CHEMICAL AND MICROSCOPIC ANALYSIS OF WATER TREATMENT SLUDGE (WTS) FROM BUKIT SEBUKOR WATER TREATMENT PLANT

Abdul Rafeq Saleman[1], Ridhwan Jumaidin[2], Al Amin Mohamed Sultan[2], Umar Al-Amani Azlan[2,*], Fudhail Abdul Munir[1] and Ahmad Fuad Ab Ghani[1]

[1]Fakulti Teknologi dan Kejuruteraan Mekanikal, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya 76100 Durian Tunggal, Melaka, Malaysia.
[2]Fakulti Teknologi dan Kejuruteraan Industri dan Pembuatan, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia

*umar@utem.edu.my

Abstract. In the process of water treatment plant (WTP), a by-product known as water treatment sludge (WTS) are being accumulated in the landfills and thus will become a major concern in the near future. WTS has been examined to look into the potential reuse and rejuvenation. It is known that each WTP produces a different chemical composition which depends on the raw water intake and the treatment process. Thus, in this study, the chemical composition of the WTS will be assessed consistently for a period of 6 months. The samples were then examined using x-ray diffraction (XRD) and scanning electron microscopy with energy dispersive x-ray (SEM-EDX). Peaks observed in the XRD patterns of WTS were associated with the presence of zirconium, oxygen, aluminium, silicon, and sulphur. The results of SEM-EDX revealed that the WTS contained 29.94% to 47.65% of oxygen with aluminium in the range of 20.57% to 23.43%, silicon at 23.88% to 27.79%, and iron ranges from 4.48% up to 19.24%. The same elements were found in all WTS gathered during the six-month period. However, the results are slightly different from our preliminary data. Based on the results, it is concluded that the WTS is a clay with 70-90% purity. Further investigation is needed to find the sources iron and zirconium elements in the WTS.

Keywords: Water treatment sludge (WTS), chemical composition, scanning electron microscopy-energy dispersive x-ray spectroscopy (SEM-EDX)

Article Info

Received 8th March 2023
Accepted 13th July 2023
Published 20th December 2023

ISSN: 1823-7010, eISSN: 2600-7444

Introduction

Clean water is important for human daily activities. Therefore, nowadays clean water is treated at water treatment plants (WTP) and supplied to houses for safe use. WTP requires the process of coagulation, flocculation, sedimentation, filtration, disinfection and pH adjustment [1–3]. Coagulation is a process where electrostatic charges are used to remove solid particles that are suspended in water. Flocculation on the other hand is a process of solid particles clustered to form larger solid and subsequently removed from the water. The process where the particles clustered, sediment at the bottom of the water is sedimentation. Then the water along with the particles clustered is filtered through sands via filtration process. This is then followed by disinfection where the bacteria are eliminated, and finally the raw water undergoes pH adjustment process [3-4]. Throughout the process of WTP, the water treatment sludge (WTS) is produced as a by-product where the particles are clustered as a result of flocculation process and subsequently separated through the filtration process [1-2,5]. These processes have been applied in most of WTP plant in the world. In Malaysia, WTPs significantly produces a large number of WTS which are then accumulated in landfills. The disposal process of WTS is, however bound by a stringent regulation of waste management by the Department of Environment [6]. Therefore, a rejuvenation method towards WTS is a must.

In the pasts, there are researchers that had looked into solving this problem base on the potential of being reuse [1,7–10] Since the process of water treatment plant requires a large amount of alum consisting of Aluminium, there are researchers whom had looked at recovering the aluminium from the WTS [1-3,5]. Among the past studies, researcher had looked into the WTS characterisations [1-2,10], the safe disposal of the WTS [2,4,11]. And finally the potential of reuse and application in engineering applications such as construction materials [7,10,12–14] Thus, in this paper also, the WTS characterization is taken into account as the first step before further investigation on the potential usage of WTS is carried out.

