

TREATMENT OF GALVANIC SLUDGE IN AN ARC THERMAL PLASMA
REACTOR

BALA ISAH ABDULKARIM

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Faculty of Engineering
Universiti Teknologi Malaysia

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DEDICATION

I hereby dedicate this Ph.D research work to Allah (SWT), my late Father Abdulkarim Ali (Manager), lovely wife (Hafsat Isah Ismail) and my children (Ali, Khadeejah, Sumayyah and Isah)

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ABSTRACT

The performance of arc thermal plasma in treating galvanic sludge was investigated. The inductively coupled plasma optical emission spectrometry analysis conducted to characterise the raw galvanic sludge reveals the presence of chromium (Cr), copper (Cu), iron (Fe), magnesium (Mg), manganese (Mn), lead (Pb) and zinc (Zn). Product gas was measured at varying applied current of 170 A to 190 A and residence time of 2 to 5 minutes. The fourier transform infrared spectrometry analysis of the gaseous product identified the presence of carbon monoxide (CO), carbon dioxide (CO₂), methane (CH₄), ethene (C₂H₄), ethyne (C₂H₂) and water (H₂O) in different concentrations. In general, the trend in CO and CO₂ concentrations initially shows an increase as the applied current was increased and thereafter decreases. Low concentrations of hydrocarbon were persistent, which is due to the low amount of hydrocarbon in the galvanic sludge. From the analysis the concentration of CH₄, C₂H₄ and C₂H₂ revealed were 46 ppm, 39 ppm and 73 ppm respectively. A quadratic model for CO concentration in the gaseous product was developed. The developed model using the response surface methodology showed a good fit with correlation coefficients of 99.63%. The optimum operating conditions for the treatment were 170.4 A, 20.13 L/min and 3.12 minutes for applied current, plasma gas flow rate and residence time respectively. The optimum response of CO obtained from the optimization of the reaction system was 1622 ppm with desirability of 1. Treatment at applied current of 170 A revealed metals were either within the toxicity leaching characteristic limit or not detected. The concentration of the metals found in the treated sludge were Cr (0.410 mg/l), Cu (0.350 mg/l), Fe (4.0 mg/l), Zn (0.8 mg/l) while magnesium, nickel and lead were not detected. The applied current of 170 A represents the minimum applied current sufficient for immobilisation of metals contained in the galvanic sludge. The economic analysis of arc thermal plasma for treatment of galvanic sludge reveals a profit margin of RM 5.89 per kg of sludge. The cost of arc thermal plasma treatment is RM 6.49 which is far cheaper than the cost of treatment (RM 27.811) using incineration. Furthermore, a positive return on investment of 5.81% would be realized in the fifth year of operation. Thermal plasma treatment technique is, therefore, a viable alternative method for treating galvanic sludge.

ABSTRAK

Prestasi plasma arka haba dalam merawat enapcemar galvanik dikaji. Analisis spektrometri pelepas plasma optik induktif yang dilaksanakan untuk mencirikan enapcemar galvanik mentah mendedahkan kehadiran kromium (Cr), tembaga (Cu), besi (Fe), magnesium (Mg), mangan (Mn), plumbum (Pb) dan zink (Zn). Produk gas diukur pada arus pelbagai dari 170 A hingga 190 A dan residence time 2 - 5 minit. Analisis transformasi fourier inframerah bagi produk gas menunjukkan kehadiran karbon monoksida (CO), karbon dioksida (CO₂), metana (CH₄), etana (C₂H₄), etyne (C₂H₂), dan air (H₂O) dalam kepekatan yang berbeza. Secara umumnya, trend kepekatan CO dan CO₂ pada awalnya menunjukkan kenaikan apabila arus yang digunakan meningkat dan kemudian berkurangan. Kepekatan rendah hidrokarbon adalah berterusan, disebabkan oleh jumlah hidrokarbon yang rendah dalam enapcemar galvanik. Dari analisis kepekatan CH₄, C₂H₄ dan C₂H₂ menunjukkan masing-masing 46 ppm, 39 ppm dan 73 ppm. Model kuadratik untuk kepekatan CO dalam produk gas telah dijana. Model yang dijana menggunakan kaedah sambutan permukaan menunjukkan kelekapan baik dengan pekali korelasi sebanyak 99.63%. Keadaan operasi optimum bagi rawatan adalah masing-masing pada 170.4 A, 20.13 L/min dan 3.12 minit bagi arus yang digunakan, kadar aliran gas plasma dan masa mastautin. Tindak balas optimum CO yang diperolehi daripada pengoptimuman sistem tindakbalas adalah 1622 ppm dengan keinginan 1. Rawatan pada arus 170 A menunjukkan logam berada dalam had prosedur penyingkiran ciri toksik atau tidak dikesan. Kepekatan logam yang ditemukan dalam enapcemar yang telah dirawat adalah Cr (0.410 mg/l), Cu (0.350 mg/l), Fe (4.0 mg/l), Zn (0.8 mg/l) manakala magnesium, nikel dan plumbum tidak dikesan. Arus pada 170 A mewakili arus minimum yang diperlukan untuk menghalang pemindaan logam yang terdapat dalam enapcemar galvanik. Analisis ekonomi plasma arka haba untuk rawatan enapcemar galvanik menunjukkan margin keuntungan sebanyak RM 5.89 per kg enapcemar. Kos rawatan plasma arka haba adalah RM 6.49 yang jauh lebih murah daripada kos rawatan (RM 27.811) menggunakan pembakaran. Di samping itu, pulangan positif pelaburan sebanyak 5.81% boleh direalisasikan pada tahun kelima operasi. Teknik rawatan plasma merupakan kaedah alternatif yang sesuai untuk merawat enapcemar galvanik.

