
Characterisation of nano-sized particles in chemical mechanical polishing wastewater

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Abstract: Treatment of chemical mechanical polishing (CMP) wastewater and fluoride-containing wastewater in semiconductor manufacturing are conventionally separated. The combined treatment of these two streams of wastewater was studied in terms of zeta potential and z-average hydrodynamic diameter profiles as a function of pH 2 to 12. Optimum pH for CMP wastewater was at pH 6 with zeta potential value of -10 mV and mean particle size of 180 d.nm. Meanwhile, for fluoride-containing wastewater the optimum pH obtained was at pH 9 with zeta potential of 10 mV and mean particle size of 5,214 d.nm. These two streams were combined together at their respective optimum pH and resulted a zeta potential value of 0.55 mV and mean particle size of 12,590 d.nm. Results indicated that the combined treatment for both polishing and fluoride-containing wastewater were beneficial as a larger flocs of fluorosilicate (SiF_6^{2-}) was generated without the presence of coagulation chemicals. It is proposed that positively charged particles present in fluoride-containing wastewater become adsorbed on the surface of silica nanoparticles in CMP wastewater in which act as nuclei and enhances flocculation since repulsive force of both wastewater is decreased.

Keywords: semiconductor wastewater; chemical mechanical polishing; CMP; pH; zeta potential; particle size; aggregation; disaggregation.

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Biographical notes: Noor Aina Binti Mohamad Zuki graduated with a Master's degree from the Universiti Sains Malaysia in 2016 based on the study of 'Development of natural coagulant aid from *artocarpus heterophyllus* seeds starch in leachate treatment'. During her two and a half years of study, she managed to publish three articles and attended four local conferences. She is currently in her second year of PhD study with research field on nanoparticles in semiconductor wastewater. Her study is focusing on using dynamic light scattering technique as an alternative methodology to pre-determine the optimum pH and dosage range, in terms of zeta potential and hydrodynamic diameter. Despite of her student life, she also acts as a researcher for an international company located in Hi-Tech Kulim, Kedah, Malaysia under Public Private Research Network, collaborating with Universiti Sains Malaysia together with the Ministry of Higher Education.

Norli Ismail obtained her Bachelor's in Environmental Science from the Universiti Putra Malaysia, Master's in Chemical Processes and PhD in Environmental Technology from the Universiti Sains Malaysia. Currently, she is a Lecturer at School of Industrial Technology USM. She has research experience in water quality, management issues, and treatability studies in relation to water, wastewater and analytical testing. She is actively involved in the technical aspects of biological and physico-chemical treatability studies, bioremediation research, environmental analytical techniques, sampling and data validation. She was involved in a research project on wastewater treatment and acclimatisation studies for a semiconductor industry and as a co-researcher for sewage treatment plant development modification of private company; and also as a co-researcher for the Bioremediation of an Industrial Waste (Latex Effluent) project. She has research experience collaboration on hydrothermal gasification of palm oil mill effluent (POME) with Osaka Gas Ltd., Japan, at the Eco-energy Department.

Fatehah Mohd Omar graduated from the University of Geneva, Switzerland in 2015 based on the study of 'Characteristics, behaviour, fate and transport of ZnO nanoparticles in aqueous systems' using state-of-the-art analysis equipment to study the properties of nanoparticles and the transformations that may governed the behaviour of the nanoparticles that affects its fate and transport. She has expanded her scope of research on the characteristics of other manufactured nanoparticles, i.e., silica dioxide (SiO₂) and ferrous oxide (Fe₂O₃) generated from industrial wastewater. In pursuance of her research, she has received numerous research grants which include *International Foundation for Science*, L'Oreal – UNESCO Malaysian Fellowship for Women in Science 2016 and State Secretariat for Education, Research and Innovation. She has also formed collaboration ties with multinational companies to tackle certain wastewater treatment processes. To date, she has published up to 24 international peer-reviewed journals related to water pollution, water and wastewater treatment.

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1 Introduction

Semiconductor industry has become a representative and major industry as it has been growing rapidly at a double-digit rate (Chuang et al., 2012). The semiconductor manufacturing involves in many complex processes including silicon growth, oxidation, doping, planarisation, cleaning, etc. (Chuang et al., 2012). The semiconductor wastewater is separated into three different streams; acid-base, chemical mechanical polishing (CMP), and fluoride-containing wastewater. Wastewater discharged from the semiconductor industry contains high organic and inorganic compounds as well as turbidity (Yang et al., 2011). When membranes systems are used for semiconductor wastewater reclamation without pre-treatment, membranes will soon be clogged by the nano-sized particles.

