CHARACTERIZATION AND BEHAVIOUR OF FLUORIDE-CONTAINING SEMICONDUCTOR WASTEWATER WITH THE PRESENCE OF CALCIUM CHLORIDE ($CaCl_2$) FOR OPTIMIZATION TREATMENT PROCESSES

by

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This dissertation is submitted to

UNIVERSITI SAINS MALAYSIA

as partial fulfillment of requirements for the degree of

MASTER OF SCIENCE
(ENVIRONMENTAL ENGINEERING)

School of Civil Engineering
Engineering Campus
Universiti Sains Malaysia

July 2017

ACKNOWLEDGEMENTS

First and foremost, Alhamdulillah. All praises to Allah, the Most Gracious, the Most Merciful. His guidance and great loving kindness has brought me together and encourages me to complete this dissertation.

I would like to express my deepest gratitude and sincere thanks to my supervisor, Dr. Fatehah Binti Mohd Omar for giving me an opportunity and continuous support for my study and research in this field. Also, for her patience, motivation and guidance throughout my research. Thanks to her continuous encouragement, I am able to finish this research and thesis writing.

Thank you to Kementerian Pendidikan Malaysia for the scholarship awarded to further my study in this field. Hopefully this knowledge may help me improve myself and the department.

For my beloved husband, Mohd Said and lovely children, Muhammad Adam Arsyad and Nur Ameena Aisyah, thank you for always understand and supporting during the stressful and hardship moment. I am very sorry, for all the lacking and chaos that I done in achieving my dream. Dear my parent and gorgeous sisters, Amilia and Ainaa thank you for everything. Without you all this journey may be more difficult. Love you all.

Special thanks to all technicians from the School of Civil Engineering, especially the technicians Mrs. Shamsiah, Mr. Zaini, Mr. Mohad from both Environmental Laboratory 1 and 2.

Last but not least, to all my friends and classmate. Thank you.

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LIST OF ABBREVIATIONS

UV	Ultraviolet
IR	Irradiation By Infrared
E&E	Electrical And Electronics
GNI	Incremental Gross National Income
WHO	World Health Organization
UPW	Ultrapure Water
MOSFET	Metal-Oxide Semiconductor Field-Effect Transistor
COD	Chemical Oxygen Demand
BOD	Biochemical Oxygen Demand For
TOD	Total Oxygen Demand
TOC	Total Organic Carbon
CMP	Chemical Mechanical Polishing
FCSWW	Fluoride-Containing Semiconductor Wastewater
TS	Total Solid
SS	Suspended Solids
TSS	Total Suspended Solids
BOE	Buffered Oxide Etch
HF	Hydrofluoric Acid
$CaCl_2$	Calcium Chloride
NPs	Nanoparticles
ISO	International Organization For Standardization
ASTM	American Society Of Testing And Materials
NIOSH	National Institute Of Occupational Safety And Health

SCCP	Scientific Committee On Consumer Products
BSI	British Standards Institution
TEM	Transmission Electron Microscope
SEM	Scanning Electron Microscope
MALDI-TOF	Matrix-Assisted Laser Desorption/Ionization Time-Of-Flight Mass Spectrometry
FTIR	Fourier Transform Infrared Spectroscopy
XPS	X-Ray Photoelectron Spectroscopy
AFM	Atomic Force Microscopy
XRD	Powder X-Ray Diffraction
NTA	Nanoparticle Tracking Analysis
DLS	Dynamic Light Scattering
PZC	Point Of Zero Charge
IEP	Isoelectric Point
PVC	Polyvinyl Chloride
PAC	Poly-Aluminium Chloride
USEPA	United States Environmental Protection Agency
EPA	Environmental Protection Agency
NPDES	National Pollutant Discharge Elimination System
NPDWR	National Primary Drinking Water Regulations

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Publication 1 Submitted to International Conference for Environmental Research and Technology (ICERT) 2017 Conference Proceedings 93

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Publication 3 Journal Article in Preparation for Submission to Journal of Colloid and Interface Science (Impact Factor 2016: 4.233) 130