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LIST OF ABBREVIATIONS

A	-	Ampere
Ar	-	Argon
AAS	-	Atomic Absorption Spectrophotometer
ASTM	-	American Standard Test Method.
CO	-	Carbon monoxide
Cu	-	Copper
Ba	-	Barium
CO ₂	-	Carbondioxide
CH ₄	-	Methane
C ₂ H ₄	-	Ethane
C ₂ H ₂	-	Ethylene
Cr	-	Chromium
DC	-	Direct Current
EC	-	Energy Consumption
EDX	-	Energy Dispersive Spectroscopy
EPA	-	Environmental Protection Agency
FC	-	Fixed Carbon
Fe	-	Iron
FE-SEM	-	Field Emission Scanning Electroscop Microscopy
FTIR	-	Fourier Transformed Infrared Spectroscopy
FWW	-	Forestry Wood Waste
GCV	-	Gross Calorific Value
H ₂	-	Hydrogen
H ₂ S	-	Hydrogen Sulphide
He	-	Helium
IBA	-	Incineration Bottom Ash
IC	-	Inorganic Carbon
ID	-	Internal Diameter
ICP-OES	-	Inductively Coupled Plasma Optical Spectrometry
LC	-	Leachate Concentration

Mrs	-	Mass Of Raw Galvanic Sludge
M _S	-	Mass Of Solid Product
Mn	-	Manganese
Mg	-	Magnesium
MJ	-	Mega Joule
MSW	-	Municipal Solid Waste
MT	-	Metric Tonne
Ni	-	Nickel
NO	-	Nitric Oxide
NO ₂	-	Nitrogen Dioxide
NO _x	-	Oxides Of Nitrogen
OD	-	Outside Diameter
P	-	Power
PAED	-	Pulsed Arc Electrohydraulic Discharge
Pb	-	Lead
PARF	-	Plasma Application Research Facility
ppm	-	Part Per million
RGS	-	Raw galvanic sludge
SEM	-	Scanning Electron Microscopy
Si	-	Silicon
SO ₂	-	Sulphur (IV) Oxide
SW	-	Scheduled Waste
TC	-	Total Carbon
TCLP	-	Toxicity Characteristics Leaching Procedure
TGA	-	Thermogravimetric Analysis
TIG	-	Tungsten Inert Gas
TOC	-	Total Organic Carbon
V _{RS}	-	Volume of Raw Galvanic Sludge
VM	-	Volatile Matter
WPS	-	Wet Paper Sludge
WPC	-	Westinghouse Plasma Corporation
WW	-	Waste Water

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Galvanic sludge is generated in the process of applying a protective zinc coating to iron or steel so as prevent rusting and improve the lifespan of metallic material. The method of galvanizing mostly applied is the hot dip galvanizing, whereby Zinc coating is produced on the material by immersion of material in a bath of molten zinc at a temperature of about 450 °C. This is applied for different type of material ranging from small metal piece to very large structural shapes. The constituent of the galvanic sludge depend on the installed process, but basically it is a heavy metal (chromium, nickel, zinc, cadmium, tin, lead, copper, etc.) laden waste that is toxic and hazardous to the environment (Nikolay and Maria, 2017a; Rossini and Bernardes, 2006).