CMP process is mainly used for polishing the device side of a semiconductor wafer via the mechanical downward force of a slurry abrasive in association with the chemical oxidation of the wafer surface (Chih et al., 2012). However, the CMP process has a major drawback as it consumes a large amount of ultrapure water during washing and cleaning step of CMP slurry (Drouiche et al., 2007; Yang et al., 2013). Consequently, this process has generated a significant amount of wastewater that is difficult to be treated (Sheng and Chung, 2012; Browne et al., 2010; Golden et al., 2010). The major contaminants in the CMP wastewater are nano-sized particles. Other than that, the majority of inorganic and organic contaminants in the CMP effluent derived from the CMP slurry (Drouiche et al., 2007). For example, inorganic contaminants in CMP wastewater consist of SiO_2 , Al_2O_3 , or CeO_2 in which exists as a suspended solids. Meanwhile, organic contaminants may include complex agents, surfactants, stabilisers and rheology control agents (Golden et al., 2010). Eventually, these contaminants of CMP end up in the wastewater even after the wafer washing is completed. CMP wastewater is highly turbid with milky colour due to its high solid contents present in it (Sheng and Chung, 2012).

Many researchers have investigated different methods to treat CMP wastewater. One of the earliest studies on CMP wastewater treatment was done by Belongia et al. (1999) who discovered that electrochemical method was able to remove silica and alumina from CMP wastewater. The electro-microfiltration processes can effectively remove SiO_2 from CMP wastewater (Chang and Liu, 2011). The dispersed air flotation (DiAF) has been shown to be effective to remove silica from CMP wastewater (Hu et al., 2010). Coagulation and flocculation method is one of the most prevalent procedures for treating CMP wastewater (Lai and Lin, 2004). During treatment processes, CMP wastewater need to recycle large quantities of water, thus over dosing the coagulant to form sufficient hydroxide precipitates to ensure that the particles are effectively separated from the water (Corlett, 2008). This approach is commonly known as sweep flocculation as is especially important when the particle concentration is low and inter-particle collisions are infrequent (Yang et al., 2009). This method produces large amount of sludge and increases its treatment cost.

Fluoride presence is naturally in the Earth's crust and industrial activities (steel, glass, electroplating, aluminium, and fertiliser industries). However, the discharge of the wastewater leads to fluoride contamination of ground and surface water. At higher concentration of fluoride can produce serious health problems that result in skeletal fluorosis (Chuang and Liu, 2012). Fluoride-containing wastewater contributes 40% of hazardous waste produced from semiconductor manufacturer (Chuang and Liu, 2012;

Lai and Lin, 2014). The amount of fluoride-containing wastewater generated for semiconductor fabrication facilities ranges from 350 m³/d to 700 m³/d with fluoride concentration varying from 50 mg/L to 1,000 mg/L (Lai and Lin, 2014).

According to the previous research, there are several methods that have been developed to remove the fluoride content in the semiconductor wastewater. Based on study conducted by Chang and Liu (2011), they claimed that fluoride content in the semiconductor wastewater can be removed effectively by using a packed-bed reactor with granular calcite. Aldaco et al. (2005) used a crystallisation method in fluidised bed reactor in viable technically conditions as an alternative to the conventional precipitation. In addition, fluoride-containing wastewater can be treated by modifying the conventional chemical precipitation using addition and regeneration of Al(OH)₃ and make process more efficient. Precipitation flotation methods including DiAF and dissolved air flotation (DAF), are feasible alternative processes for the removal of fluoride from semiconductor wastewater (Lo and Low, 2008; Huang et al., 2009).

Semiconductor manufacturer rarely combined treatment process for CMP wastewater and fluoride-containing wastewater (Den and Huang, 2013). As these two streams are mixed, then wastewater is expected to be high in turbidity, total solid content, silica and fluoride. There has been no study on the efficiency of combined both wastewater in terms of dynamic light scattering (DLS) technique to pre-determine the optimum pH and dosage range. It is unknown to which extent that nanoparticles will agglomerate depending on the conditions and the balance between the attractive and repulsive forces among the nanoparticles.

