAC) in the treatment of pulp and paper mill wastewater were studied. The molecular weights used were 8.5×10^4, 8.8×10^4, 10.5×10^4 and 15.7×10^4 g/mol. The flocculation performance test was carried out in jar tests with polyDADMAC dosages ranging from 0.4 to 2.0 mg L^{-1}. The test was conducted with rapid mixing at 200 rpm for 2 min, followed by slow mixing at 40 rpm for 10 min and a settling time of 5 min. The effectiveness of the flocculation was measured based on the reduction of the turbidity, the total suspended solids (TSSs), chemical on demand (COD) reduction and zeta potential measurements. It was found that the flocculation performance of higher molecular weight samples was more efficient compared with that of lower molecular weights. This might be due to the bridging mechanism, which occurred concurrently with the charge neutralization effect during flocculation.

1. Introduction

The rapid increase in human population will lead to an increase in the demand for industrial establishments to meet human requirements. This phenomenon has created such problems as the overexploitation of available resources, leading to pollution of land, air and water environments [1]. All of these pollutants basically come from waste, which has been defined as a moveable object with no direct use that is discarded permanently. This definition of waste includes solid, liquid, gaseous, hazardous, radioactive, and medical waste [2]. One of the major industries utilizing huge amounts of lignocellulosic materials and water during the manufacturing process is the pulp and paper mill industry.

This industry can consume as high as 250–300 m^3 of freshwater per metric ton of paper produced [3]. This industry generates a considerable amount of pollutants, which can be characterized by the biochemical oxygen demand (BOD), the chemical oxygen demand (COD), the suspended solids (SSs), the toxicity and the color when untreated or poorly treated effluents are discharged to receiving waters [4]. All these criteria describe conditions that are toxic to aquatic organisms and that exhibit strong mutagenic effects and physiological impairment.

To investigate this problem, many studies have been conducted involving biological methods and chemical coagulation. For the biological approach, conventional aerobic and anaerobic treatment has been used [5–9]. For the chemical coagulation approach, alum, ferric chloride and lime have been studied extensively [10–12]. All of the methods mentioned above have their respective weaknesses and limitations.

Recently, the use of flocculants, such as synthetic polyelectrolytes, for the removal of suspended solids in wastewater treatment has grown rapidly [13,14]. This increase in use is due to their ability to produce a higher sedimentation rate, better final water quality, a lower sludge volume, and better sludge quality compared with those values obtained by mineral coagulation [15]. One of the synthetic polyelectrolytes extensively used in industry is polydiallyldimethylammonium chloride (polyDADMAC). polyDADMAC belongs to an ionic group because of the positive charge (cation) in its structure. polyDADMAC molecules have a backbone of cyclic units and a charged quaternary ammonium group found in each chain unit, as shown in Fig. 1. It is also a high-charge-density cationic polymer, which makes it well-suited for the flocculation process.

Many publications have addressed the flocculation behavior of polyDADMAC in simulated waste [17–21] but few have discussed its application in actual industrial waste. Leiviskä et al. [22] have used polyDADMAC in their research on the coagulation of wood extractives in chemical pulp bleaching filtrate.

The characteristics of pulp and paper mill effluent depends upon the type of manufacturing process adopted and the extent of water recycling employed in the plant. These variables make it complicated to design treatments for individual mills [23]. Currently, the flocculation optimization practices in the industry are still reliant, to a very large extent, on trial and error because of the highly

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1385-8947/$ – see front matter
doi:10.1016/j.cej.2010.11.011

