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CHARACTERIZATION OF SEWAGE SLUDGE FOR SUSTAINABLE URBAN ENVIRONMENTS: ASSESSING HEAVY METAL ENRICHMENT, THERMAL DECOMPOSITION, AND PYROLYSIS BEHAVIOR

Mohd Syazwan Mohd Ghazali[1]*, Syed Shatir A. Syed-Hassan[3]
***Corresponding Author**
[1]Section of Environmental Engineering, Universiti Kuala Lumpur, Malaysia Institute of Chemical and Bioengineering,
78000 Alor Gajah, Melaka, Malaysia
[3]School of Chemical Engineering, College of Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia

msyazwan@unikl.edu.my[1], shatir@uitm.edu.my[3]
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ABSTRACT

Rapid urbanisation and population growth have led to a surge in sewage sludge (SS) generation, posing environmental challenges due to its complex composition, particularly heavy metal content. This study investigates heavy metal analysis, thermal decomposition behaviour, and slow pyrolysis of SS to address these concerns. SS samples were collected from a sewage treatment plant and analysed for heavy metals using ICP-OES. Thermogravimetric analysis (TGA) clarified thermal decomposition behaviour, while slow pyrolysis experiments at varying temperatures provided insights into product yields. Results revealed significant concentrations of heavy metals and other elements in SS, with pyrolysis temperatures between 450°C and 600°C ensuring complete conversion of volatile matter. Slow pyrolysis predominantly yielded biochar, indicating limited suitability for bio-oil production. Additionally, pyrolysis enriched biochar with heavy metals while decreasing cobalt concentration, highlighting complex mechanisms in heavy metal redistribution. This research contributes to understanding heavy metal behaviour and thermal conversion dynamics in sewage sludge, informing sustainable waste management strategies for mitigating *environmental impacts.*

Keywords: *Sewage sludge, Pyrolysis, Heavy metals*

INTRODUCTION

Sewage sludge (SS) refers to a mixture of solid or liquid waste generated during the partial or complete sewage treatment process, but it does not include the treated sewage discharged (Laws of Malaysia, Act 655, Water Services Industry Act, 2006). Rapid urbanisation and global population growth are resulting in an upsurge in SS generation. Nowadays, SS becomes a huge environmental burden, estimated to be produced globally in the amount of over 45 million dry tons yearly (Jellali et al., 2021; Mohd Ghazali et al., 2024). In Malaysia, according to a report by Indah Water Konsortium Sdn Bhd (IWK), approximately 250 tons of sewage (biosolids) are produced daily (Wealth, 2019). This is related to overcoming the increased amount of waste, precisely SS, due to the rising population (Mustafa et al., 2022). Hence, being environmentally conscious, or "green," entails caring for our surroundings (Anis et al., 2023). In addition to its quantity, the complexity of SS presents a significant challenge. SS comprises a blend of undigested organics (human waste, plant residue, paper, oils, food, and faecal material), microorganisms, and inorganic material (Trinh et al., 2013). Despite the potential of sewage sludge as a resource for energy recovery and soil amendment due to its carbon-rich material (Roslan et al., 2023; Syed-Hassan et al., 2017). On the other hand, SS contains a significant amount of inorganic material, particularly ash and metals. Hence, its utilisation is limited by concerns related to heavy metal contamination and the efficiency of conversion processes.

SS intrinsically contains harmful substances such as pathogens, pharmaceuticals, hormones, heavy metals, and persistent organic pollutants (Syed-Hassan et al., 2017). Due to this, the application of sewage sludge becomes regulated by the legislation of the European Union (EU, Directive 86/278/CEE), which aims to qualify sewage sludge (Fonts et al., 2012; Trinh et al., 2013). This concern stems from the potential hazards that SS poses to human health and our environment's delicate balance, as noted by Sánchez et al. (2009). Exposure to sewage sludge can lead to various health risks, including the spread of disease and toxic contamination related to accumulating heavy metals (Moran, 2018; Rorat et al., 2019). Hence, it highlights that SS needs special attention to ensure it complies with environmental sustainability.