Past researchers have examined the potential reuse of WTS however, since the ratio of each element in the WTS depends very closely with the raw water intake. Therefore, different WTP exhibits different materials and percentage of materials compositions, hence it complicates the process [1,10,15-16] Although the percentage of materials compositions have differences between each WTP, it was identified that WTS usually exhibits aluminium, calcium, silicon, and oxygen [2,10,15,17]. In our preliminary investigation, we examined WTS produces in Loji Air Sebukor (LAS) and determined that the WTS exhibits nearly the same common materials which are carbon, aluminium, silicon, and oxygen. However, the percentage of each element were different from previous studies. Furthermore, some anomalies also have been found where the WTS in the LAS exhibits additional element which are zirconium. Thus, in this study we would like to clarify the materials and the consistency of the WTS contents.

Materials and Methods

In LAS, the raw water intake comes from bunded storage (water retention pond). Raw water in the bunded storage is obtained from the overflow of Sungai Melaka. The water treatment process in LAS involved aeration, coagulation, flocculation, sedimentation, filtration and finally, chlorine dosing. Throughout this process, WTS formed the by-product. The filtration process separates WTS from clean water. In LAS, the WTS go through additional process of a pressing machine to remove the large content of water, that creates the formulation of dry sludge or WTS paste. The WTS paste was then dumped at a dump site before further disposal. Figure 1 show the dump site of the WTS in LAS. In this study the WTS sample was collected monthly for a 6-month period, from December 2021 until May 2022 from the dump site. In this study, the samples were examined using scanning electron microscopy with energy dispersive x-ray spectroscopy (SEM-EDX) (brand: Jeol, model: JSM6010 plus /LV) technique. Preparation of the sample is done by coating the sample with platinum using sputter coated before being analyzed by SEM-EDX. The platinum coating is done to make sure a proper reflection from the SEM-EDX is acquire during the analysis.

Figure 1: Landfill of WTS at Loji Air Sebukor (LAS)

Results and Discussion

In this section, the results from the preliminary data as well as the data collected in the 6 months mentioned are presented. For consistency of the results, all of the data has been collected using the same method and the element analysis was performed using SEM-EDX.

Preliminary Analysis

a) Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy (SEM-EDX)

In this analysis, the WTS examined using SEM-EDX at three different spots, as shown in Figure 2. From the results, it was determined the elements detected were carbon, oxygen, aluminium, zirconium and silicon with different weight percentage of the elements present reflecting the possible composition in WTS. Additionally, it was revealed that the powder samples exhibited significant agglomeration in their morphology, which could be linked to their small and fine powder sizes.