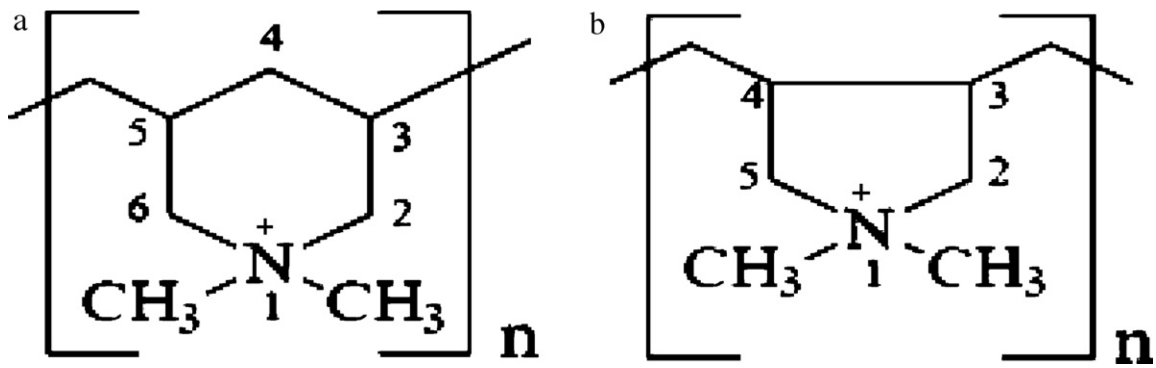

Fig. 1. Structure of polyDADMAC [16].

complex nature of the flocculation process and the large variety of polyelectrolytes available [24]. One of the ways to optimize the flocculation process is by selecting or controlling the range of the molecular weight of the polymer. Different molecular weights produce different flocculation mechanisms—neutralization or bridging [25]. A better understanding of the effect of the molecular weight on the flocculation performance may lead to improved water treatment processes and better choices for flocculants in specific industrial applications.

The main objective of the present study is to investigate the flocculation efficiencies of different molecular weights of polyDADMAC in the wastewater treatment plant of Nibong Tebal Paper Mills (NTPM). The turbidity, total suspended solids (TSSs) and chemical oxygen demand (COD) concentrations were used as the evaluating parameters. The ζ potential of the supernatant for each pulp and paper mill wastewater sample after it was treated was also investigated.

2. Experimental

2.1. Materials

The polyDADMAC flocculants used in this study were self-synthesized with various monomer concentrations. The molecular weights were obtained by synthesizing various monomer concentrations, as shown in Table 1. Distilled water was used to prepare all the polyDADMAC feedstock solutions of 1%. Wastewater was collected from the wastewater treatment plant equalization tank of a paper mill in Penang, Malaysia, in accordance with ASTM E 300-03. This factory produced about 3000 metric tons of tissue paper a month, and the wastewater produced by the plant was 96 m^3 per ton of paper produced. The characteristics of the wastewater collected from the factory are shown in Table 2.

2.2. Experimental procedure

A jar test was performed with the conventional jar apparatus (Velp Scientifica FC6S model) using 500 ml wastewater samples with polyDADMAC dosages of 0.4, 0.8, 1.2, 1.6 and 2.0 mg L^{-1}. The selected dosage was added to 500 ml of wastewater and stirred for a period of 2 min at 200 rpm. This was followed by further slow mixing for 10 min at 40 rpm. The flocs formed were allowed to settle for 5 min. After settling, the turbidity, TSSs, Zeta potential and COD of the supernatant were determined. The experiments were repeated several times to obtain an average value.

2.3. Analytical techniques

COD was evaluated using COD vials (Hach, United States) with different sensitivity ranges. Sample digestion was performed in a DRB200 reactor (Hach) over 2 h at 150 °C. Sample digestion was cool at room temperature before measured by DRB200 Digital Reactor, 15-Wells (Hach). The turbidity before filtration was measured with a turbidity meter (from Lovibond). A pH meter (CyberScan model, Eutech Instruments, Singapore) was used to measure the pH of the solution. The TSS concentration was determined by filtering a well-mixed sample through a glass fiber filter (GA 55, Advantec, Japan), and the residue retained on the filter was dried in the oven at 103 °C for 60 min prior to weighing. The ζ potential was determined with a Malvern Mastersizer 2000.

