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Research paper

The impact of microwave treatment on the chemical properties of sewerage sludge

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Abstract: Due to urbanization, the population in the major cities in Malaysia is approximately 72.8% of its total population. The increase of population density has directly increased the amount of sewerage sludge waste that poses threat to the environment. In line with the green initiatives, alternative method to develop good quality concrete material from sewerage sludge waste can be further explored. Traditionally, sewerage sludge waste is processed using incinerator that require high energy and it is time consuming. In this study, microwave heating which require less energy consumption and less time consuming is used for sewerage sludge preparation. Prior to heating process, sewerage sludge waste is over dried at 105°C for 24 hours. Three types of microwave heating namely medium heating, medium high heating and high heating has been used. The chemical and physical properties microwaved sewerage sludge ash (MSSA) was tested using X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). Based on the result, the recommended temperature for the MSSA production for the concrete is High Mode Temperature. This is due to the result of MSSA for X-Ray Fluorescent test as its shows the highest in the content for pozzolanic element which are SiO_2

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and Fe_2O_3 that produce after the microwave burning process. The mineralogical composition and the crystalline phase of the High temperature MSSA due to X-Ray Diffraction test also shows high content of SiO_2 as the major component as it is good for pozzolanic reaction in concrete. From the Scanning Electron Microscope test, it is observed that particle of High heated MSSA are slightly smaller than other temperature. Also, the densification occurs at High temperature MSSA. Hence, the optimal burning temperature mode for MSSA is High Mode temperature.

Keywords: microwave heating, pozzolonic activity, sewerage sludge

1. Introduction

Sewerage sludge is the by-product of the wastewater treatment plant mainly from flushing of toilet, washing and etc [1–3]. In the past, the sewerage sludge managed through direct dumping to the landfill without proper treatment that caused critical environment issues [4]. Due to higher level of environmental awareness, sustainable sewerage disposal management has been imposed in many developing and developed countries to offset further deterioration to the environment. Thus, much studies are conducted by using different types of treatment and resultant pollution content for applications in different field such as agriculture, manufacturing and construction [5–7]. Combustion, landfill disposal, incineration, pyrolysis, and gasification are the popular treatment used for sewerage sludge [8].

Some findings from the past researchers found the existence of heavy metals in the sewerage sludge [4, 5, 7, 8]. However, the composition of heavy metals in the sewerage sludge are largely dependent on the input and the treatment options of the sludge. However, the initial findings in Malaysian domestic sewerage sludge revealed that there is high concentration of solid and organic matters with significant presence of pathogens, nutrients, organic and inorganic pollutant [9]. Nur-Nazirah et al. [10] further added that the sewerage sludge is acidic and contain high amount of heavy metals.The presence of heavy metals such as Zn, Cu, Ni, Cd, Pb, Hg and Cr is restricting the use of sludge for agricultural purposes [11, 12].

Many studies show that there is existence of pozzolanic materials in the form of siliceous and aluminous materials in sewerage sludge. Trusilewicz et. al [13] further suggested that SiO_2 and Al_2O_3 in SSA are responsible for the pozzolanic activity in the cement-based materials. However, the pozzolanic activity for SSA is relatively low and worth to be used as alternative resources for cement production since it increases the mechanical properties of concrete [14]. Table 1 show the findings on the chemical composition of sewerage sludge ash by the past researchers. From Table 1, it is observed that the main composition of sewerage sludge are SiO_2, Al_2O_3, Fe_2O_3, CaO, P_2O_5, MgO, and SO_3. From the findings, it is observed that, the range of chemical compositions are relatively high. High amount of SiO_2 in sewerage sludge promotes higher pozzolanic reaction in the early hydration process. Besides that, the hydration of CaO improves the structural strength to the concrete.

CaO or limestone takes part in hydration reaction of the cement paste and develop strength in cement at the early stage. The reaction between $CaCO_3$ of limestone and

