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# Solubilizing Waste Activated Sludge via Thermal Treatment to Enhance the Biodegradability

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Abstract. The current work intends to unveil the performance of thermal treatment in improving the biodegradability of waste activated sludge (WAS). The thermal treatments of WAS were conducted at different temperatures ( $30^{\circ}$ C,  $60^{\circ}$ C,  $75^{\circ}$ C,  $90^{\circ}$ C) as well as treatment durations (0, 1, 2, 4, 8, 16 hours), and its effects on the sludge biodegradability were measured in terms of soluble chemical oxygen demand (SCOD). The ANOVA analysis showed that both treatment temperature (P < 0.001) and duration (P < 0.001) had significantly enhanced the biodegradability of WAS. Accordingly, the results showed that the SCOD increased with increasing treatment temperature, with 90°C thermal treatment at 16 hours producing the highest SCOD of 163.39g/L. Although SCOD increased with longer treatment duration, the highest SCOD increased within the first 4 hours of treatment before slowing down gradually. Besides, the interaction between treatment temperature and duration was also significant (P < 0.001). This was because the effect of treatment duration becoming less significant when the treatment temperature was raised. Finally, the energy-effective thermal treatment condition was found to lie between 75°C and 90°C within 2 to 4 hours in achieving the highest concentration of SCOD per consumed energy of 0.27 - 0.32 g/L per kJ. Hereafter, the impending study is to administer the treated WAS for growing black soldier fly larvae, targeting to produce valuable larval biomass feedstock for biodiesel industry.

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#### INTRODUCTION

Owing to the rapid development of urban city and growth of human population, the volume of wastewater being produced has increased steadily. Similarly, waste activated sludge (WAS), which is a by-product generated from conventional wastewater treatment process has also been skyrocketing to a new high globally [1]. The WAS is hazardous in nature. It is richly inhabited by pathogens such as *E. coli* and *Salmonella* spp. Furthermore, it contains a high amount of organic matters and heavy metals. If left untreated, these organic matters and heavy metals could be leached into the ground, causing land pollution and even groundwater pollution [2]. Nonetheless, the high organic matter content also can be translated to a possibility of carbon and nitrogen recoveries that will promote a circular economy. In the past, landfilling and ocean dumping are used to be the most common ways to deal with WAS. These methods are no longer welcomed now. This is attributed to the rise in environmental awareness as well as stricter regulations introduced. European Commission, for example, has announced that starting from 2005, the organic matters in landfilling waste must be lower than 5% to prevent any possibility of organic matters leaching into the ground [3]. As a consequence, numerous treatment technologies such as incineration and anaerobic digestion have been adopted to treat WAS.

Nonetheless, most of these treatment plants are only concentrated in developed countries such as Netherlands, Germany, United States, and China due to the high capital costs involved. Developing countries such as Malaysia is still using the conventional landfilling method. Hence, there is a dire need for an economical sludge treatment method that could be implemented even by developing countries. Recently, BSFL treatment has been adopted widely as an economical and feasible approach to treat and valorize organic wastes [4]. The innate nature of BSFL being a heavy eater allows them to consume organic wastes such as restaurant waste and animal manure rapidly, and then convert the nutrition within the organic wastes for their own development across the six instar stages. Subsequently, the mature BSFL can be harvested to be used as an insect meal, or even process furthered into biodiesel [5,6]. The WAS, which is high in organic matters and has a similar carbon/nitrogen ratio as compared with food wastes [7], could be possibly valorized by BSFL. However, the WAS is hardly biodegradable in nature due to the presence of extracellular polymeric substances (EPS) [8]. The EPS is a mesh-like structure that encloses the protein and carbohydrate tightly, disallowing these nutrients to be accessed by any extracellular enzyme. Consequently, the raw WAS is unsuitable to be used as the BSFL feeding substrate, unless the EPS is broken down to release the trapped carbohydrate and protein. In this work, the performance of thermal treatments in improving WAS biodegradability was studied at different treatment temperatures and durations. The WAS biodegradability was measured in terms of SCOD concentration, which reflected the amount of organic matters solubilized, e.g., released protein and carbohydrate from WAS.