3. Results and discussion

The turbidity reduction of the wastewater after being treated with different M_ws of polyDADMAC is shown in Fig. 2 as a function of dosages. It shows that the turbidity reduction increases with increasing dosages of polyDADMAC as a function of the M_w. However, beyond 1.2 mg L^{-1}, the turbidity reduction starts to decrease. The polyDADMAC coated the suspended waste particles and neutralized their charge by Van der Waals forces [26]. This neutralization allows particles to come close together and results in agglomeration. This agglomeration reduces the turbidity of the waste particles. The decrease in the turbidity reduction after 1.2 mg L^{-1} was due to the reversal of the particle charge. After complete neutralization, other chains of polyDADMAC will attach or adsorb onto the neutralized particles. These attached chains carry N^+, causing the particles to become positively charged and thus restabilized [27].

This creates repulsion between particles, thus causing the turbidity to increase again. Similar results were also observed by Cheng et al. [28] in their study on the removal organic sub-

Table 1
Molecular weight of polyDADMAC samples.

Prepared samples	Molecular weight (g/mol)
PDM01	8.5×10^4
PDM02	8.8×10^4
PDM03	10.5×10^4
PDM04	15.7×10^4

Table 2
The pulp and paper mills wastewater characteristic.

Parameters	Value
pH	7 ± 0.5
Total chemical oxygen demand (mg L^{-1})	2900 ± 90
Turbidity	4585 ± 30
Suspended solid (mg L^{-1})	6000 ± 50
Zeta potential (mV)	-18 ± 1

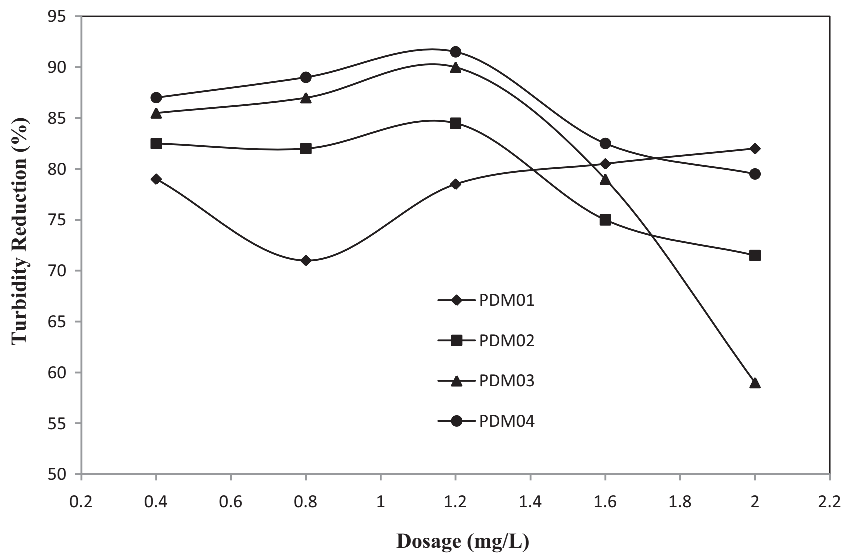

Fig. 2. Turbidity reduction of wastewater after treatment with different M_w polyDADMAC at various dosages.

stances using polyaluminum chloride. For PDM01 with a low M_w of 8.5×10^4 g/mol, the turbidity reduction increased constantly with increasing polyDADMAC dosages. This trend might be due to the nonexistence of surplus charges, which contributes to charge reversal. The low molecular weight of polyDADMAC will give a lower N^+ positive charge. The probability for charge reversal to occur is lower for low molecular weights compared with high M_ws at the same dosages. In another publication, Das et al. [29] recorded that after complete neutralization, higher molecular weights could not flocculate alumina compared with low molecular weights at the same dosages. This figure also shows that the optimal dosing of polyDADMAC for samples PDM02, PDM03 and PDM04 with molecular weights of 8.8×10^4, 10.5×10^4 and 15.7×10^4 g/mol was 1.2 mg L^{-1} because this dosage shows the highest level of flocculation [30].