ABSTRAK

Air sisa semikonduktor mengandungi sebatian organik dan bukan organik yang terhasil daripada beberapa proses yang sangat kompleks dan rumit. Air sisa dari industri ini secara amnya dibahagikan kepada tiga aliran utama iaitu yang mengandungi fluorida, berasaskan asid dan dari proses gilapan kimia. Kajian awal telah dijalankan untuk menganalisis julat optimum pH melalui interaksi air sisa semikonduktor yang mengandungi fluorida dan koagulan dari segi potensi zeta dan pengukuran diameter hidrodinamik berdasarkan fungsi pH (pH 2-12). Pada setiap pH yang ditentukan, caj permukaan dan saiz zarah diukur menggunakan teknik serakan cahaya dinamik. pH awal air sisa semikonduktor dicatatkan pada pH 5.5, +15.54 mV bagi potensi zeta dan 1952 d.nm bagi pengukuran diameter hidrodinamik. pH titik sifar caj ditemui pada pH 7.1 dengan saiz zarah 4500 d.nm. Analisis ciri pada $CaCl_2$ menunjukkan bahawa pada awalnya pH 5.7, potensi zeta memberikan nilai negatif -8. MV dengan purata saiz zarah adalah 770 d.nm. Seterusnya, julat dos optimum melalui interaksi (proses pengagregatan dan pengasingan) antara air sisa semikonduktor yang mengandungi florida dan koagulan ($CaCl_2$) pada nilai pH yang berbeza secara sistematik dengan menggunakan pH_{PZC} bagi air sisa sebagai titik rujukan. Eksperimen dijalankan dalam tiga keadaan pH iaitu pada keadaan i) pH <pHPZC, ii) pH = pHPZC dan iii) pH> pHPZC. Set ujian eksperimen yang dijalankan adalah untuk mengukur kecekapan penyingkiran fluorida dalam air sisa semikonduktor dalam dos koagulan yang optimum. Apabila $CaCl_2$ ditambah, peratus penyingkiran fluorida untuk ketiga-tiga keadaan adalah 13.9%, 35.5% dan 18.6%. Ini dibandingkan dengan analisis yang dilakukan menggunakan ujian balang di mana peratusan penyingkiran adalah sebanyak 21.0%, 54.9% dan 32.4%. Walau bagaimanapun, kedua-dua analisis dalam

eksperimen ini menunjukkan bahawa keadaan pH = pHPZC memberi peratusan terbesar penyingkiran fluorida. Keputusan dos yang digunakan untuk ketiga-tiga keadaan pH ialah 140 mg / L, 35 mg / L dan 35 mg / L masing-masing

ABSTRACT

Semiconductor wastewater contains high organic and inorganic compounds generated from several highly complex and delicate processes. The wastewater is generally divided into three different main streams, i.e. fluoride containing, acid base and chemical mechanical polishing. In this research, a preliminary study was conducted to analyse the optimum pH range via characterization of fluoride-containing semiconductor wastewater and coagulant ($CaCl_2$) in terms of zeta potential and hydrodynamic diameter measurements as a function of pH (pH 2-12). At each adjusted pH, the surface charge and particle size were measured using the dynamic light scattering technique. The initial pH of semiconductor wastewater suspensions were recorded at pH 5.5, +15.54 mV and 1952 d.nm for zeta potential and hydrodynamic diameter measurement respectively. The pH of point of zero charge was found at pH 7.1 with a particle size of 4500 d.nm. Characteristic analysis on $CaCl_2$ indicated that at initial pH 5.7, zeta potential gave a negative value of -8. mV with an average particle size of 770 d.nm. The subsequent stage were optimum dosage range via interaction (aggregation and disaggregation process) between fluoride-containing semiconductor wastewater and coagulant ($CaCl_2$) at different pH values in a systematic way by using the pH_{PZC} of the wastewater as a point of reference. Experiment were carried out in three pH regions which are experiments set conducted were, i) $pH < pH_{PZC}$, ii) $pH = pH_{PZC}$ and iii) $pH > pH_{PZC}$. The final experiment set conducted were to measure the removal efficiency of fluoride in fluoride-containing semiconductor wastewater within the coagulant optimum dosage. When $CaCl_2$ were added the percentage of fluoride removal for the three conditions were 13.9%, 35.5% and 29.9% respectively. These were compared to the analysis done by jar test where by the removal percentage were

notes as 21.0%, 54.9% and 18.6 %. However, in experiments both analysis show that the condition $pH=pH_{PZC}$ gives the biggest percentages of fluoride removal. The dosage determined for the three pH region were 140 mg/L, 35 mg/L and 35 mg/L respectively.