Therefore, in this pilot-scale study, the combination of both wastewater; CMP wastewater and fluoride-containing wastewater will be studied by analysing the zeta potential and hydrodynamic diameter of each wastewater in which to determine the optimum pH conditions for both. It stands to reason that pH has a huge influence on zeta potential value by changes in acidity and alkalinity of ions present in the wastewater as it represents the charge of a nanoparticle with respect to that ambient surroundings. Moreover, at certain pH values can strongly affect the suspension stability (agglomerate or disaggregate).

2 Methodology

2.1 CMP wastewater and fluoride-containing wastewater

The source of water regeneration came from the effluent of the wastewater treatment plant of an industrial park located at Kulim Hi-Tech Park, Kulim, Kedah, Malaysia. Wastewater samples were collected four times per month from the effluent location of the wastewater treatment plant. A 100 mL of each wastewater samples were prepared and the initial pH was monitored using a pH metre (780 Metrohm). The samples were taken out at specific pH values and the electrophoretic mobility and z-average hydrodynamic diameter were determined with a Zetasizer Nano ZS (Malvern) at 25°C. Experiments were done in triplicate and the average zeta potential and z-average hydrodynamic diameter were determined. Sodium hydroxide (NaOH) and hydrochloric acid (HCL) of 1 M respectively was used to adjust pH from pH 2 to 12.

2.2 Optimum pH

During each test run, 100 mL of wastewater samples was placed on the magnetic stirrer. The magnetic stirrer was turned on and set at 500 rpm. A steady temperature of 25°C was maintained for all test runs. The stirrer speed was found to be sufficient to provide good mixing and yet not strong enough to break up the flocs formed during the process. The initial pH was recorded as soon as the samples reached a constant pH value. Next, the pH was adjusted by using a 10 µL micropipette by adding NaOH or HCl until it reach certain desired pH values. The average zeta potential and z-average hydrodynamic diameter was tested at each pH value from pH 2 to 12.

2.3 Zeta potential and particle size measurements

Zeta potential of CMP wastewater and fluoride-containing wastewater were determined according to the Smoluchowski equation while the nanoparticles size was measured using the DLS method with the Zetasizer Nano ZS (Malvern). During the measurement, a 633 nm He-Ne laser beam passes through the particle suspension and scattered light is detected and collected by a photo-detector at a fixed scattering angle of 173°. The light signal is then fed in the correlator, which accumulates a light scattering intensity autocorrelation function. The fluctuation of autocorrelation function over decay time is related to the diffusion coefficients of the particles that undergo Brownian motion. The hydrodynamic diameter of the particles is determined from the diffusion coefficient in the terms of the Stoke-Einstein equation. For a poly-dispersed suspension, the obtained mean particle size is called the 'z-average' diameter (dz), which is determined by the intensity of the light scattered by the particles. It should be noted that DLS only measures particles ranging from 2 nm to 6 µm. Nanoparticle suspensions were injected into polystyrene vials using a syringe for DLS measurements. All vials were used only once and were rinsed with nanopure water before being filled with the suspensions. Measurements were conducted for 5 min at room temperature (25°C) to obtain the z-average particle size.

3 Results and discussion

3.1 Semiconductor wastewater characteristics

Raw samples for CMP wastewater and fluoride-containing wastewater were sampled from Kulim Hi-Tech Park, Kulim, Kedah. Table 1 represents the characteristics of both wastewater according to four consecutive weeks. From the data obtained can be concluded that both samples; CMP wastewater and fluoride-containing wastewater were all in a good condition without exceed the limit provided by EQA. This show that these samples generated from this industry will not harm the surroundings as the value of COD, turbidity and suspended solids were under controlled.

Figures 1 and 2 show the zeta potential of the CMP wastewater and the corresponding particle size as a function of the CMP wastewater pH. The solution pH was adjusted by using a 10 µL of micropipette and maintained by the addition of the required amount of NaOH or HCL. The pH of semiconductor wastewater was at $\text{pH } 9.37 \pm 0.2$ with zeta