The TSSs removed from pulp and paper mill waste after treatment with different M_ws of polyDADMAC at various dosages is shown in Fig. 3. The optimum dosage of polyDADMAC for the removal of TSSs is 1.2 mg L^{-1} for PDM02, PDM03 and PDM04 and 1.6 mg L^{-1} for PDM01. Generally, the TSS value reflects the turbidity at which it will decrease when flocs are produced; this contributes to the increase of the weight of the suspended solids [31].

According to Earhart [32], there is a linear relationship between the amount of suspended material and the amount of light scattered. Many particles in suspension will produce more light scattering, thus making the value of the total suspended solids low. Larger and more numerous flocs contribute to a higher concentration of suspended solids that can be removed [33]. This contribution to both results is almost the same. Sample PDM04 yielded the highest TSS removal. This might be due to the bridging mechanism that occurred concurrently with the charge neutralization mechanism. High molecular weight polyDADMAC will produce longer chains, and the tendency for loops and tails to form in these chains is higher. After neutralization, these loops and tails on the adsorbed polymer structure on one particle protrude into the solution and can attach to a second particle; this results in bridging between the first and second particles. This bridging mechanism makes the total suspended solids higher compared with other low M_w polymers [34].

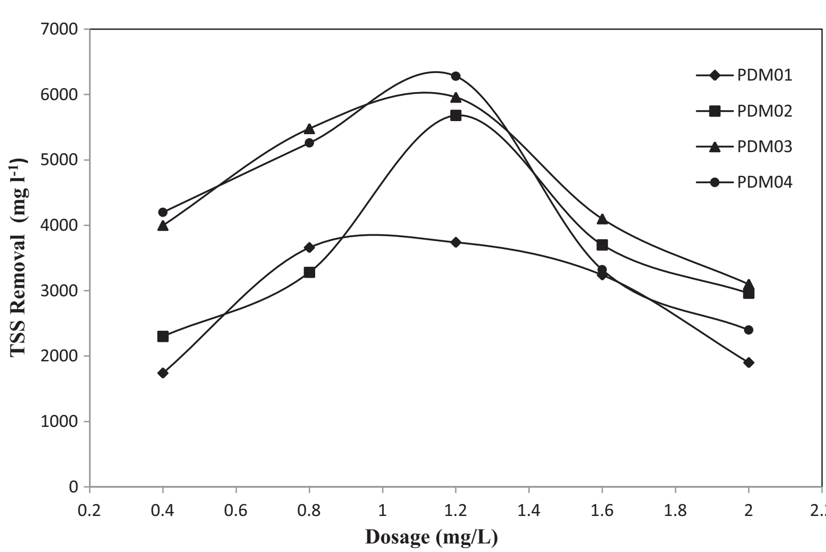

Fig. 3. Total Suspended Solid of wastewater after treated with different M_w polyDADMAC at various dosages.

