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Characterisation of Galvanic Sludge from Hot Dip Galvanising Process for Metal Surface Treatment

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ABSTRACT

Galvanic sludge is generated from Hot Dip Galvanizing (HDG)process during metal surface treatment. The sludge is a heavy metal laden waste that require efficient treatment to meet up with increasingly stringent environmental regulation, but this cannot be achieved without proper investigation into the properties of the sludge. This research work characterised galvanic sludge from KISWIRE Sdn Bhd so as to determine the physiochemical and other relevant properties of the sludge prior to treatment in a direct current transfer arc low thermal plasma. The characterisation is required to determine the extent of treatment using thermal plasma technique. The result of proximate analysis reveals the galvanic sludge to have 67.055%, 11.485% 19.30%, and 2.16% (as air dry basis) of moisture content, volatile matter, ash content andfixed carbon respectively. The brownish galvanic sludgehas a density of 1.396g/cm^3 andgross calorific value of -21.4cal/g. Total Organic Carbon (TOC)of thegalvanic sludge is 2.073%, while Total Carbon (TC) and Inorganic Carbon are 2.094% and 0.02031% respectively. Elemental (metal) analysis of the galvanic sludge indicates the presence of twelve metals (Al, Cd, Cu, Cr, K, Mg, Mn, Ni, Zn, Pb, Si, and Fe)of differentconcentration. Copper and lead with concentrations of 5793.44 mg/kg and 420mg/kg respectively wereabove the US EPA standard, 1993.The very high concentration levels of iron (59290.6 mg/kg) and zinc (5411.22mg/kg) also reflects the nature of the process activity of KISWIRE Sdn Bhd.

***Keywords**: Galvanic Sludge, MoistureContent, Heavy Metals, Ultimate Analysis*

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I. INTRODUCTION

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[1, 2]. The method of galvanizing applied in KISWIRE Sdn Bhd is the hot dip galvanizing, whereby Zinc coating is produce on the material by immersion of material in a bath of molten zinc at a temperature of about 450^{o}C[3].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), cyanide, surfactants, oil and grease, hydroxides and hydrate oxide [4, 5].

In the year 2012, 173837.06 MT/ Year of heavy metal containing sludges was generated in Malaysia and galvanic sludge arising from galvanizing processes is a major contributor[6]. Galvanic sludge is a category of schedule waste refer to as SW105 in Malaysia[7]. This category of waste is hazardous and no longer allowed to be disposed directly to landfill. Traditionally galvanic sludges are treated in incinerators in Malaysia. This method does not completely destroy heavy metals and other toxic substances. It also generate hazardous fly ash and bottom ashes that requires further treatment[8, 9].Characterisation of the galvanic sludge will provide informations on its physiochemical properties and relevant information needed foroptimal treatment using thermal plasma technique.

II. EXPERIMENTAL PROCEDURE

2.1 Collection and Preparation of Sample

Dark brownish galvanic sludge from filter press was collected from the KISWIRE Sdn Bhd, manufacturers of steel wire and other related products. It was stored in a properly labelled airtight polythene bag in order to prevent loss of moisture to the atmosphere and to retain galvanic sludge sample in its original state.

2.2 Characterisation of Sample

In conducting proximate analysis, moisture content, volatile matter, ash, fixed carbon and heating value was determined according to ASTM D3173-11, ASTM D3175-11, ASTM D3174-11 and ASTM D3172-07a respectively[10].The ultimate analysis method determine carbon, hydrogen,nitrogen, oxygen and sulphur (CHNOS).The presence and concentration of carbon hydrogen and nitrogen was determined using ASTM standard procedure (ASTM D5373) while the sulphur content by ASTM D4239 [11]. The difference is basically the result for oxygen content of the galvanic sludge. The analysis was carried out using *VarioMICRO V3.1.1* cube elemental analyser.