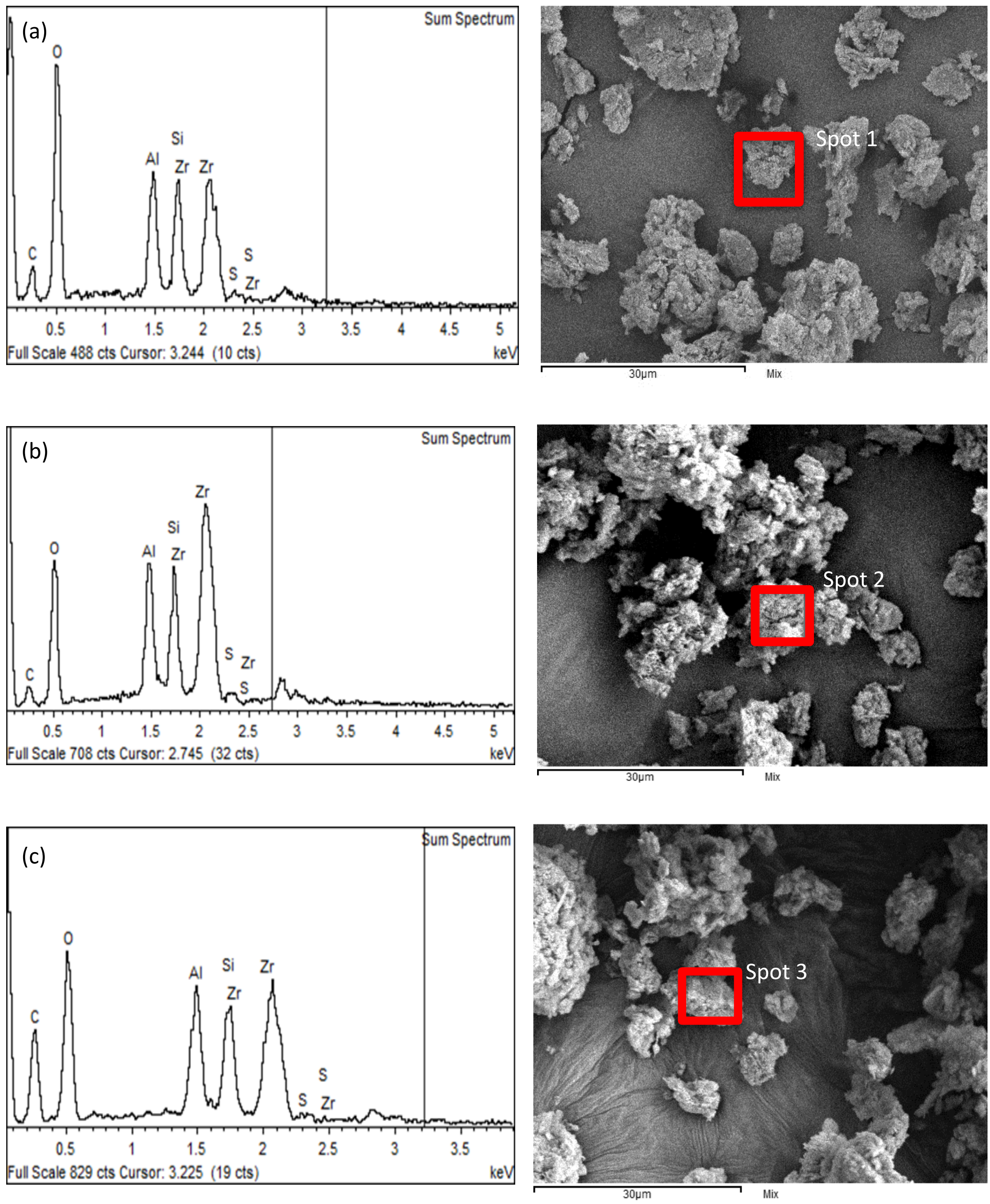

Figure 2: Element analysis using SEM-EDX (a) Spot 1, (b) Spot 2 and (c) Spot 3

Table 1 presents the composition of elements in WTS according to the weight percentage. The weight percentage is calculated as follow; the weight of one component or element is divided by the entire weight of the sample. Regardless of the 3 spots, zirconium exhibited the highest weight percentage in WTS. The values are 30.39% for Spot 1, 46.28% for Spot 2 and 28.78% for Spot 3. The second highest is oxygen with 40.93%, 28.45% and 29.04% for Spot 1, Spot 2 and Spot 3, respectively. It is believed that oxygen was prominently detected in clay composition. The third and fourth highest element contents are carbon and aluminium, respectively. High contents of carbon and aluminium are expected since the WTP involves the use of chemicals with significant aluminium for the sedimentation process. The existence of a high weight percentage of zirconium requires further explanation and analysis, which might contribute to the highest atomic weight.

Table 1: Element and weight percentages of WTS sample

	Weight %		
Element	**Spot 1**	**Spot 2**	**Spot 3**
Carbon	10.92	7.25	26.85
Oxygen	40.93	28.45	29.04
Aluminium	7.54	7.59	6.83
Silicon	8.70	8.41	7.33
Sulphur	1.52	2.02	1.18
Zirconium	30.39	46.28	28.78

b) X-ray Diffraction (XRD)

The preliminary sample was also analysed under the x-ray diffraction (XRD). Figure 3 displays the results of the XRD patterns. The outcomes revealed that several materials are present in WTS, such as zirconium, oxygen, aluminium, silicon, sulphur, etc. Based on these results, the peak for zirconium oxide was at 35.3^{o} and 36.3^{o} of 2θ. The same value of 2θ for zirconium oxide was also found in references [16,19]. The results were compared with previously conducted research. No existence of zirconium in the WTS characterization was found in references [1,4,13]. As known from past assessments, chemical usage (such as alumina and calcium carbide) are heavily utilised in the WTP process. Therefore, Al and Ca are common elements in WTS. However, the existence of Zr can be considered as unique, requiring further clarification from different characterization methods. Comparisons have been made between the current results of XRD and previous outcomes in references [1,4,13]. None of the previous investigations have found Zr in their samples.