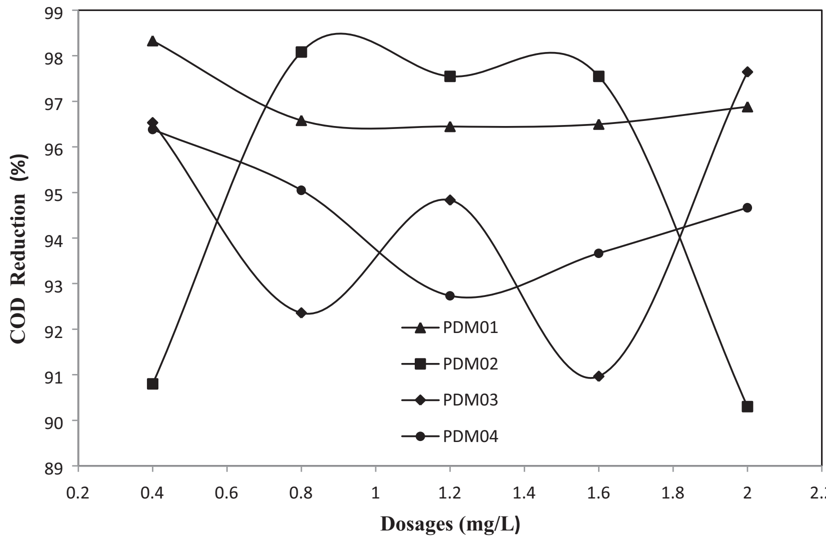

Fig. 4. COD reduction of wastewater after treated with different M_w polyDADMAC at various dosages.

The COD reduction in the wastewater after treatment for polyDADMAC as a function of dosage and M_w is shown in Fig. 4. It can be observed that each polyDADMAC sample produced a COD reduction above 90%. According to the standard of the Interim National Water Quality Standards (INQWS), a maximum oxygen demand between 200 and 1000 mg L^{-1} must be reached before wastewater or industrial water can be returned to the environment. These obtained results were increased from metal based coagulant which is alum.

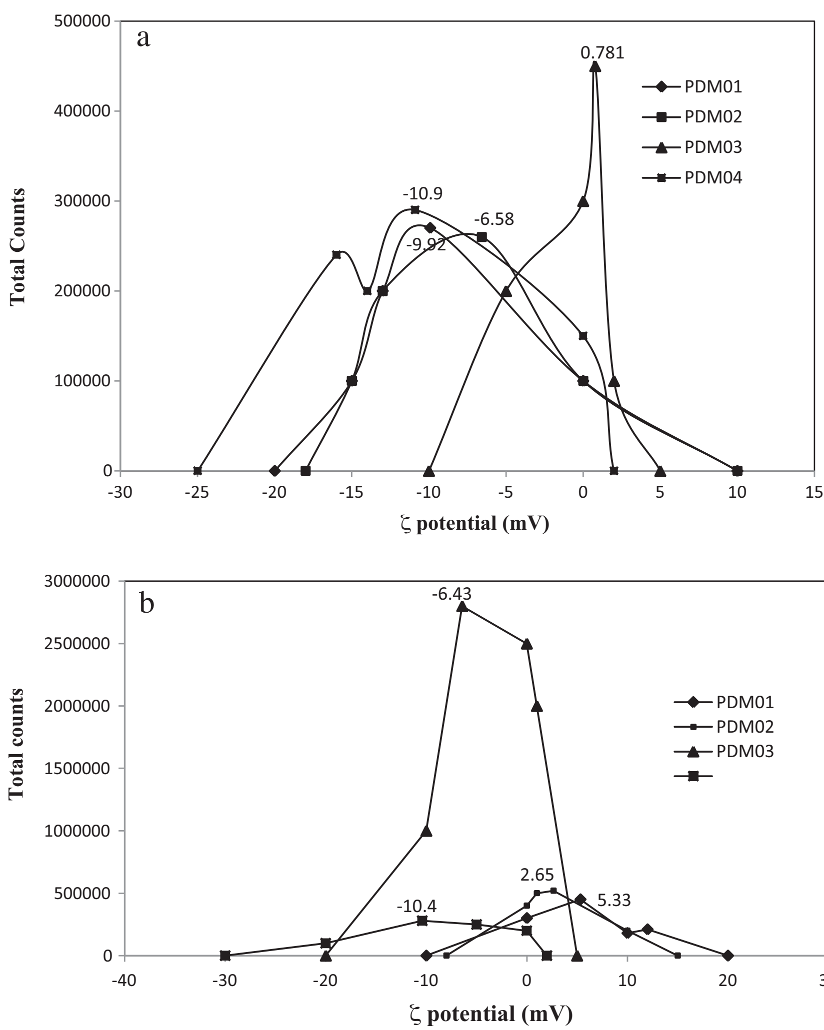

Fig. 5. Relationship Total Counts with ζ potential for different M_w at various dosages (mg L^{-1}): 0.4, (b) 0.8, (c) 1.2, (d) 1.6, (e) 2.0.