Majorly employed thermal method for treatment of sludge includes carbonization of dehydrated sludge (Kato *et al.*, 2005), low temperature pyrolysis (Shen and Zhang, 2005; Wang *et al.*, 2007) and gasification of dried sludge (Dogru *et al.*, 2002; Murakami *et al.*, 2006; Werle, 2015a, 2015b) but these technologies are associated with the production of secondary solid waste and/or product gas containing CO₂, dioxins and furans. Most recently thermal plasma technology which is a short residence time process and environmentally friendly waste management technique is been applied to handle large volume of hazardous industrial sludge. Plasma technology basically involves the creation of a sustained electrical arc by passing electric current through a gas in a process referred to as electrical breakdown. Since electrical resistivity exist across the system, significant heat is generated, which strips away electrons from the gas molecules resulting in an ionised gas stream, or plasma.

Air is the most common gas used, but several inert gases such nitrogen (N₂), Helium (He) and Argon (Ar) have been widely used (Hrabovsky, 2002; Watanabe and Shimbara, 2003). In order for air to conduct electricity, it must be subjected to a large differential in electrical potential. This is done between two electrodes which are separated by air. When this potential is large enough, electrons can be pulled from the normally neutral molecules in the air. These electrons then move with the electric field and impact other molecules, releasing more free electrons at an exponential rate. This phenomenon is called an electron cascade and once enough electrons are moving with the electric field, an arc is created between the electrodes. At temperatures of about 3000 °C gas molecules lose electrons and become ionised (Auciello and Flamm, 1989). There are basically two types of plasma for industrial processes: thermal or (equilibrium) plasmas and non-equilibrium plasmas (Boulos, 1991; Kogelschatz, 2004).

Thermal plasma has advantages of high temperature, high intensity, non-ionizing radiation and high energy density. According to Tandero *et al.*, (2006) thermal plasmas can reach temperatures of above 20,000 K but the equilibrium temperature utilised in treatment of the waste could be far less. The advantages of thermal plasmas are employed in many industrial applications. Plasma gases were used to simulate high temperature conditions similar to those when missiles re-enter the atmosphere. Today thermal plasma technology covers a wide spectrum of applications such as thermal plasma coating techniques (plasma spraying and plasma chemical vapor deposition), thermal plasma synthesis of fine powders, thermal plasma metallurgy and thermal plasma waste destruction.

According to Heberlin and Murphy (2008) the approach for plasma treatment of different types of waste may consist of the following; thermal break down of chemical components without oxidation which is called plasma pyrolysis, the incomplete oxidation of organic fraction of waste and generation of syngas and other gases which can be used to generate electric power or drive steam turbines known as plasma gasification, compaction and vitrification of solid wastes by gasifying organic material, melting inorganic material and binding hazardous metals in a ceramic matrix (e.g. a silicate). The integration of plasma pyrolysis, compaction and

vitrification process or a combination of plasma gasification, compaction and vitrification can be applied for solids with high fractions of organic.

In this research, arc thermal plasma was applied to treat galvanic sludge. A plasma field created by directing an electric field through a low pressure gas stream generate high temperatures. The intense high temperature of between 1700 °C and 2200 °C achieved at varying applied current and time can be used to dissociate waste into their atomic element in the reaction chamber. Heat generated from the plasma torch can melt and vitrify the inorganic portion of the galvanic sludge while the organic components can be vaporized and decomposed by the intense heat.

1.2 Problem Statement

Large volume of heavy metal containing sludges is generated globally, and galvanic sludge arising from galvanizing processes is a major contributor. Galvanic sludge is a category of Scheduled Waste (SW) refer to as SW105 in Malaysia that is hazardous and no longer allowed to be disposed directly to landfill. The sludge majorly constitutes heavy metals (Emine *et al.*, 2013; Kobya *et al.*, 2015). Apart from the presence of heavy metal found in the sludge, it also contains, surfactants, oil and grease, hydroxides and hydrate oxides.

The disposal of untreated or not properly managed heavy metal containing sludge such as galvanic sludge has various environmental, human and health effect. Excess levels of heavy metals introduced to soil by sludge can lead to elevated uptake of heavy metals by plants. This may cause irreparable damage to the plants. It can also affect human health upon the consumption of the plants since these plants consist of accumulated levels of the heavy metals within their tissues. The heavy metal can also lead to underground water pollution. Incineration is the most common treatment method applied to hazardous waste such as galvanic sludge. This method reduce the sludge volume for final disposal, however it has a lot of drawback. It does not completely destroy the metals in the sludge, which means that the metals in the sludge will remain and still pollute the environment. It also generate hazardous fly

ash and bottom ashes that requires further treatment.(Cedzynska *et al.*, 1999; Chun-Teh *et al.*, 2007). It also generate fly ash and bottom fly ash that are not free of toxic substances which will still require landfilling. The chemical methods of treatment has the major problem of waste separation and large consumption of reagents while ion exchange is only effective when dealing with low concentration of heavy metals (Nikolay and Maria, 2017b). Therefore, there is the need for a treatment options such as thermal plasma that will convert the remnant waste into inert or harmless material.