The thermochemical conversion process offers a further approach of managing SS (Syed-Hassan et al., 2017), enabling effective volume reduction and conversion of the carbon-rich organic fraction into valuable energy and fuel. Pyrolysis, in particular, emerges as a promising method for sewage sludge treatment. One compelling reason supporting this assertion is that pyrolysis represents a zero-waste approach with significant potential for addressing wastewater management challenges (Samolada & Zabaniotou, 2014). Exceptionally, the process may extract the benefit from the decomposition of organic matter and massive volume reduction, and even reduce the adverse effects with sterilisation of pathogens and stabilisation of heavy metals (Liu et al., 2018). Thus, recently pyrolysis has been the predominant mode among researchers. While the presence of heavy metals in sewage sludge is well-documented, there is a lack of clarity regarding how these metals migrate within the sludge matrix during thermal conversion processes.

The paper aims to investigate two significant aspects, the heavy metals analysis of the SS and the thermal conversion behaviour of the SS via slow pyrolysis. Through the analyses, it exposes the complex interplay between heavy metal migration and the thermal decomposition behaviour of sewage sludge during slow pyrolysis. Heavy metal migration was conducted using inductively coupled plasma optical emission spectroscopy (ICP-OES), while the thermal conversion behaviour of the SS was investigated through slow pyrolysis, employing thermogravimetric analysis (TGA). Furthermore, pilot-scale pyrolysis experiments were conducted to analyse the distribution of products resulting from the thermal conversion process.

METHODOLOGY

Sewage Sludge Collection

The sample was collected from the centralised sewage treatment plant located in Sungai Udang, Melaka. The collected sample was dried in the oven at 105 ºC for 24 h and subsequently it was crushed in powder form. The sample was sieved to obtain the homogenous form at size 0.2 mm. Table 1 shows the proximate and ultimate analysis of sewage sludge.

Table 1. Ultimate and Proximate Analysis of Sewage Sludge

Sample		Sewage Sludge [a]
Ultimate analysis (wt. %)	C	24.7
	H	2.7
	N	7.1
	S	0.9
	O [b]	17.3
Proximate Analysis (wt. %)	FC [c]	15.7
	VM	37.0
	Ash	47.3

[a] Dry basis
[b] Determined by difference.
[c] FC=100-(Ash+VM)
Source: Author

Metallic and Non-metallic Elements

For the elemental analysis, the samples were first acid digested according to the SW 846 method. After digestion, the elemental analysis was carried out using an inductively coupled plasma–optical emission spectrometer (ICP-OES); model Perkin-Elmer Optima 2000 DV as described in SW 846 Method 6010C, which provided guidelines for determining multiple elements using ICP-OES.

Thermal Decomposition

Thermogravimetric (TG) measurements of the SS were performed on a TGA-DSC analyzer (TGA-DSC 1 STAR, METTLER TOLEDO). A sample weight of 10.0 mg ± 0.5 mg was used for the TG analysis, which was conducted under a nitrogen flow rate of 15 mL/min and a heating rate of 10ºC/min from 30 ºC to 900 ºC (Ghodke et al., 2021).

Pyrolysis of Sewage Sludge

Pyrolysis experiment performed with 10 g of SS sample at temperature; 550°C, 650°C, and 720°C at constant heating rate of 35 °C/min. Sample was placed into the reactor. The nitrogen gas was injected to purge the air and create an oxygen-free condition. Reactor was placed into the furnace. Three series of the tar traps were installed to maximise condensation capability for trapping the bio-oil shown in Figure 1. During the heating process, the nitrogen gas was continued to supply into the reactor at flow rate 0.5 L/min. As it achieves the reaction temperature at 550°C, 650°C and 720°C, heating of the reactor remains for 1 hour and the process is stopped once the gas released is finished. The solid residue or biochar was collected after 12 hours of reaction complete. To determine the product distribution, the weights of biochar and tar collected from the tar trap will be measured. For the gas, we use the following formula:

$$\text{Gas (wt. \%)} = 100\ \% - \text{Biochar (wt. \%)} - \text{Tar (wt. \%)}$$