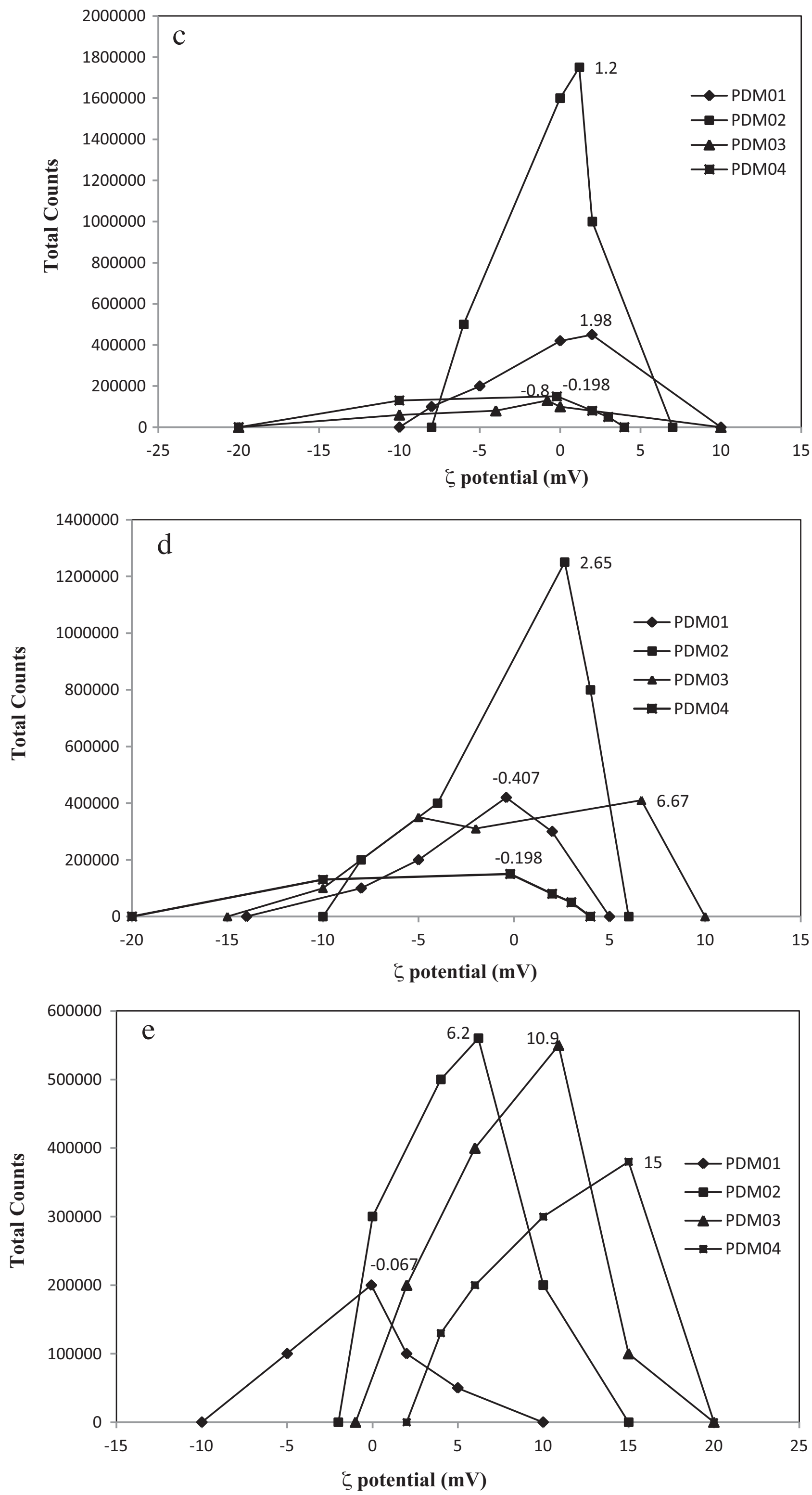

Fig. 5. (*Continued*).