Table 1. Chemical composition of sewerage sludge ash

Chemical composition	Country	SiO_2	Al_2O_3	Fe_2O_3	CaO	P_2O_5	MgO	SO_3
Nakic, 2018 [15]	Croatia	35.0	7.59	–	34.3	5.81	4.21	4.56
Chen et al., 2018 [16]	Hong Kong	27.91	12.26	18.32	10.47	9.77	3.16	6.13
Baeza-Brotons et al., 2014 [17]	Spain	17.27	9.64	8.52	30.24	14.25	3.22	8.95
Chen et al., 2013 [18]	France	30.1	26.3	5.63	7.35	11.8	4.3	0.13
Chakraborty et al., 2017 [19]	Seoul	47.05	25.28	11.28	4.72	–	2.5	–
Dyer et al., 2011 [20]	United Kingdom	29.4	10.9	13.0	14.3	13.5	1.4	1.3
Rezaee et al., 2019 [21]	Iran	9.3	2.53	1.43	12.06	0.22	0.91	1.07
Mejdi et al., 2020 [22]	France	28.6	17.6	4.4	20.1	19.5	2.3	2.0
Husillos Rodríguez et al., 2013 [23]	Spain	3.72	2.56	7.58	7.62	6.43	0.6	3.9
Oliva et al., 2019 [24]	Chile	12.9	6.6	5.8	11.2	0.8	1.3	3.6
Abdel-Gawwad et al., 2020 [25]	Egypt	34.15	15.36	9.58	10.36	7.65	2.97	9.73
Krejcirikova et al., 2019 [26]	Denmark	15.83	4.4	16.3	23.37	18.33	1.82	2.0
Istuque et al., 2016 [27]	Brazil	38.28	20.72	11.27	5.51	–	1.91	4.18

aluminate phase of cement (C_3A) takes place. Thus, it is important to have balance SiO_2 and CaO for more complete reaction.

In general, partial replacement of SSA reduces the compressive strength at the early stage since the pozzolanic activity only occurs after the hydration of cement with water. Thus, the early strength of mortar with SSA replacement should be thoroughly studied as it is important during the demoulding process. Removal of specimen with insufficient concrete strength may cause the structure to fail collapse. Besides that, excessive amount of SSA may reduce the compressive strength of the concrete [26, 29].

On top of the chemical content of SSA, the fineness of the sewerage for cement replacement is also important for better pozzolanic reaction. Ground SSA has more surface area compared to raw SSA [29]. Ground SSA makes ash particles smoother and less porous, thus improving the workability of the concrete mixture. Besides that, Ing et al. [30] found that 10% of SSA replacement produces optimum compressive strength compared to control. Similar finding is confirmed by other researchers [26, 31]. In this research, the effect of microwave treatment of sewerage sludge on the chemical and mechanical properties of sewerage sludge concrete.

2. Preparation of MSSA

One of the major problems that causes to environmental issue is the present of heavy metals in sewerage sludge. Traditional method of incineration requires high energy consumption and is also time consuming. Microwave heating which supplies volumetric heating process expedite the heating process while require less energy consumption [32–34]. Besides that, microwave heating provides a more homogeneous heating resulting in more homogenous SSA. The sewerage sludge was taken from the treatment plant owned by Indah Water Konsortium (IWK), Kuantan branch. The treatment plant served private and business region and categorized as domestic sludge. At the treatment plant, the sewerage sludge was sun dried at the sludge bed. As shown in Fig. 1, only the top part of the sludge bed was collected by using scoop.

Fig. 1. Collecting sewerage sludge at sewerage treatment plant

Fig. 2 shows the raw sewerage sludge collected from sewerage treatment plant. To remove the moisture content, the raw sewerage sludge was oven dried at the laboratory for 24

Fig. 2. Raw sewerage sludge

hours at 105°C [35]. Next, the oven dried sewerage then burned by using microwave different temperature which is medium (231°C), medium high (297.5°C) and high temperature (350°C) for 30 minutes to find the optimum temperature. The model of the microwave oven is ELBA EMO-A2072 (SV) with the operation frequency of 2450 MHz and rated power outlet of 700W. The wavelength of microwave is 12.2 cm. The dried sewerage sludge was placed on the clay pot and put into the microwave oven. The microwaved sewerage sludge ash (MSSA) shown in Fig. 3 was ground into powder form. Only particle size passing through 75 µm was used since it is relatively close to the cement particle size of 50 µm [12].

Fig. 3. Microwave Sewerage Sludge Ash (MSSA)

3. Result and discussion

3.1. X-Ray Diffraction (XRD)

In this research, XRD was performed by using an XRD machine with 2θ scanning ranging between 5° and 80°. MSSA with the particle size passing through 75 µm sieve (Fig. 4) were tested [14]. The analysis scan run at 0.05° steps, with 1 second counting time. The optimum microwave burning methods were identified from the mineralogical composition.

X-Ray Diffraction test used to identify the chemical composition of the different heated temperature of MSSA which was medium, medium high and high temperature mode. Fig. 5 presents the XRD result for medium temperature of MSSA. The graph show that the MSSA consist of mainly SiO_2 and P_2O_5. The peak at 27° with intensity of 92 cps represented SiO_2 followed by P_2O_5 at 21° with 33.21cps. From the finding, it is observed that SiO_2 is the main mineral in SSA. Similar findings are confirmed by the past researchers [15,16,18,27].