CHAPTER ONE

INTRODUCTION

1.1 Background

Semiconductor has applicability to be used to conduct electricity under some condition or unique situation. It also known as a substance that usually a stable chemical element or composite. Semiconductor being used in many electrical circuits because it can control the flow of electrons creating an excellent medium for electrical current to be controlled. The existing of surface and interface state result from the remarkable electronic and structural properties of semiconductor surfaces and interfaces (Mönch, 2013). However, many factors that affect the semiconductor conductance such as visible light, ultraviolet (UV), dependent on the current or voltage use to a control electrode, or on the intensity of irradiation by infrared (IR), or X rays.

Therefore, semiconductor becomes an important electronic industry in Malaysia. It is extensively used in mobile devices (smart phones, tablets), optoelectronics (photonics, fibre optics, LEDs), embedded technology such as integrated circuits, storage devices (cloud computing, data centre's), medical test equipment and electronic control devices (Lee et al., 2010). As the growing demand, average yearly growth of semiconductor industry increases quickly and is predictable to last in the predictable future (Kim et al., 2009). Studies have shown that the electrical and electronics (E&E) manufacturing is the important sector in Malaysia's industrial sector, contributing remarkable to the country's employment (23.7%) and exports (33.4%) (Malaysian Investment Development Authority, 2014) The international semiconductor market was predictable to grow at a multiple annual growth rate of about 15 per cent from year 2015 to 2019 and it has been strengthen by the development of the industry through its Economic Transformation Programme which is projected to generate an incremental gross national income (GNI) impact around RM53.4 billion which creates about 157,000 new jobs by 2020 (Borneo Post, 2016).

This industry involves several extremely difficult and elusive processes that consist of several steps of silicon growth, oxidation, doping, photolithography, etching, stripping, dicing, metallization, planarization, cleaning and etc.(Yoshino et al., 2014, Lien and Liu, 2006). Semiconductor manufacturing is also becoming one of the largest water-consuming industries since a huge quantity of water is required during the cleaning and rinse-out process of semiconductor wafers production (Liu et al., 2016, Aoudj et al., 2015). Wastewaters are produced by manufacturing processes including photolithography, photo resist stripping, etching, pure water washing and soon (Zhang et al., 2016, Yoshino et al., 2014, Soni and Modi, 2013). A large quantity of hydrofluoric acid is currently used in the industry unit such as photovoltaic cell manufacturing and electronics plants (Aoudj et al., 2016) in order to perform the manufacturing process activities. The concentration of fluoride in semiconductor wastewater exceeds 1000 mg/L which correspond to a huge threat to the environment (Huang and Liu, 1999). Other than that, different organic and inorganic compounds including organic solvents, ammonium hydroxide, and phosphoric acid, are utilized during the manufacturing processes (Bang et al., 2016).

Semiconductor wastewater that produced from fabrication facility is normally separated into three main streams: fluoride containing, acid base and chemical mechanical polishing. These separation practice is due to waste minimization purposes and pollution prevention. The wastewater that produced from semiconductor industry generally contains high levels of total ammonia nitrogen (TAN), fluoride (F-) (Aoudj et al., 2016) and phosphate (PO4-P). Research shows that, many contamination of heavy metal or metalloid has enter the water system from semiconductor manufacturing activity (Hsu et al., 2011, Rainbow, 2002, Bryan and Langston, 1992) The existing of the significant nutrient substances will induced water eutrophication which too much of nutrients in a lake or other body of water that causes a dense growth of plant life and death of animal life from lack of oxygen. As they occur in large quantities in the water bodies, large amounts of algae and microorganisms would breed, resulting in a higher dissolved oxygen depletion and fish toxicity (Huang et al., 2017, Amin et al., 2014).