Dilek and Gokcay [35], reported 96% removal of COD from the paper machine while Rohella et al. [36], they found the reduction was about 20%.

From this result, the COD reduction by each polyDADMAC concentration is aligned with the respective standard. When focused on the optimal dosing for turbidity reduction and TSS removal which is 1.2 mg L^{-1}, it can be seen that lower molecular weights (PDM01 and PDM02) result in a higher COD reduction compared with higher molecular weights (PDM03 and PDM04). In a like manner, PDM03 shows a higher COD reduction compared with PDM04. This result indicates that the COD reduction has a significant relationship with the molecular weight, but it is inversely proportional to the results obtained by Wong et al. [24]. However, because of the limited number of publications that stress the effect of the molecular weight of polyelectrolytes, an early inference could be made here suggesting that the molecular weight is not critical in the COD reduction of polyDADMAC.

3.1. ζ Potential measurements

Many of wastewaters are composed of similarly charged particles that repel each other, with the repulsive forces creating a stable and colloidal system. Zeta potential measurement is needed to determine and monitor this system. Zeta potential is electrokinetic potential in colloidal system. Knowledge and information of the zeta potential can reduce the time needed to produce trial formulations. It is also an aid in predicting long-term stability before settling (without flocculant). This zeta potential value or results indicates the degree of repulsion between adjacent similarly charged particles in dispersion [37]. It can be seen in Fig. 5 that all the peak values of the ζ potential obtained were decreases with magnitude than the initial ζ of the wastewater (see Table 2). In stability condition, electrostatic repulsion becomes significant when two colloids approach each other and their double layers begin to interfere. Energy is required to overcome this repulsion between particles. The maximum energy is related to the surface potential and the zeta potential. According to DLVO theory, in order agglomeration to occurs, two particles on a collision course must have sufficient kinetic energy due to their velocity and mass, to “jump over” this energy barrier. In this study, charged polyDADMAC function is to overcome this barrier by neutralize the negatively charged particles by Van Der Waals force. When the dosage of polymer is progressively increased, the strength of the repulsive force between the particles decreases as the charge neutralization point is approached [38].

Optimal flocculation will be achieved when the zeta potential is zero [39]. This is because there is no repulsion between negatively charged particles when the zeta potential is zero. The optimal flocculation for PDM02, PDM03 and PDM04 were achieved at a dosage of 1.2 mg L^{-1} which are −0.198, −0.8 and 1.2 mV respectively as shown in Fig. 5 (c). The optimal flocculation for PDM01 was achieved at 2.0 mg L^{-1} which is −0.067 mV as shown in Fig. 5 (e). The dosage of polymer required to neutralize the particle charge is inversely proportional to the molecular weight. Higher molecular weight polyDADMAC will give more repeating units of positive charge (see Table 3), resulting in dosages for optimal flocculation for PDM03 and PDM02 that are lower compared with PDM01. Also seen in Fig. 5, after optimal dosing, the value of the ζ potential increases with increasing dosage. The absorption of excessive polyDADMAC into the particles causes them to be positively charged. These positively charged particles cause the ζ value to increase, as reported by Wang et al. [40]. They recorded that once optimal flocculation is achieved, further increases in the dosage of flocculent causes the suspended particles to become positively charged. These positively charged particles increase the value of the ζ potential.

Table 3
ζ Potential measurement for different molecular weight of polyDADMAC at pH 7.