Fig. 4. Ground MSSA

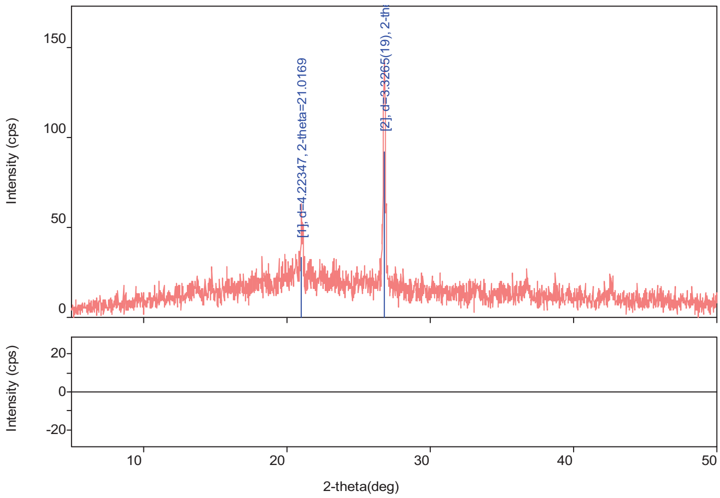

Fig. 5. XRD of Medium Temperature MSSA

Fig. 6 shows the result of XRD from MSSA at medium high temperature. From the graph, MSSA heated with medium high temperature consist mainly SiO_2. The peaks of XRD patterns showed the intensity of the SiO_2 is 1334 cps at 27.16° and 374cps at 21.32°. Fig. 7 indicates that the XRD pattern of MSSA microwaved heated with high temperature. Based on the pattern, only SiO_2 exists in the specimens. High temperature MSSA consist high amount of SiO_2 with intensity of 627cps at 31.42°, 2452cps at 27° and 1211cps at 68.64°.

From the XRF test of MSSA in different microwave temperature, it was observed that main phase for all of the tested MSSA is SiO_2. The results indicated that, higher microwave

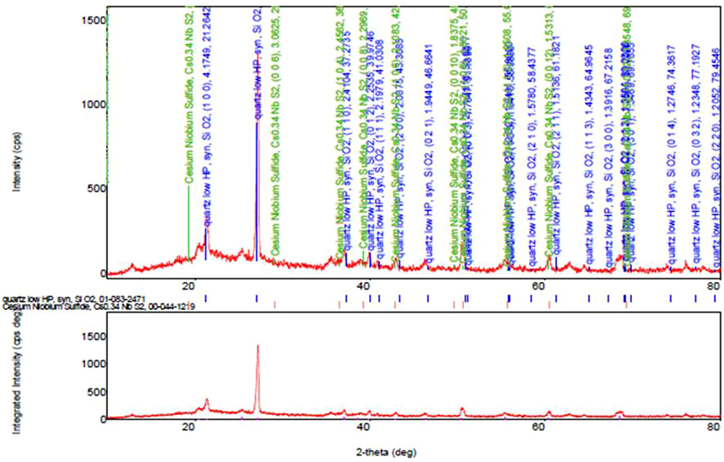

Fig. 6. XRD graph for Medium High temperature of MSSA

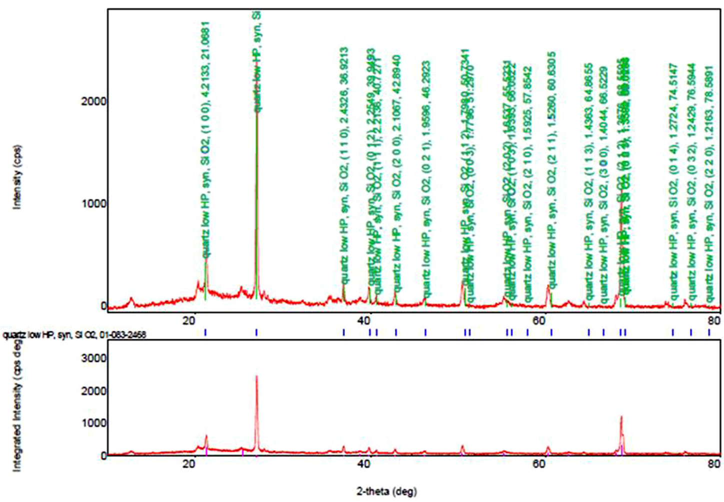

Fig. 7. XRD result for High Temperature MSSA

burning temperature produces high percentage of SiO_2 by observing the results of MSSA at different burning temperatures.