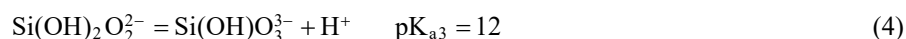
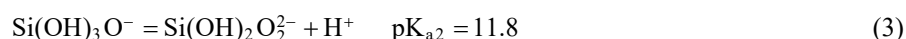
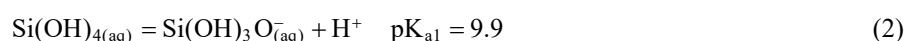
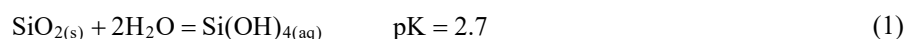
potential value was -60.9 mV. As can be seen from Figures 1 and 2, an increase in pH resulted in a greater degree of negative zeta potential and a decrease in the corresponding particle size. From the graph, by increasing the pH of the CMP wastewater from 2 to 12, the zeta potential varied from -3.2 to -66 mV and the mean particle size decreased from 167.1 d.nm to 151.9 d.nm. This happens to the pH increase in CMP wastewater tended to adsorb more OH^- ions on the particle surface and created Si-O^- on the surface, thus causing greater negative zeta potential (Duan and Gregory, 2010). This indicate that the pH has a definite effect on the zeta potential as the change in the value of zeta potential was found to alter the stability of CMP nanoparticle suspension.

Table 1 CMP wastewater and fluoride-containing wastewater characteristics according to four consecutive weeks

Weeks	CMP wastewater			Fluoride-containing wastewater			
	COD (mg/L)	Turbidity (NTU)	Suspended solids (mg/L)	COD (mg/L)	Turbidity (NTU)	Suspended solids (mg/L)	Fluoride content (mg/L)
W1	159	36.9	19.3	94	4.4	3	0.5
W2	141	65	19.3	99	5.3	3	0.5
W3	157	52	16	34	4.6	3	0.3
W4	73	67	22	26	10.3	5	0.2

Notes: *EQA permissible limit: COD = 100 mg/L, suspended solids = 100 mg/L, fluoride content = 1.5 mg/L.

Based on the graph plotted in Figure 2, as the pH increased from 2 to 5, the size of particle was fluctuate in the range between 151.1 d.nm to 167.8 d.nm. According to the equations for SiO_2 dissolution [equations (1) and (4)], the dissolution of SiO_2 raises with pH increasing (Swaddel, 2008).



In addition, the electrostatic repulsion between the particles of the electric double layers with greater negative potential drives suspended particles apart, achieving particle stability and reducing a smaller particle size. Therefore, a relatively smaller mean particle size associated with CMP wastewater could be attributed to its relatively large negative zeta potential (Lin and Yang, 2010). pH 2 has less negatively charged of zeta potential compared to pH 5, thus the particles has a larger agglomeration. As the pH increased to 6, the particle reached its stability with bigger particle size. Meanwhile, from pH 9 to 12 the size of CMP nanoparticles remain unchanged with the smallest agglomerate size of particles existed. At high pH where the nanoparticle displayed a strongly negative charged surface, thus the intense charge on the surface increased the electrostatic repulsion between the particles (Yang et al., 2010).

Figure 1 Zeta potential of CMP varies with pH

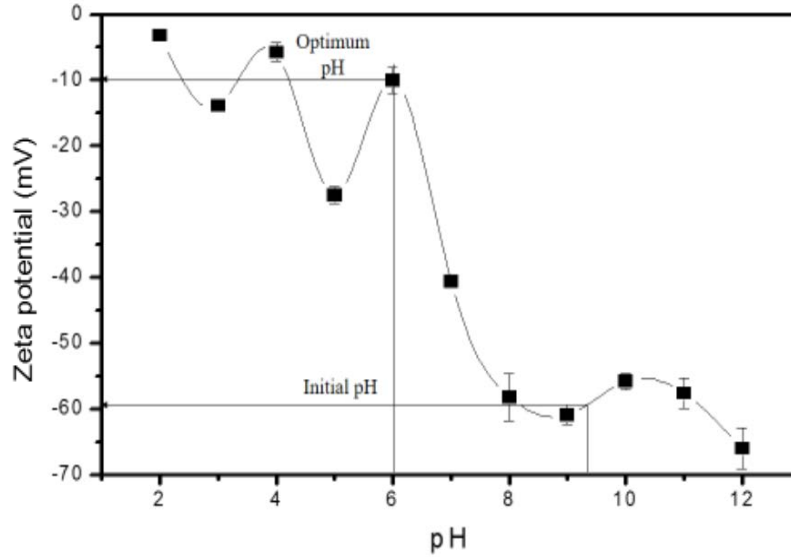
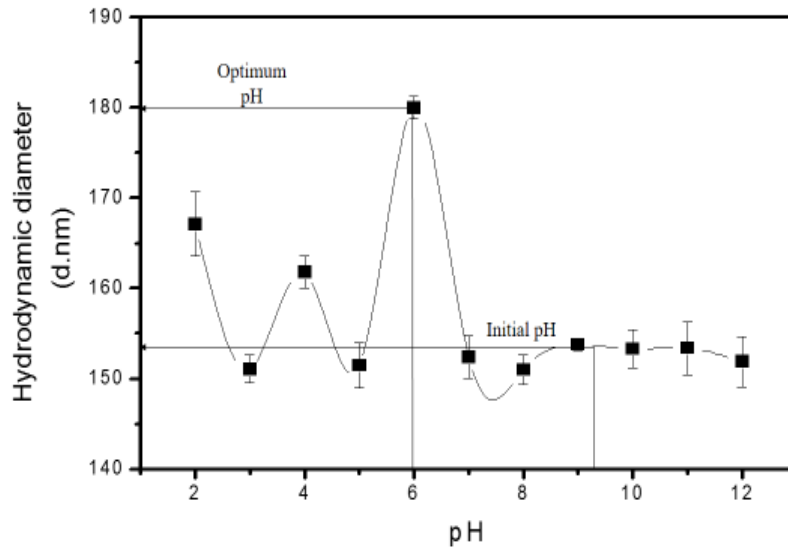