Molecular weights (g/mol)	ζ Potential (mV)
8.5×10^4	38.20
8.8×10^4	40.15
10.5×10^4	42.10
15.7×10^4	48.60

3.1.1. Effect of pH

In this research, 1.2 mg L^{-1} was selected because it showed the highest flocculation for normal wastewater conditions, as discussed previously. In flocculation processes driven by polymers, not only are the M_w and dosages of the polymer important but also other process parameters, namely the pH and the electrolyte concentrations, assuming they are significant [41]. In contrast, according to Wong et al. (2006), polyelectrolyte is less dependent of pH. pH are dependent on the H^+ and OH^- concentration which can be observed by ζ potential. The effect of different pH on polyDADMAC will be presented based on the value of ζ potential

As shown in Fig. 6, the flocculation was more efficient at a pH below 9 because the value of the ζ potential was near zero while the value of the ζ potential above pH 9 were above −10 mV. As mentioned earlier, low values of the zeta potential decrease the repulsive force between negatively charged particles, thus increasing the tendency for flocs to occur. At a pH above 9, the value of the zeta potential was highly negative and further from zero, indicating that the flocculation was less efficient at pH values above 9. Increases in the pH produce high concentrations of OH^- in the wastewater sample. Adsorption of these OH^- ions on the interface of a negatively charged particle results in a large diffuse double layer with a higher ζ potential value [42]. This high value indicates that the repulsive force between particles is greater than it was initially, and thus the dosage of polyDADMAC was not sufficient to neutralize the particles.

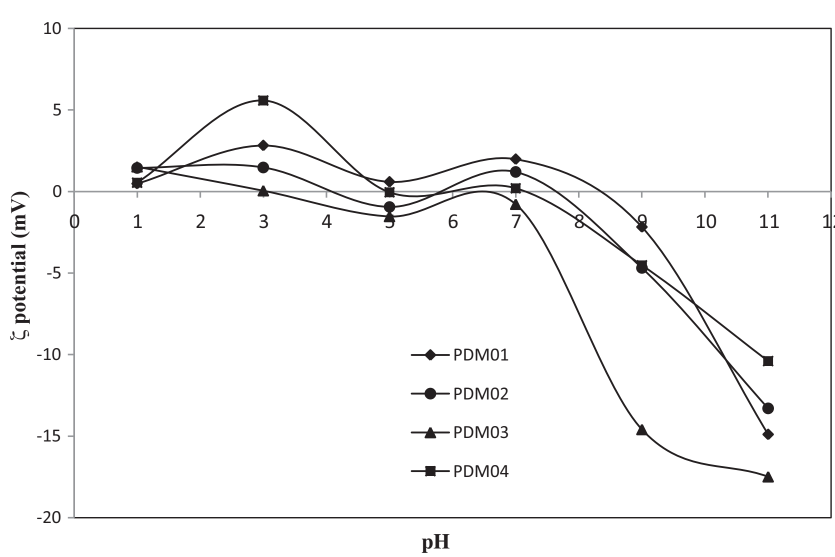

Fig. 6. ζ Potential of wastewater supernatant after treated with different M_w polyDADMAC at dosages 1.2 mg L^{-1} at different initial pH.

4. Conclusion

TSSs, the reduction of turbidity, COD removal, and the ζ potential have been studied using different M_ws of polyDADMAC as flocculants in treating pulp and paper mill wastewater. Based on the present investigation, it can be concluded that higher M_ws show a higher efficiency of flocculation compared with lower M_ws of polyDADMAC. This is a result of the charge neutralization effect aligned with the bridging mechanism, which creates an increasing amount of flocs. Long chains with a high molecular weight create loops and tails, and these loops and tails contributed to bridging mechanism. Another conclusion that could be made here is that the highest flocculation occurred at a lower value of the ζ potential. Decreasing the value of the ζ potential decreased the repulsive forces between particles, and this increased the tendency for flocs to occur. In summary, flocculation of pulp and paper mill wastewater by polyDADMAC is efficient at pH values below 9.

Acknowledgement

The authors wish to acknowledge the financial support provided by USM PGRS (8043024) and also wish to thank GNT Sdn Bhd for the financial support.

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