3.2. X-Ray Fluorescent (XRF)

XRF analysis is to determine the oxide component in MSSA. The XRF use x-ray to detect the chemical composition in solid, liquid as well as powder form samples. This test is considered as non-destructive test that allowed to be used in wide range of the chemical elements from sodium till uranium which able to provide the concentration up to 100%.

Ground MSSA with different burning temperature were sent to be tested by Bruker S8 XRF spectrometer to identify the chemical composition of MSSA.

The main chemical component of each different samples of MSSA that burned in three different temperatures was summarized in Table 2. The main oxide group found in all of three samples were SiO_2, Fe_2O_3, Al_2O_3, CaO and P_2O_5 followed by heavy metal such as ZnO, TiO_2, BaO, CuO and MnO.

Table 2. X-Ray fluorescent result for different temperature of MSSA

Oxide Group	Medium temperature (%)	Medium high temperature (%)	High temperature (%)
SiO_2	9.41	14.79	14.94
Fe_2O_3	10.31	9.25	11.14
SO_3	7.34	6.05	6.86
Al_2O_3	5.14	5.66	5.36
P_2O_5	3.17	2.93	3.57
CaO	2.31	2.00	2.57
ZnO	1.16	1.02	0.83
K_2O	0.84	0.84	0.75
TiO_2	0.76	0.73	0.62
MgO	0.34	0.27	0.30
Cl	0.19	0.19	0.15
BaO	0.11	0.10	0.11
MnO	0.34	0.07	0.07
CuO	0.09	0.09	0.07

MSSA treated with high temperature shows the highest content of SiO_2 of 14.94%, followed by medium high temperature of 14.79%. However, the MSSA treated with medium temperature shows very low content of SiO_2 of only 9.41%. It can be suggested that the burning process triggers the formation of SiO_2 in MSSA that enhanced the pozzolanic activity in the cementitious material [25]. Besides the SiO_2, the content of Fe_2O_3, Al_2O_3 and CaO are relatively high since alumina salt, ferric salt and lime are used as part of the sewerage water treatment.

Even though the SiO_2 content in medium high and high temperature MSSA are relatively similar, the heavy metal content such as P_2O_5, TiO_2 and ZnO is higher in MSSA that burned in medium high temperature. The content of heavy metal in MSSA with high temperature is slightly lower as compared to medium and medium high temperature MSSA. The present of high amount of heavy metal may cause adverse effect to the mechanical strength of concrete [12]. From the XRF result, it can be concluded that the MSSA with high temperature provided the optimum treatment for sewerage sludge waste for the use of MSSA as cement replacement in concrete.

3.3. Scanning electronic microscopy

In this research, Hitachi TM3030Plus table top microscope was used to identify the microstructural of MSSA burned in three different microwave modes which were medium, medium high and high temperature mode. The Scanning Electron Microscope (SEM) of MSSA was carried out to analyse the surface morphology, crystallography and surface composition of the samples with different burning temperatures. Fig. 8 shows the micrograph of Medium-MSSA at 200 × (a) and 10000 × (b). From the micrograph in Fig. 8a, it was obtained that the Medium-MSSA has mostly angular shape particles. The shapes of the particles are irregular due to the potential organic matter content inside the medium heated sewerage sludge ash. The image in Fig. 8b shows that the sewerage sludge ash also contained different porous structure.

a) b)

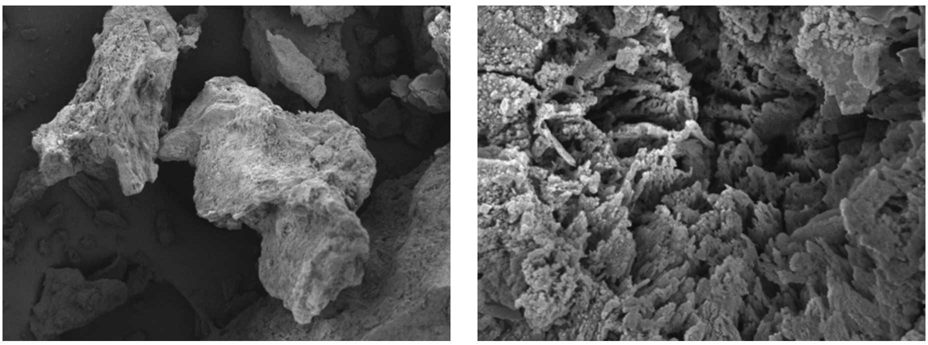

Fig. 8. SEM image of Medium heated MSSA at 200× (a) and 10000× (b)

Fig. 9 shows the Medium High-MSSA at 200× and 10000× magnification. There is formation of discrete and spherical particles as shown in Fig. 9b in random arrangements. The increases burning temperature starting to break down the sewerage sludge particles into smaller pieces compared to medium temperature heating of MSSA as shown in Fig. 9a. Thus, the structure of MSSA in Fig. 9b shown discrete and less porous compared to medium heated MSSA.