Analysis of Samples for 6-month Period

The collection of 6 months' data involves samples collected at a certain date of the month and the samples were examined under the SEM-EDX where the same method was applied in the collection of the preliminary data. However, in the 6 months' data, average data of each element from all three spot were calculated and presented in Table 2. Based on the results, it is found that the WTS samples consistently contained oxygen in the range from 29.94% to 47.65%, aluminium in the range of 20.57% to 23.43%, silicon in the range of 23.88% to 27.79%, and iron ranges from 4.48% up to 19.24%. Although based on the results the sample characterization of the WTS shows consistency in the elements, the sample that we collected for the 6 months' period does not contain any zirconium. Rather a new element found was iron. Based on the results, it is confirmed that at different period of time, the WTS collected will exhibit different quality and quantity of element content. Therefore, in order to make sure the content of the WTS the research need to identify the content for the raw water intake before the water treatment taking place. In terms of data consistency, it is found that the sample elements were consistent for the 6 months' period. In addition, from the results the composition of the elements is approximately similar to clay composition, however since there is existence of zirconium or iron, the purities of the clay are reduced and needed further clarifications and further investigations.

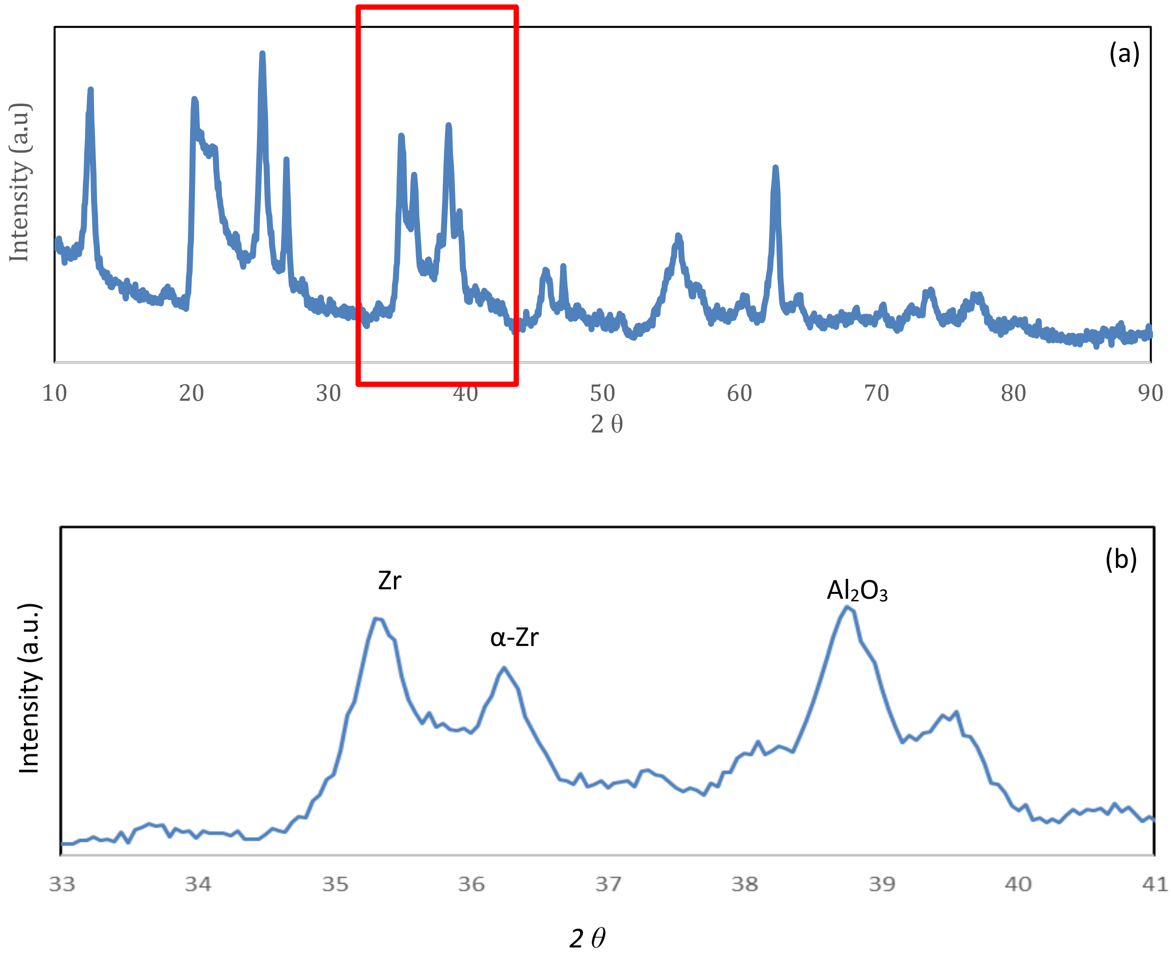

Figure 3: X-Ray Diffraction (XRD) of Water Treatment Sludge (WTS) (a) overall spectrum and (b) enlargement of figure 3(a) for *2θ* ranges from 33 to 41

Table 2: Elements and weight percentages of WTS for 6 months

Month	Dec 2021	Jan 2022	Feb 2022	Mar 2022	Apr 2022	May 2022
Element	**Weight (%)**					
Oxygen	29.94	43.69	47.65	44.17	46.36	40.73
Aluminium	23.76	21.45	20.96	23.43	20.57	24.32
Silicon	27.07	25.78	25.73	27.79	23.88	27.06
Potassium	0.00	0.00	1.19	0.00	0.67	0.00
Iron	19.24	6.89	4.48	4.62	6.29	5.41
Carbon	0.00	0.00	0.00	0.00	0.00	2.49
Copper	0.00	1.26	0.00	0.00	0.35	0.00
Sulphur	0.00	0.94	0.00	0.00	0.00	0.00

Conclusions