Figure 2 Hydrodynamic diameter of CMP varies with pH



The CMP wastewater mainly contains suspended, nano-sized solids originated from slurry abrasive particles of SiO₂, Al₂O₃ or CeO₂, depending on the nature of the CMP applications (Wang et al., 2009). Based on the FTIR obtained (Figure 3) proved that the present of silica group (Si-H group) in the CMP wastewater at 1,635.71 cm⁻¹, while the hydroxyl group was also found in the wastewater at 3,333 cm⁻¹.

Figure 3 FT IR of CMP wastewater

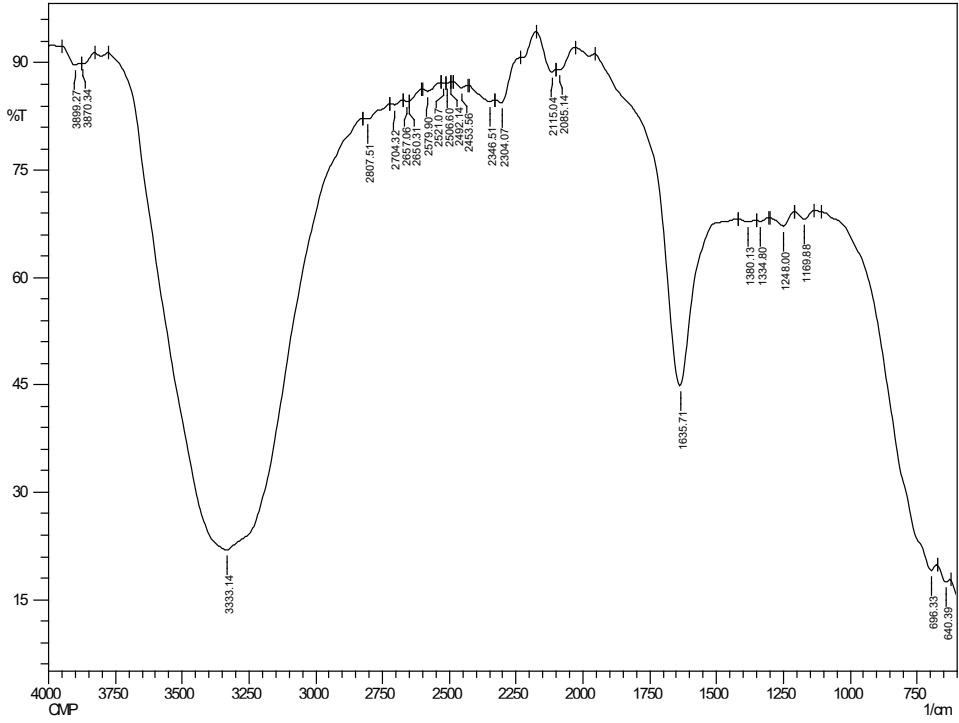


Figure 4 Zeta potential of fluoride-containing wastewater varies with pH

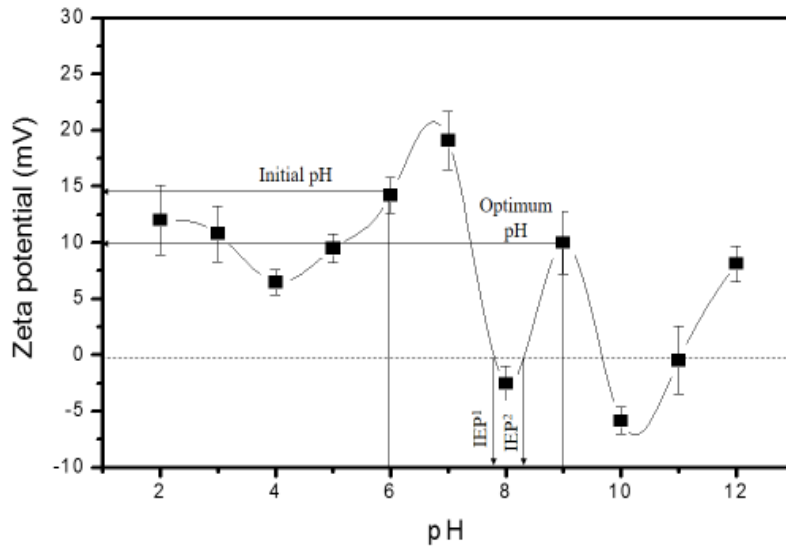


Figure 5 Hydrodynamic diameter of fluoride-containing wastewater varies with pH

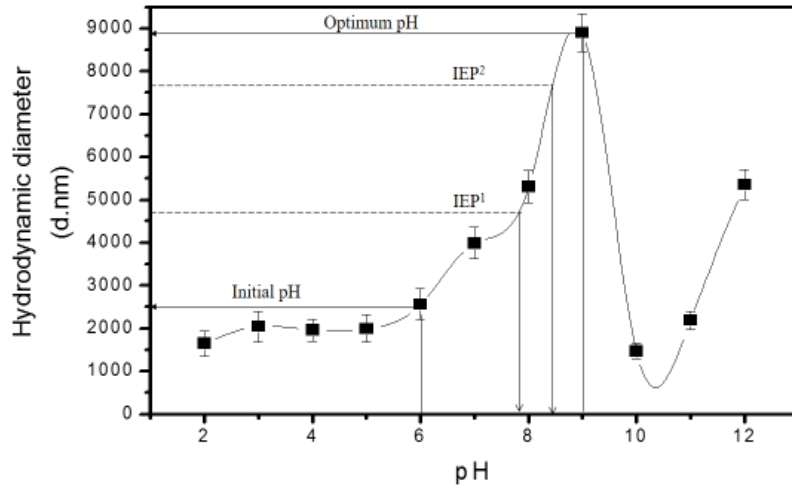
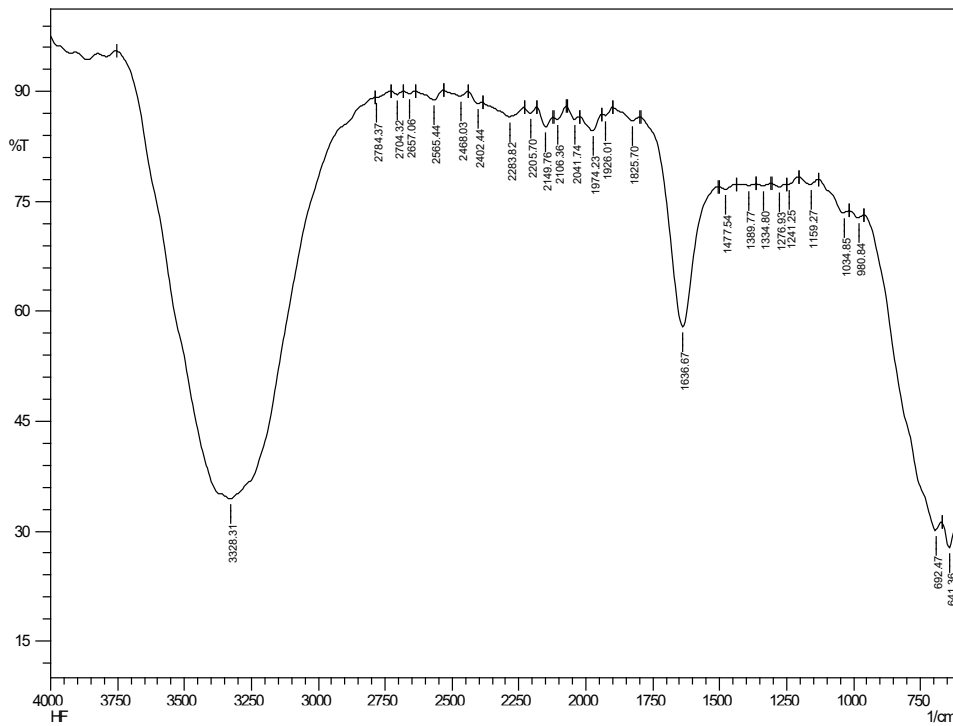