The gross calorific value of the galvanic sludge was determined according to ASTM D5865-10a, Standard Test Method for Gross Calorific Value usingIsoperibol bomb calorimeter*(LECO Model AC- 350)*. One (1) gram of the galvanic sludge was weighed into a bomb cup and is placed in a sample holder within the combustion bomb. The combustion bomb was charged to 500 psig with pure oxygen and submerged in a known volume of water. Sludge was ignited and the heat imparted to the water is used to calculate the calorific value of the sample. Pycnometer (*AccuPyc 1330*) determined the density of the sludge by measuring the pressure change of helium in a calibrated volume.Total Organic Carbon (TOC) was measured using Total Organic Carbon sample Analyzer (*SSM-5000A)*. Heavy metals concentrations were analysed by digesting five (5) grammes of sludge in7ml of 65% HNO_3 and 1ml of 30% H_2O_2 in a microwave oven digester prior to analysing in Inductively Coupled Plasma Optical Emission Spectrophotometer *(ICP-OES) Agilent model 710*.

III. RESULT AND DISCUSSION

3.1 Proximate and Ultimate Analysis

The result of moisture content, ash, volatile matter and fixed carbon of the galvanic sludge is shown in Table 1.

Table 1. Proximate analysis of galvanic sludge

Test parameter	Moisture Content Test method: ASTM D3173 - 11	Ash Test method: ASTM D3174 - 11	Volatile Matter Test method: ASTM D3175 - 11	Fixed Carbon Test method: ASTM D3172 – 07a
Result (%) (as Air dry basis)	67.055	19.30	11.485	2.16

The density and Gross Calorific Value (GCV) of the sludge are 1.396g/cm^3 and -21.4cal/g respectively. The high percentage water content (67.055%) of the galvanic sludge is close to the values of 64.35% and 68% reported by [12-14]. The ultimate analysis (CHNOS) shows the galvanic is made up of 0.0984%, 1.8260%, 2.1256%, 0.4076% and 95.5424% of nitrogen, carbon, hydrogen, sulphur and oxygen respectively. The result is presented in Table 2. The low values of nitrogen and sulphur found in the galvanic sludge are also similar to values of N; 0.17%; S 0.26% obtained by [15].This indicate the low possibility of formation of NO_x and SO_x which are poisonous gases. Result of Total Organic Carbon (TOC) analysis reveals galvanic sludge to have 2.094% Total Carbon (TC), 2.073% Total Organic Carbon and 0.02031% Inorganic Carbon. The TOC (2.073%) is also higher than 1.73% of galvanic sludge characterised and treated in direct current thermal plasma by*Leal Vieira Cubas* and co- researchers[12].

Table 2: Ultimate analysis of galvanic sludge

Elements	C	H	N	S	O
Test results (wt %)	1.8260	2.1256	0.0984	0.4076%	95.5424
Galas et al 2016 (wt %) [15]	10.03	1.12	0.17	0.26	88.15
Pérez-Villarejo *et al; 2015* (mass %) [16]	8.758	2.253	0.111	0.133	88.745

3.2 Elemental Analysis

Heavy metal levels in the galvanic sludge was determined using Inductively Coupled Plasma Optical Emission Spectrophotometer (ICP-OES).The result (Table 3) of metal concentration in the galvanic sludge indicates that cadmium, chromiumand nickelhaving concentrations of 2.8708mg/kg, 96.5602mg/kg and 17.558mg/kg respectively are below the US EPA Standard, 1993, while copper and lead with concentrations of 5793.44 mg/kg and 420mg/kg respectively were above standard.

Table 3. Concentration of metals in galvanic sludge

Heavy metals	Concentration (mg/kg)	US EPA Standard, (1993).
Al	54.626	-
Cd	2.8708	85
Cr	96.5602	3000
Cu	5793.44	4300
Fe	59290.6	-
K	322.104	-
Mg	47.636	-
Mn	565.754	-
Ni	17.558	75
Pb	620.046	420
Si	10.2834	-
Zn	5411.22	7500

The very high levels of copper, iron and zinc reflects the nature of the activity of KISWIRE Sdn Bhd, majorly dealing with the production of different kind/types of wire (galvanised steel wire, oil tempered wire, spring wire, hose wire, bridge wire, superconductive wire and tire reinforcement).