In this study, the water treatment sludge (WTS) has been examined using the scanning electron microscopy with energy dispersive x-ray spectroscopy (SEM-EDX). In order to verify the consistency of the WTS elements, the data were collected for 6 periods where the sample have been assessed at specified date for each of the month. It is found that, the WTS contained highest content of oxygen, aluminium, and silicon. The same elements were found in the clay composition thus based on the composition of elements, the WTS is a clay however the percentages of purities for the clay needs further investigation. The impurities are due to the existence of additional of other metals for this case is the zirconium and iron. Although in this study it is found that the elements content is consistent however further investigation is needed as to find the sources of existence of iron and zirconium in the WTS.

Acknowledgements

This research was supported in part by a Kurita Overseas Research Grant (21Pmy087) provided by Kurita Water and Environment Foundation. The author is grateful to Universiti Teknikal Malaysia Melaka and Ministry of Higher Education Malaysia for the financial support provided through research grant FRGS/1/2022/FKM.

Author Contributions

All authors contributed toward data analysis, drafting, and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare.

Compliance with Ethical Standards

The work is compliant with ethical standards.

References

[1] Ooi, T. Y., Yong, E. L., Din, M. F. M., Rezania, S., Aminudin, E., Chelliapan, S., Rahman, A. A. & Park, J. (2018). Optimization of Aluminium Recovery from Water Treatment Sludge using Response Surface Methodology. *Journal of Environment Management.* 228, 13–19.

[2] Ahmad, T., Ahmad, K. & Alam, M. (2016). Characterization of Water Treatment Plant's Sludge and its Safe Disposal Options. *Procedia Environmental Sciences.* 35, 950–955.

[3] Awab H., Paramalingam, P. T. T. & Mohd Yusof, A. R. (2012). Characterization of Alum Sludge for Reuse and Disposal. *Malaysian Journal of Fundamental & Applied Sciences.* 8(4), 251–255.

[4] Ahmad, T., Ahmad, K. & Alam, M. (2016). Sustainable Management of Water Treatment Sludge through 3 'R' Concept. *Journal of Cleaner Production.* 124, 1–13.