This contaminant may trigger some of environmental issues especially fluoride and the concentration is more than 1000 mg/L (Guissouma et al., 2017, Aoudj et al., 2016). The excessive fluoride intake can result in bone disease (pain and tenderness of the bones) even though fluoride is one of the necessary elements of the human body, and children may get mottled teeth(Yadav et al., 2017). Continuing drinking of water holding high fluoride content can lead to the problem of softening bones, ossification of tendons and ligaments and several neurological damages in other cases(Näsman et al., 2016, Levy et al., 2014). These types of wastewater can cause groundwater contamination when enters to the surface water. According to World Health Organization (WHO), less than 1.5 mg/L is the safe approved fluoride level in drinking water.

Many techniques have been studied and practice to answer the problem of fluoride-containing semiconductor wastewater including chemical coagulation with calcium salts (Kaszuba et al., 2010, Attard et al., 2000, Kim et al., 2006); precipitation using poly-aluminium chlorides; adsorption onto montmorillonite (Ezzeddine et al., 2015, Tolkou and Zouboulis, 2015)or calcite (Erdemoğlu and Sarikaya, 2006); electrocoagulation (Krajewski et al., 1998, Liao et al., 2009, Xu, 2001, Hu et al., 2003); precipitate flotation (Xu and Deng, 2003) and reverse osmosis (Ström et al., 1985, Vandamme et al., 2013). The cheaper way to remove fluoride from industrial wastewaters by using chemical precipitation (Kachi et al., 2013). One of the studies is to investigate the simultaneous removal of fluoride (F-) from semiconductor wastewater by chemical precipitation using magnesium salts and which fluoride present could significantly inhibit the struvite crystallization (Huang et al., 2017). Suspended matter and fluoride are simultaneously eliminated by combining coagulation and electro-flotation (Aoudj et al., 2016). Study was also done on removal of fluoride and turbidity from semiconductor industry wastewater by combined coagulation with electro flotation and adsorption using activated clay (Ezzeddine et al., 2015).

Calcium chloride ($CaCl_2$) is the main focus as coagulant to remove fluoride for this research. Often together with flocculation Calcium chloride ($CaCl_2$) has a function as a coagulant or precipitant in the treatment process. Coagulation consist of charge neutralization in which a negatively charged pollutant will combines irreversibly with a cationic species (Ca^{++}). $CaCl_2$ reacts with fluoride to form insoluble calcium fluoride (CaF) which precipitation generates an unsolvable substantial that will resolves out (Aldaco et al., 2007). In order to enhance settling and filtration, flocculation agglomerates small charge-neutralized, coagulated and precipitated particles. Often used together with flocculants this coagulant has ability in reducing fluoride ions from wastewater generated by various material. Example of the material that contain fluoride is such as electroplating, glass, aluminium, steel, ceramic, phosphate rock, fertilizer, TV tube metal finishing, , and fluoride chemical sectors. Application of $CaCl_2$ in the industry because it provides pH adjustment and calcium ions. Recent year, add on lime to prepared calcium ions and adjust pH have been practiced by many industries. The highly soluble $CaCl_2$ provides more calcium ions than lime without increasing pH. An average about 50% of sludge decreasing, lower dewatering and scavenging costs when use $CaCl_2$ as a coagulant (Teh et al., 2016). However, $CaCl_2$ is common coagulant that use in the industry.

1.2 Problem Statement

Due to growing demand of semiconductor current years, the semiconductor industries have made considerable strides in development. However, this speedy development phenomenon has also trigger some environmental issues including the generation of large amount of wastewater. This thirsty industry used a vast amount of water during the semiconductor manufacturing process such as cleaning and rinsing wafer. Toxic dissolved fluoride pollution is a critical environmental problem for the semiconductor industry.

The nanoparticles released from diverse nanomaterials used in the semiconductor manufacture find their method over waste disposal routes into the wastewater treatment centers and turn out in wastewater sludge. Additionally the discharge of these nanoparticles into the effluent will pollute the aquatic and environment. For this reason, real understanding of the presence, behaviour and impact of these nanoparticles in wastewater and wastewater sludge is crucial and well timed. The increased use of nanomaterials introduces the nanoparticles purposely or not into the waste streams through wastewater treatment centers. Research has shown that these