# **MATERIALS AND METHODS**

#### **Procurement of WAS**

The raw WAS was collected from Jelutong Wastewater Treatment Plant in Penang, Malaysia. The sludge was subsequently frost in a chiller at -10°C until further usage was required. Prior to using, the frozen WAS was defrosted overnight for 12 hours. The moisture content of WAS was  $75.25 \pm 3.26$  wt%.

# **Thermal Treatment of WAS**

Next, the samples for thermal treatment were prepared by weighing 30 g wet weight of WAS, followed by inserting them into different glass bottles. Accordingly, a total of 63 bottles was prepared to fulfill the treatment conditions of different temperatures (30°C, 60°C, 75°C, 90°C) as well as treatment durations (1, 2, 4, 8, 16 hours). A set of control was also performed at 30°C at 0 hour. Each treatment condition was performed in triplicate. Thermal treatment was commenced as soon as the glass bottles were placed into water bath at a pre-determined temperature. After the targeted treatment duration had reached, respective bottles were removed and allowed to cool down at ambient condition. Subsequently, 10 g wet weight of WAS was separated to be used for further analysis in this work, while the remaining of 20 g was kept for BSFL rearing later.

#### **SCOD** Analysis

Treated WAS was then analysed for its SCOD concentration according to the Standard Methods APHA 5520C [9]. First, the solid WAS was diluted with distilled water at a ratio of 1:80 (0.5 g wet weight WAS : 40 g distilled water). Next, the sample was centrifuged at 6000 rpm for 15 minutes to allow the separation of SCOD into water layer to occur. After centrifugation, the supernatant was poured through a 0.45  $\mu$ m Whatmann filter paper to ensure only solubilized matters were collected for SCOD analysis from the filtrate.

#### **Statistical Analysis**

Statistical analysis of data was performed using software SPSS Version 24.0. The data was validated by two-way ANOVA with replication. The confidence interval was also set at 95% (P < 0.05).

# **Energy Efficiency Analysis**

Energy efficiency of thermal treatment at specific temperature and duration was calculated based on Equation 4. The total energy required for the thermal treatment of WAS at a specific temperature and duration was a summation of the initial heat required to heat up the WAS to a specific temperature and subsequently, the energy needed to compensate for the heat loss from heater for a specific duration, as shown in Equation 1. The heat loss from water bath heater to surrounding was considered as a radiation process, where an object emitted heat to a vast laboratory space as shown in Equation 3, where  $\sigma$  refers to the Stefan-Boltzmann constant, e is the emissivity of heater, A is the surface area of heater and t is the treatment duration.

$$Q_{total}(kJ) = Q_{WAS}(kJ) + Q_{loss}(kJ)$$
(1)

$$Q_{WAS}(kJ) = mC_p \Delta T \tag{2}$$

$$Q_{loss}(kJ) = \sigma e A(T_{heater}^4 - T_{surrounding}^4) t$$
(3)

Energy efficiency 
$$\left(\frac{g/L\ SCOD}{kJ}\right) = \frac{\Delta SCOD_{temp}}{Q_{total}}$$
 (4)