[5] Sun, F., Hu, W., Pei, H., Li, X., Xu, X. & C. Ma. (2015). Evaluation on the Dewatering Process of Cyanobacteria-containing $AlCl_3$ and PACl Drinking Water Sludge. *Separation and Purification Technology.* 150, 52–62.

[6] E. Q. A. EQA, "Federal Subsidiary Legislation Environmental Quality Act 1974 [Act 127]," Ministry of Natural Resources, Environment and Climate Change, July 1977, 2–6, 2004.

[7] Babatunde, A. O. & Zhao, Y. Q. (2007). Constructive Approaches Toward Water Treatment Works Sludge Management: An International Review of Beneficial Reuses Treatment Works Sludge Management. *Critical Review in Environmental Science and Technology.* 37, 129-164.

[8] C. Exley. (2009). Darwin, Natural Selection and the Biological Essentiality of Aluminium and Silicon. *Trends Biochemical Sciences.* 34(12), 589-593.

[9] Muisa, N., Nhapi, I., Ruziwa, W. & Manyuchi, M. M. (2020). Utilization of Alum Sludge as Adsorbent for Phosphorus Removal in Municipal Wastewater: A Review. *Journal of Water Process Engineering.* 35, 101187.

[10] Wheeler, R. & Stone, A. (2019). Potential Alternative Reuse Pathways for Water Treatment Residuals: Remaining Barriers and Questions - A Review. *Water Air & Soil Pollution*. 230, 227

[11] Eyuti, A. M. & M. Lawal. (2011). Recovery of Coagulants from Water Works Sludge: A Review. *Advances in Applied Sciences Research.* 2(6), 410-417.

[12] Hai He, Z., Yang, Y., Yuam, Q., Shi, J., Liu, B., Liang, C. & Du, S. (2021). Recycling Hazardous Water Treatment Sludge in Cement-based Construction Materials: Mechanical Properties, Drying Shrinkage, and Nano-scale Characteristics. *Journal of Clean Production* 290, 125832.

[13] De Carvalho Gomes, S., Zhou, J. L., Li, W. & Long, G. (2019). Progress in Manufacture and Properties of Construction Materials Incorporating Water Treatment Sludge: A Review. *Resources, Conservation & Recycle.* 145, 148–159.

[14] Sales, A. & de Souza, F. R. (2009). Concretes and Mortars Recycled with Water Treatment Sludge and Construction and Demolition Rubble. *Construction and Building Materials.* 23(6), 2362–2370.

[15] Rodrigues, L. P., Nilson, J. & Holanda, F. (2015). Recycling of Water Treatment Plant Waste for Production of Soil- Cement Bricks. *Procedia Materials. Science*. 8, 197–202.

[16] Kumari, L., Du, G. H., Li, W. Z., Vennila, R. S., Saxena, S. K. & Wang, D. Z. (2009). Synthesis, Microstructure and Optical Characterization of Zirconium Oxide Nanostructures. *Ceramic International*. 35, 2401–2408.

[17] Sadeghinezhad, E., Mohammad, M., Saidur, R., Mehdi, M., Sara, T. L., Amir Reza, A. &

Metselaar, H. S. C. (2016). A Comprehensive Review on Graphene Nanofluids: Recent Research, Development and Applications. *Energy Conversion Management.* 111, 466–487.

[18] Saleman, A. R., Jumaidin, R., Mohamed, A. A., Azlan, U. A., A. & Zakaria, M. Z. (2021). Characterisation of Water Treatment Sludge (WTS) at a Water Treatment Plant in Melaka. In The Proceeding of the 7th International Conference on Recent Advances in Materials, Minerals & Environment (RAMM2022), Malaysia, 19-20 July 2022.

[19] Zhang, H. X., Fruchart, D., Hlil, E. K., Ortega, L., Li, Z. K., Zhang, J. J., Sun, J. & Zhou, L. (2010). Crystal Structure, Corrosion Kinetics of New Zirconium Alloys and Residual Stress Analysis of Oxide Films. *Journal of Nuclear Materials.* 396(1), 65–70.