3.3 Thermogravimetric Analysis

There is a rapid increase in percentage weight loss between the temperature of 25°C and 150°C, a weight loss of about 75% was attained as indicated in the Thermogravimetric Analysis (TGA) curve in Figure 1. The derivative weight loss was also rapid at 25°C to 100°C but decreased sharply between 100°C to 150°C. This may be as a result of disappearance of highly volatile substance and remnant moisture. This stage represent moisture content removal.

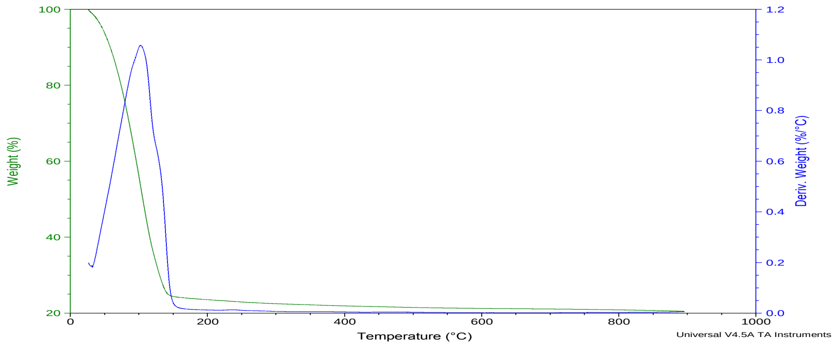
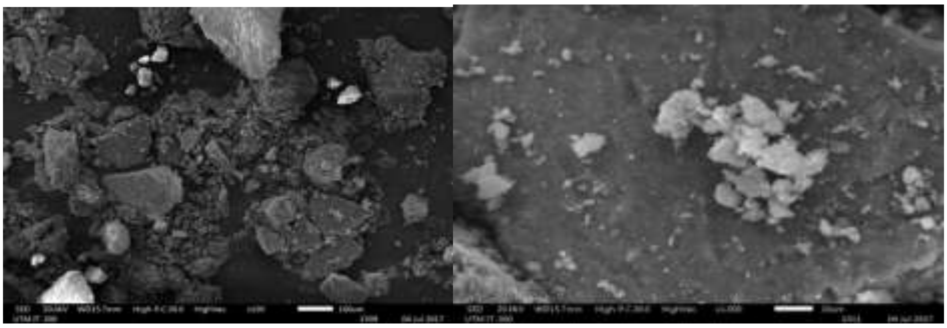


Figure 1: Thermogravimetric Analysis (TGA) of galvanic sludge.

The decomposition of sludge and complete removal of volatile matter is represented between temperatures of 150°C to 900°C. It can be concluded from the TGA that a total weight loss of 80% was attained and beyond 900°C there was no further loss in weight and this account for ash content of the galvanic sludge.

3.4 Scanning Electron Microscope

The images of galvanic sludgein Figure 2,captured with variable pressure scanningelectron microscope at x300and x1000 magnification shows the surface topography of the galvanic sludge is non-uniform, complex and irregular shapes which is similar to images capture by work done by [15].



A **B**

Figure 2: SEM images of galvanic sludge at two different magnifications.(A) SED 20kV, WD 15.7mm, (X 300), (100 µm), (B) SED 20kV, WD 15.7mm, (X 1000), (10 µm)

IV. CONCLUSION

Galvanic sludge obtained from KISWIRE Sdn Bhd is a heavy metal laden sludge. It consist majorly of high concentration of Cu (5793.44mg/kg), Pb (420 mg/kg), Fe (59290.6mg/kg) and Zn (5411.22 mg/kg) thatrequires efficienttreatmenttechnique such as thermal plasma torender harmless prior to disposal or reuse. It has a moisture content, volatile matter and fixed carbon of 67.055%, 11.485%, 19.30%, and 2.16% (as air dry basis) respectively. TheSludge is non-uniform, irregular and of low carbon content (1.8260 wt %) compared to other characterised galvanic sludge found in the literature.

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