#### **RESULTS AND DISCUSSION**

## **Impact of Treatment Temperatures and Durations on SCOD**

The ANOVA analysis showed that treatment temperature (P < 0.001) and duration (P < 0.001) both had a significant impact in improving the biodegradability of WAS, as reflected in the increase of SCOD. This finding was aligned with previous works [10–12], which also reported that SCOD increased with increasing treatment temperature and duration. This was because during thermal treatment, the heat energy was provided to the molecules in WAS. This allowed them to vibrate more vigorously and break the chemical bonds. Consequently, the EPS structure broke down, releasing the trapped organic matters into the aqueous phase in which resulting in higher SCOD [13]. From Fig. 1, all the samples had shown an increase in SCOD concentrations after the thermal treatment was conducted. For both thermal treatments at 75°C and 90°C, the SCOD increased tremendously in the first 4 hours. Indeed, the most drastic increments happened in the first hour of treatments. Eventually, the increments slowed down for both temperatures. On the other hand, 60°C treatment showed a modest and stable increment throughout the 16 hours, although the slope for first 4 hours was steeper. For control set at 30°C, the WAS biodegradability altered very slightly as opposed to the other thermal treatment sets. Overall, the increasing tread was observed, with the readings fluctuating between the range of 37.17 to 62.95 g/L. This indicated that the solubilization of organic matters could be an innate

process that happened naturally even without thermal treatment, but at a very slow pace. Future work could extend the treatment duration at 30°C sample to measure its solubilization effect.



FIGURE 1. SCOD from WAS after the thermal treatments at different temperatures and durations.

Based on the two-way ANOVA analysis, the interaction effect between two independent variables (temperature and duration) was also significant, F(15,71) = 34.1, P < 0.001. This indicated that the effect of treatment duration in improving SCOD was significantly different for various temperatures. Indeed, referring to Table 1, the 60°C treatment had recorded a 30% SCOD increment when the treatment duration was prolonged from 8 to 16 hours. However, the same trend was not found for 90°C treatment. When the duration was prolonged from 8 to 16 hours, the SCOD did not increase, but registering a slight drop of 0.49 g/L instead. It also worth to highlight that the effect of treatment temperature had a higher impact on SCOD than treatment duration. For instance, 90°C treatment for just 4 hours had yielded a higher SCOD of 147.62 g/L as compared with 145.81 g/L for the treatment at 75°C for 16 hours. This statement was validated by the smaller P value of treatment temperature ( $P = 6.8 \times 10^{-40}$ ) as compared with duration ( $P = 3.8 \times 10^{-36}$ ). Concisely, the effect of duration was less significant when treatment temperature rose.

Duration (h)	SCOD (g/L)					
	30°C	60°C	75°C	90°C		
0	$37.17\pm 0.96$	$37.17 \pm 0.96$	$37.17 \pm 0.96$	$37.17\pm 0.96$		
1	$35.33\pm0.79$	$52.47 \pm 1.02$	$96.34\pm0.07$	$118.19\pm0.58$		
2	$45.18\pm1.19$	$66.07 \pm 1.58$	$112.29\pm1.64$	$121.43\pm1.14$		
4	$49.27\pm0.88$	$80.56\pm0.55$	$125.07\pm2.59$	$147.62\pm2.74$		
8	$45.19\pm0.27$	$88.65 \pm 1.57$	$132.69\pm2.97$	$163.88\pm1.14$		
16	$62.95\pm3.11$	$115.32\pm3.03$	$145.81\pm2.14$	$163.39\pm10.96$		

TABLE 1. Average SCOD values from WAS and their respective standard deviation after the thermal treatments.

# **Energy Efficiency Analysis**

From previous section, it is highlighted that the similar SCOD could be achieved at higher temperature, but shorter duration. Therefore, an energy efficiency analysis was conducted to determine the energy-efficient cluster that gave

the highest WAS solubilization per input of energy. Control set of 30°C was not included in the analysis as it did not undergo thermal treatment; and hence, there was no change in temperature. Treatment duration of 0 hour was also omitted as it was merely used as a reference for the calculation of increment in SCOD. The energy efficiency was calculated on the basis of 1 kg of WAS. The heat capacity of WAS was estimated to be 4.18 kJ/kg [14] and the initial temperature of surrounding was the same as the control set at 30°C. The Q<sub>WAS</sub> in which depicted the energy required to heat up WAS to a specific temperature was constant for all duration at a specific temperature. Next, the Q<sub>loss</sub> was calculated as a product of power loss by radiation heat transfer for a certain period of time. The T<sub>surrounding</sub> was again set at 30°C. The Stefan-Boltzmann constant,  $\sigma$ , was given as 5.67x10<sup>-8</sup> J/s·m<sup>2</sup>·K<sup>4</sup>. The emissivity of heater, *e*, was considered to be 0.1, which was the emissivity of a 316L stainless-steel [15]. The A was a surface area of heater and assigned to be 0.18 m<sup>2</sup>, since the entire water bath heater was made up of insulating materials, except for the top stainless-steel cover in which had a dimension of 0.3 m x 0.6 m. Lastly, *t* was referred to the treatment duration.