Figure 6 FT IR of fluoride-containing wastewater



Figures 4 and 5 show the zeta potential and mean particle size with varies of pH of fluoride-containing wastewater. At initial pH of fluoride-containing wastewater of 6.28 ± 0.2 with positive zeta potential of 14 mV shows mean particle size of 2,565 d.nm. Agglomeration of nanoparticles occurs when the individual particles are held together by

weak inter-particle interaction, electrostatic attraction and van der Waals forces. The point at which the nanoparticles exhibit no net charge is termed as isoelectric point (IEP) (Golden and Carrubba, 2001). Fluoride-containing wastewater achieved its charge neutralisation at two different points; pH 7.8 and pH 8.2 with hydrodynamic diameter of 4,752 d.nm and 7,786 d.nm, respectively.

From the graph illustrated, increased in pH value resulted in decreased of zeta potential with bigger corresponding particle size. For example, by increasing the pH from pH 2 to 7, the zeta potential varied from 12 to 19.1 mV and the mean particle size increased from 1,653 d.nm to 3994 d.nm. Consequently, the electrostatic repulsion between the particles tends to move the particles closer, resulted in particles agglomeration (Den and Huang, 2013). Optimum pH is obtained at pH 9 where the zeta potential is 10 mV with particles size of 8,900 d.nm. In addition, from Figure 6, fluoride-containing wastewater shows the presence of functional group containing mainly hydroxyl group (OH^-) at $3,328.3 \text{ cm}^{-1}$ and also N-H group ($1,636.7 \text{ cm}^{-1}$).

3.2 Interaction between CMP wastewater and fluoride-containing wastewater

The interaction between CMP wastewater with fluoride-containing wastewater has not been studied in details in terms of zeta potential and hydrodynamic diameter. Very little is known about the advantages and disadvantages of combined treatment. The optimum pH of CMP wastewater is selected to be at pH 6 with negative zeta potential of -10 mV and mean particle of 180 d.nm. Meanwhile, the optimum pH of fluoride-containing wastewater is at pH 9 with positive zeta potential and mean particle size of 10 mV and 8,900 d.nm, respectively. Combining both wastewater at each optimum pH has resulted in changes of pH to $\text{pH } 8.58 \pm 0.2$ with negative zeta potential of -22.5 mV and corresponding particle size of 5,214 d.nm.

Figure 7 Zeta potential of interaction between CMP wastewater and fluoride-containing wastewater varies with pH

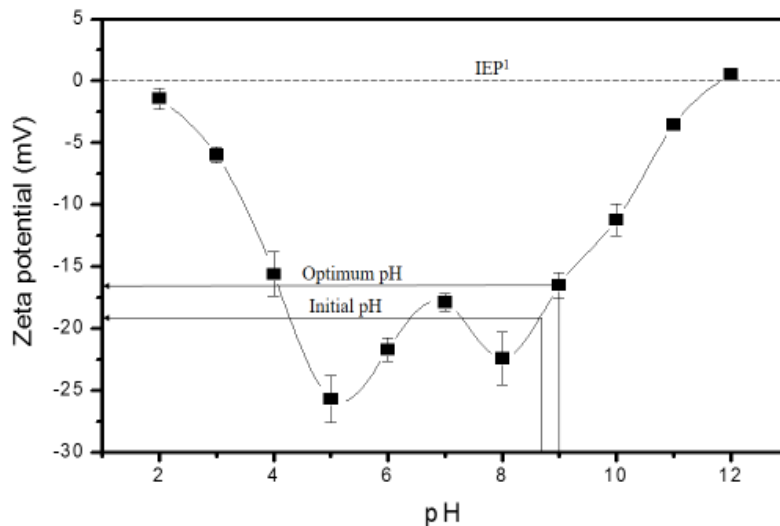


Figure 8 Hydrodynamic diameter of interaction between CMP wastewater and fluoride-containing wastewater varies with pH