Thermal plasma technology has the ability to efficiently detoxify hazardous waste such as galvanic sludge. This has been proven by previous researches conducted by (Bień *et al.*, 2013; Chang *et al.*, 2008; Cubas *et al.*, 2014b; Gomez *et al.*, 2009; Li *et al.*, 2012; Li *et al.*, 2015a; Szente *et al.*, 1998) have proven thermal plasma technology to be an efficient means of detoxification of the sludge but notwithstanding the treatment process comes along with gaseous product (Syngas) that has gotten no research attention. The only researches so far conducted on treating galvanic sludge using thermal plasma technology focused on inertisation and vitrification (Cubas *et al.*, 2014b). Presently, there is no documented research investigation on the effect of process variable on the gaseous product from thermal plasma treatment of galvanic sludge. It is therefore the intention of this research to investigate the effect of process variable on gaseous product and level of inertisation of slag (solid product) obtained from treatment of galvanic sludge. The immobilisation of the metals in the solid product can be achieved as a result of the exposure of the galvanic sludge to the high temperature from thermal plasma. The high temperature renders non-heavy metals which are non-volatile species chemically bonded and forms a non-leachable solid matrix.

1.3 Research Aim and Objectives

- i. To characterised galvanic sludge prior to thermal plasma treatment.
- ii. To determine the effect of applied current and residence time on solid and gaseous product.

- iii. To optimize CO in the gaseous product using surface response methodology (RSM).
- iv. To conduct economic analysis on the treatment of galvanic sludge in an arc thermal plasma.

1.4 Scope of Research

The scope of this research includes the collection of galvanic sludge from KISWIRE Sdn Bhd. The characterization of the galvanic sludge to determine the moisture, volatiles, ash content, fixed carbon, total organic carbon (TOC), elemental composition and morphological structure. The designing and fabrication of the arc plasma reactor. The analysis of the solid and gaseous to determine the total organic compound, heavy metal composition, leachability and composition of the gaseous product. Finally, Optimization and economic analysis of the laboratory arc thermal plasma.

1.5 Significance and Contribution of the Research

Recently, galvanic sludges are treated in incinerators in Malaysia. This method does not completely immobilise heavy metals. It also generate fly and bottom ashes that are toxic to the environment. It also generate fly ash and bottom ashes that requires further treatment. Thermal plasma technology can efficiently treat galvanic sludges. It can transform the sludge into non-toxic and harmless materials. Investigations on effect of applied voltage and residence on the composition of gaseous product from treatment of galvanic sludge in thermal plasma will provide data and information that will help safeguard human and the environment against toxic pollution arising from galvanic industries. The developed thermal plasma system is expected when upgraded to pilot or industrial scale to serve as an alternative treatment technique which does not give rise to secondary pollutant and reduce the large volume of heavy metal containing sludge and other related hazardous waste such as Scheduled Waste in Malaysia.

1.6 Organisation of Study

This research work conducted consist of five chapters as follows:

Chapter One is the introduction: The chapter covers introductory background of the research. It also critically outline the aim and objectives, the scope and significance of the research.

Chapter Two is the literature review: The chapter, basically focus on the relevant previous researches to the study area. The principle of thermal plasma technology and its application to waste treatment.

Chapter Three is the methodology of research: This chapter covers procedure on sample collection and preparation obtained from KISWIRE Sdn Bhd, description of process equipment, methods of characterisation of galvanic sludge, procedure on thermal plasma treatment of galvanic sludge in thermal plasma system. It also entails characterisation of gaseous and solid product.

Chapter Four is results and discussion: This chapter present results and elaborate discussion of experiment and analysis done in this research. It documents results and discussion on characterisation of galvanic sludge and products from thermal plasma system, effect of applied current and residence time on the composition of gaseous products, leaching test analysis, optimisation and economic analysis of the treatment process.

Chapter Five is the Conclusion and recommendation: In this chapter important inferences were reached based on the results and the discussion. Possible Recommendation on future work was suggested.

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