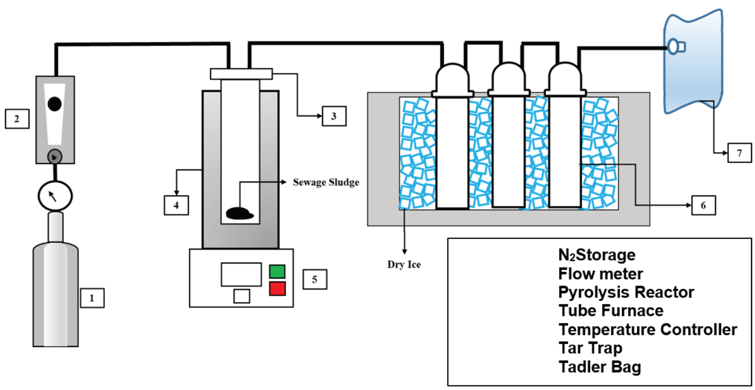

Figure 1. Equipment Configuration for Pyrolysis of Sewage Sludge
Source: Author

RESULT AND DISCUSSION

Metallic and Non-metallic Elements

Table 2 shows the metallic and non-metallic elements in SS. This provides the idea on pollution concerns of inorganics, expected to be generated and also potential of inorganics in participating in pyrolysis (Chan & Wang, 2016). The data show that SS contains various heavy metals and high in alkali and alkali earth metals (AAEM) such as calcium, potassium and sodium. The content of other inorganics such as aluminium, iron, magnesium, phosphorus and zinc also considerably high with the value more than 500 mg.kg-1. The highest amount of inorganic was the aluminium (688,047 mg.kg-1), undoubtedly from the chemical used as the coagulant for separation between water and SS. The source of this inorganics is mostly from industrial premises and also agricultural farms. Unfortunately, the predominant concerns(Zain et al., 2002) regarding SS; it that lead to hygienic concern (Gao et al., 2014; Irena Twardowska, 2005) and environmental issue despite it contains decent constituents that can be used as fertiliser (Frišták et al., 2018; Sommers, 2010) like phosphorus calcium (Frišták et al., 2018). The data prove the highly toxic metals such as lead, chromium, and arsenic exist in SS with trace amount. The data significantly inform their value can limit or even prevent spreading on the land (Canziani & Spinosa, 2019).

Table 2. Inorganic and Heavy Metals in the Sewage Sludge

Metals	SS (mg.kg-1)	Metals	SS (mg.kg-1)
Alkali Metals		Manganese	159
Calcium	10,321	Molybdenum	11
Potassium	12,250	Nickel	22
Sodium	1,217	Silver	<0.2
Alkali Earth Metals		Tin	48
Barium	337	Titanium	343
Cadmium	1.4	Vanadium	86
Magnesium	1,902	Zinc	692
Strontium	3.1	*Post Transition Metals*	
Transition Metals		Aluminium	688,047
Cobalt	< 0.7	*Metalloids*	

Chromium	73	Antimony	6.4
Copper	139	Arsenic	50
Iron	24,113	Silicon	3,217
Lead	37	*Non-metals*	
		Phosphorus	5,500

Source: Author

The amount of silicon, aluminium, iron, calcium, and phosphorus were present in relatively higher amounts, more than 3,000 mg.kg-1. All of these inorganics were major ash forming elements. For that reason, the ash content in the sample was significantly high (47.3% from Table 1). Conversely, AAEM such as potassium, magnesium, sodium and calcium might act as catalysts in some reactions that take place in the pyrolysis process (Fonts et al., 2008). According to Vamvuka et al., (2006) some mineral matter also can change pyrolysis kinetic parameters of materials. For example alkali metals such as calcium and sodium could also provoke a reduction in the yield of organic compounds, promoting the gas yield (Ruiz-Gómez et al., 2017).

Thermal Decomposition

Figure 2 exhibits the TG analysis and differential DTG curve for the sewage sludge. The total loss or remaining mass of SS is 40% of the original mass at the final temperature of 900°C. The information is significant in reviewing on several characteristics, such as reaction temperatures, devolatilization rates, and mass fractions (Grønli, 2002), with a particular emphasis on their correlation with the organic content within SS.