The SEM results of MSSA at high temperature are shown in Fig. 10. The particles sizes of the high temperature burning of MSSA are irregular as shown by Fig. 10a. However, pores of variable sizes and shapes are observed in Figure 9b that relatively consists of small pores in irregular shapes. At high temperature, the particles are compacted and form a higher density [37]. The structure of high heated MSSA as shown in Fig. 10b shows higher density compared to those heat in medium and medium high temperature. Furthermore, the result of MSSA grinding process also help in reduced the visible pores.

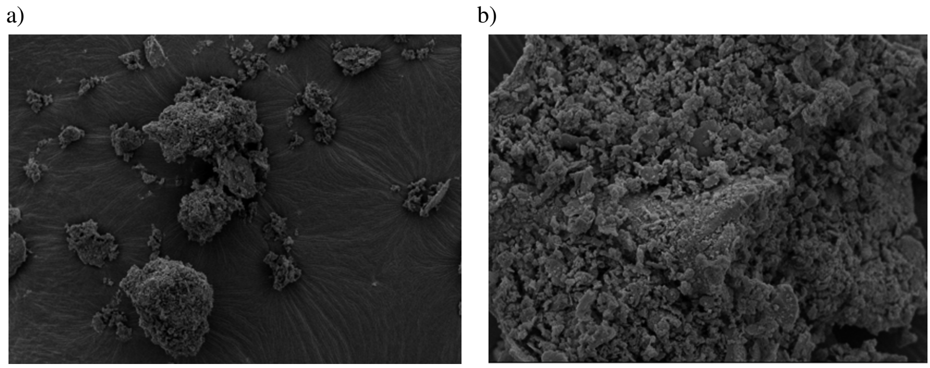

Fig. 9. SEM image of Medium-High heated MSSA at 200× (a) and 10000× (b)

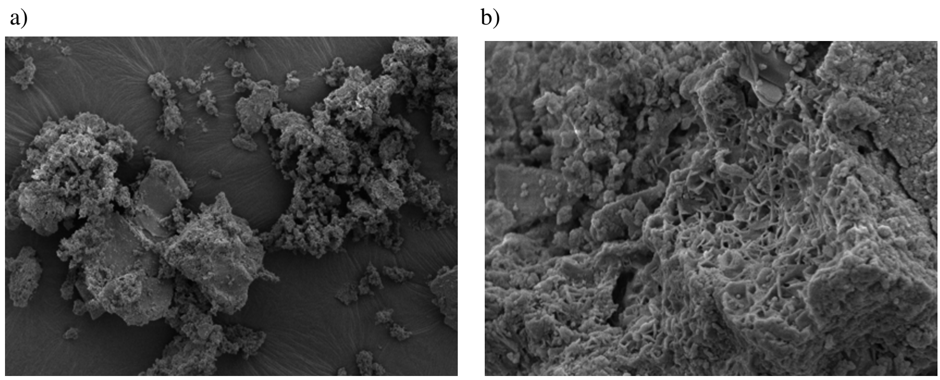

Fig. 10. SEM of High temperature heated MSSA at 200× (a) and 10000× (b)

4. Conclusion

Based on the result, the recommended temperature for the MSSA production for the concrete is High Mode Temperature. This is due to the result of MSSA for X-Ray Fluorescent test as its shows the highest in the content for pozzolanic element which are SiO_2 and Fe_2O_3 that produce after the microwave burning process. The mineralogical composition and the crystalline phase of the High temperature MSSA due to X-Ray Diffraction test also shows high content of SiO_2 as the major component as it is good for pozzolanic reaction in concrete. From the Scanning Electron Microscope test, it can be observed that particle of High heated MSSA are slightly smaller than other temperature. Also, the densification occurs at High temperature MSSA. Hence, the optimal burning temperature mode for Microwave Sewerage Sludge Ash is High Mode temperature.

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