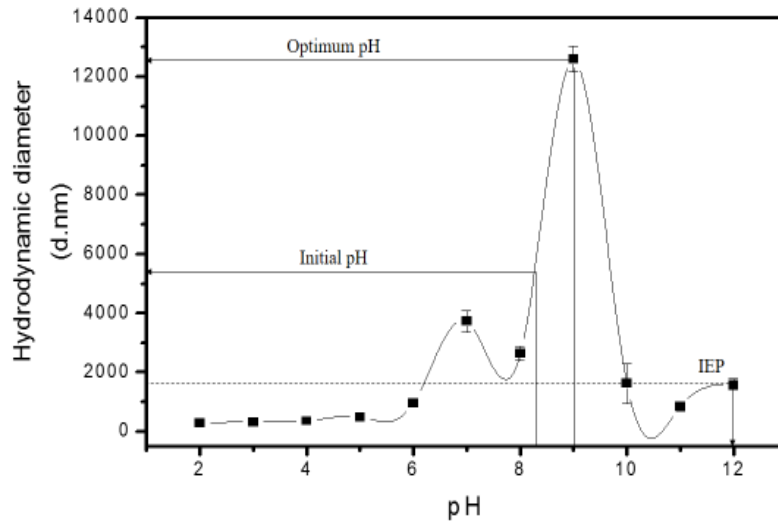


Figure 9 Illustration of the interaction of the silica nanoparticles in CMP wastewater, fluoride-containing wastewater

	ACIDIC	NEUTRAL	ALKALINE
CMP WASTEWATER			
FLUORIDE-CONTAINING WASTEWATER			
CMP WASTEWATER (pH 6) + FLUORIDE-CONTAINING WASTEWATER (pH 9)			

From Figure 6, it is demonstrated that increased in pH from its initial pH value to alkaline also increased the zeta potential from -22.5 to 0.55 mV and achieved its charge of neutralisation at pH 11.8. It is proposed that fine silica particles in CMP wastewater that have negative charge become adsorbed on the surface of positively charged fluoride

particles in fluoride-containing wastewater. It is found that at optimum pH 9, the mean particle size has increased dramatically to 1,2590 d.nm with zeta potential of -16.5 mV (Figure 7). This is due to the decreased electrostatic repulsion among silica and fluoride particles then results in larger flocs. It is also probable that fine silica particles in CMP wastewater can act as nuclei for precipitation of fluoride particles and facilitates the floc growth (Toyoda and Taira, 2007).

According to reactions (7) and (8), the reaction of silica and fluoride formed precipitate known as fluorosilicate (SiF_6^{2-}). The formation of SiF_6^{2-} did not delay the precipitation reaction due to different characteristics of wastewater, since relatively low initial volume was utilised in the experiment (Luna ad Liu, 2009). From thermodynamic modelling, it is predicted that most of dissolved silica exists as H_4SiO_4 when $\text{pH} < 9.5$ and H_3SiF_4^- when $\text{pH} > 9.5$. Vogelsberger et al. (2008) stated that SiF_6^{2-} was unfavourable to form at $\text{pH} < 9$ and recommend keeping the pH at 8 to 9 for precipitation of SiF_6^{2-} in the presence of SiO_2 . This supports with the aforementioned for the selected optimum pH to be at pH 9.



It was proposed that two different mechanisms might be included. Firstly, positively charged fluoride particle become adsorbed on the surface of negatively charged silica in CMP wastewater and enhanced particle agglomeration. From the illustration in Figure 9 showed that at optimum pH for CMP wastewater (pH 6) and fluoride-containing wastewater (pH 9), the bigger flocs formed as the repulsive force of both wastewater is decreased. Secondly, heterogeneous nucleation might be involved, with nano-sized silica act as nuclei to enhance precipitation of SiF_6^{2-} . Potential advantages for the combined treatment of CMP wastewater with fluoride-containing wastewater include lower chemical dosage, better control of process and smaller footprint.

4 Conclusions

The research study showed that the combined treatment for both polishing and fluoride-containing wastewater is beneficial. First, the combination of CMP wastewater and fluoride-containing wastewater resulted a suspension at pH 8 with corresponding particle size of 5,214 d.nm. The combined wastewater generated a larger flocs compared to a single wastewater alone. Second, the coagulation occurred without the presence of coagulation chemicals. The formation of large flocs was observed at pH 9 with mean particle size of 1,2590 d.nm.

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