The curve indicates three stages of decompositions, where the first decomposition occurs at the temperature between 50 ºC and 150 ºC. The condition was in line with the review work by Syed Hassan et al. (2017) where it refers to the evaporation of embedded moisture (100–200°C) (Syed-Hassan et al., 2017). The major decomposition occurs in a range of temperature 200ºC and 550ºC, which was comparable with the previous study by Gao (2014). This region where three phases of the decomposition took place, including the decomposition of easily biodegradable organic matter and dead organisms (occur at 200 ºC and 300 ºC). In range of 300ºC and 450ºC the phase of breakdown for organic polymers from natural polymers in SS or proceeding from their biological stabilisation and at temperature over 450 ºC where the breakdown of the difficult biodegrade organics such as cellulosic materials (Syed-Hassan et al., 2017). The final decomposition occurs at temperature over 600 ºC, this decomposition may be attributed to calcium carbonate and inorganic matters decomposition (Liu et al., 2018; Ruiz-Gómez et al., 2017).

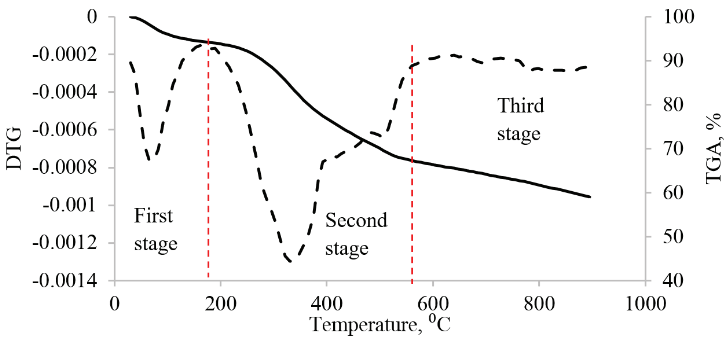

Figure 2.Thermogravimetric (TG) Analysis and Derivative Thermogravimetric (DTG) Curve for Sewage Sludge at Heating Rate of 10 min.s-1 in Nitrogen Ambience

Although inorganics may not be directly associated with TGA, the temperature of pyrolysis provides critical understanding into the migration patterns of heavy metals. Therefore, the data imply to keep the pyrolytic temperature between 450°C and 600°C to ensure that the volatile matters are completely converted into condensable and non-condensable gas phases (Liu et al., 2018). The remaining solid residue can be considered as biochar (Racek et al., 2020), which estimate 60 % of the original mass.

Pyrolysis of Sewage Sludge

The main product for slow pyrolysis was the biochar, where the value recorded more than 50% for all the temperature variation. The value was corresponding with the thermogravimetric analysis (Figure 2), where the value of solid residue remains at 60%. The high value of char yield was contributed due to the high ash content in SS (Alvarez et al., 2015; Fonts et al., 2012). This value is consistent with previous discussion, as the proximate analysis (Table 1) of SS indicates the high amount of ash and the inorganic data show the high number of ash contributing elements such as phosphorus, aluminium, iron and calcium contained in SS.

Table3. Product Yields for Pyrolysis of Sewage Sludge at Temperature; 550 ºC, 650 ºC and 720 ºC (under slow pyrolysis condition)

	Yields (wt. %)		
Temperature	Char	Tar	Gases
550 ºC	66.0	3.0	31.0
650 ºC	61.4	5.2	33.4
720 ºC	55.8	2.6	41.6

Source: Author

Table 3 shows the product yield for pyrolysis of sewage sludge. The maximum char yield was recorded at temperature 550ºC with the value at 61.4%. The char yield decreased accordingly with the increase of the pyrolysis temperature, which was 55.8% at 650ºC. This happens due to devolatilisation of organic matter at higher temperature to produce gas and liquid products (Xie et al., 2014). The decrease in biochar yield is possibly related to further pyrolysis conversion (Agrafioti et al., 2013) either related to a greater primary decomposition of the initial feedstock or to secondary reactions of the solid residue.

Besides biochar, the yield of the gases obtained is more than liquid. This due to the heating rate of the process at 35ºC/min classified as slow pyrolysis (operate at below 100 ºC/min of heating rate). Slow pyrolysis of biomass produces mainly biochar and pyrolysis gas because of the slower heating rates and longer process times, which converts most condensable organic compounds to solid carbon, light gases and condensable liquids (mostly water, carboxylic acids, and aldehydes) (Brown et al. 2011). The graph indicates the yield of gases increase with the increase of temperature and the maximum yield obtained at temperature 720 ºC with the weight percentage about 41.60%. The liquid product obtained quite low between 5.2% and 3.0% for respectively temperature, which is the least obtained product. Hence, the slow pyrolysis mainly focuses on the carbonization and is unsuitable for collecting bio-oil due to low yield.