The energy efficiency was calculated based on Equation 4 in which dividing the increment of SCOD by the total energy input to the system. A high energy efficient thermal treatment will produce a high amount of SCOD per unit input of energy. In order to identify the region of thermal treatment that is energy efficient, a heat map is constructed as in Table 2. The high energy efficient treatment condition is colored as dark green, while the least efficient treatment condition is colored as bright red. From the heat map, it was clearly shown that the thermal treatment at  $60^{\circ}$ C was inefficient. In comparison, thermal treatments at  $75^{\circ}$ C and  $90^{\circ}$ C were more efficient, especially if the treatment duration was limited to lesser than 8 hours. From the values of  $Q_{loss}$  in Table 3, once the thermal treatment approached 8 hours, the energy loss to surrounding was as high as the energy required to heat up the WAS. This made the thermal treatment of 8 hours and above to be ineffective because of more energy input was loss to the surrounding rather than being used to heat up the WAS. Furthermore, from Figure 1, it was also shown that the SCOD increased most rapidly during the first 4 hours. Therefore, limiting the treatment durations from 2 to 4 hours at  $75^{\circ}$ C and  $90^{\circ}$ C are the optimum regions as observed from Table 2.

Duration	$\Delta$ SCOD (g/L)			Energy Efficiency (g/L SCOD per kJ)			
(h)	60°C	75°C	90°C	60°C	75°C	90°C	
1	15.3	59.2	81.0	0.11	0.28	0.29	
2	28.9	75.1	84.3	0.19	0.32	0.27	
4	43.4	87.9	110.4	0.24	0.31	0.29	
8	51.5	95.5	126.7	0.22	0.26	0.25	
16	78.2	108.6	126.2	0.22	0.20	0.16	

TABLE 2. Change in SCOD and heat map of energy efficiencies.

TABLE 3. Different energies required to maintain WAS at the specific treatment temperatures.

	Q <sub>WAS</sub> (kJ)			Q <sub>loss</sub> (kJ)		Q <sub>total</sub> (kJ)			
Duration (h)	60°C	75°C	90°C	60°C	75°C	90°C	60°C	75°C	90°C
1	125.4	188.1	250.8	14.2	22.9	32.8	139.6	211.0	283.6
2	125.4	188.1	250.8	28.4	45.8	65.7	153.8	233.9	316.5
4	125.4	188.1	250.8	56.8	91.7	131.3	182.2	279.8	382.1
8	125.4	188.1	250.8	113.7	183.3	262.6	239.1	371.4	513.4
16	125.4	188.1	250.8	227.4	366.7	525.2	352.8	554.8	776.0

#### CONCLUSION

This work had proven that the thermal treatment was capable of improving the biodegradability of WAS. Treatment temperature (P < 0.001) and treatment duration (P < 0.001) both played a significant role in solubilizing the WAS. Overall, the solubilization of WAS improved with higher treatment temperature and longer treatment duration. The highest SCOD released of 163.39g/L was observed after 16 hours of treatment at 90°C. The interaction between these two independent variables was also significant (P < 0.001). The effect of treatment duration was found becoming less impactful when treatment temperature was raised. Finally, the energy efficiency analysis showed that the optimum thermal treatment condition was bounded within 75°C to 90°C from 2 to 4 hours, producing 0.27 - 0.32 g/L of SCOD per kJ of energy input.

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