Effect of Operating Variables on Heavy Metals Removal

Table 4 shows the total concentration of heavy metals remaining in the biochar. It is clear that there is a significant enrichment of heavy metals in the sample after the pyrolysis process. The increase was particularly significant for several heavy metals, including chromium, copper, nickel, tin, titanium, and zinc. This demonstrates a significant increase in the concentration of these metals after pyrolysis. This assertion is supported by Praspaliauskas et al. (2018), who noted that the pyrolysis process indeed amplifies the enrichment of heavy metals in bio-char (Praspaliauskas et al., 2018). It can be attributed to a combination of thermal decomposition, chemical transformation, surface adsorption, and volatilization mechanisms, all of which influence the retention and redistribution of heavy metals within the biochar matrix. According to Font (2012), these metals are more resistant to lixiviation than those concentrated in the ash obtained from SS combustion (Fonts et al., 2012). On the other hand, cobalt exhibits a decrease in concentration, indicating that the volatilization process leads to a reduction in the overall content in the residuals (Zhang et al., 2018).

Table 4. Heavy Metals Remaining in Biochar After Pyrolysis Process

	Sample (mg.kg-1)			
Metals	SS	BC-550	BC-650	BC-720
Cobalt	< 0.7	32	23	14
Chromium	73	1,352	148	135
Copper	139	754	822	802
Iron	24,113	31,052	38,903	38,007
Lead	37	82	99	90
Manganese	159	525	470	499
Molybdenum	11	38	20	20
Nickel	22	711	101	106
Silver	<0.2	27	42	27
Tin	48	286	429	415
Titanium	343	4,297	3,942	3,928
Vanadium	86	80	91	87
Zinc	692	2,142	2,028	2,007

BC-550: Bio-char at 550°C, BC-650: Bio-char at 650°C, BC-720: Bio-char at 720°C
Source: Author

Temperature plays a significant role, as higher temperatures during pyrolysis can increase the volatility of certain heavy metals. Consequently, this can result in their partial or complete loss from the biochar matrix. Cobalt, chromium, copper, molybdenum, silver, and vanadium exhibit decreasing concentrations as the temperature increases. This reduction in metal concentrations during pyrolysis is influenced by a combination of factors, including increased volatility, chemical transformation, reduction reactions, and enhanced leaching.

CONCLUSION

In conclusion, the characterization of sewage sludge has provided significant insights into heavy metal analysis, thermal decomposition, and pyrolysis behaviour. The analysis revealed the presence of various heavy metals and alkali and alkali earth metals, notably aluminium, iron, calcium, and phosphorus. It is crucial to maintain pyrolysis temperatures between 450°C and 600°C to ensure the complete conversion of volatile matter into gas phases, providing critical insights into heavy metal migration patterns. Slow pyrolysis predominantly yields biochar as the main product with around 60%, with diminishing yields at higher temperatures. The finding reveals the limited suitability of slow pyrolysis for obtaining bio-oil due to its low yield. Additionally, the pyrolysis process enriches biochar with heavy metals, particularly chromium, copper, nickel, tin, titanium, and zinc, while cobalt exhibits decreased concentration, underscoring the intricate interplay of thermal decomposition, chemical transformation, and volatilization mechanisms in heavy metal retention and redistribution during pyrolysis. By understanding the thermal conversion and behaviour of heavy metals, it informs strategies to reduce environmental impact and promote sustainable development in urban areas.

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AUTHOR CONTRIBUTIONS

Mohd Syazwan Mohd Ghazali: Investigation, formal analysis, visualisation, writing – original draft. Syed Shatir A. Syed-Hassan: Conceptualization, funding acquisition, methodology, supervision, project administration, writing – review & editing.

CONFLICT OF INTEREST

The authors declare that they have no known conflicting financial interests or relationships that might have influenced the work presented in this study.

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