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Effect of low microwave temperature on the physical and chemical properties of waste activated sludge

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ABSTRACT

In this study, the effect of microwave (MW) irradiation at a target temperature of 80 °C with a heating rate of two °C/min and a holding time of five minutes on the physicochemical properties of activated sludge was studied. The results showed that subjecting biosludge to MW pretreatment resulted in a reduction in particle size to 14.5, 44.6, and 72.8 for d10, d50, and d90, respectively and an increase in the specific surface area of the biosludge to 235.9 m^2/kg. Pretreatment resulted in an increase in soluble oxygen demand, sugar, and protein concentrations to 2,380.0 mg/L, 66.5 mg/L, and 1.81 g/L, respectively. A slight increase in the release of inorganic compounds, especially NH_4-N and PO_4-P, was observed in the disintegrated biosludge. While there was an increase in release of K and Zn, a decrease in concentrations of Na, Ca, Mg, and Al in the liquid was observed after MW irradiation. It was determined that the dewatering property deteriorated after biosludge disintegration, as it increased from raw sludge to disintegrated sludge by 21-fold.

Keywords: microwave irradiation, biosludge, disintegration, nutrients

INTRODUCTION

The waste activated sludge (WAS) is a byproduct of wastewater treatment (WWT) processes. One of the environmental key issues is the sustainable removal or reuse of massive amounts of WAS created due to its composition and high volume with 95.00%-98.00% water content (Kavitha et al., 2018; Tyagi et al., 2013).

WAS is already an environmental concern due to several factors such as:

(1) **High organic content:** The biosludge contains high number of organic matters, which can cause environmental problem like unpleasant odor if not treated properly before dispose,

(2) **Nutrient content (mainly nitrogen and phosphorus):** WAS contains high levels of nitrogen and phosphorus, which may eutrophication in water bodies with depleting the oxygen levels,

(3) **Heavy metals, poorly biodegradable organics, and harmful organisms:** They may all be found (Alhraishawi & Aslan, 2022; Gil et al., 2019).

Biosludge makes up roughly 2.00% of the treated wastewater volume, but the cost of treating it can account for as much as 50.00% of overall operating expenses. This implies that to

(1) correctly execute use and disposal activities,

(2) appropriately satisfy legal requirements, and

(3) foster stakeholder and public confidence, particular procedures/methods need to be devised for the end destination.

The inability to produce acceptable, even modest, levels of inorganic nutrients, particularly phosphorous, is a disadvantage of WWT since it prevents the need for intensive biological or aerobic chemical processes (Keating et al., 2016). Biosludge reduction in WWT processes can be accomplished through three main strategies:

(1) enhanced pretreatment (load reduction),

(2) decreased production, and

(3) biosludge disintegration processes, which increase the percentage of the sludge that is biodegradable and can be more readily mineralized and absorbed by biomass (Ødegaard, 2004).

Biosludge undergoes thickening, stabilization, dewatering, and ultimate disposal in the biosludge treatment line. The most popular technique for stabilizing biosludge is an anaerobic digestion (AD), which lowers the bulk of biosludge

(Wang et al., 2017). AD is an effective process for treating waste and generating CH_4; nevertheless, a slow hydrolysis of particulate organics restricts the possibility of biodegradation (Kavitha et al., 2018).

To liberate nutrients and enhance biodegradability, the biosludge must be pretreated to break down the floc and bacterial cells (Carrère et al., 2010). In fact, biosludge pretreatment is a critical step in treatment processes, especially to enhance biodegradability and release of nutrients (Carrère et al., 2010).

Several methods for biosludge disintegration, including hydrothermal, mechanical, and chemical treatments are applied (Gil et al., 2019). Microwave (MW) pretreatment is one of the thermal methods utilizing micro irradiation at 2.45 GHz for treating biosludge. The effectiveness of MW irradiation comes from its capacity to induce thermal effects and polarize large molecules. At the molecular level, MW irradiation aligns these molecules with the poles of electromagnetic field, potentially leading to the disruption of hydrogen bonds. This dual mechanism of action–thermal effects and molecular polarization–contributes to the disintegration and hydrolysis of biosludge particles (Eskicioglu et al., 2007). Because of enhanced biosludge disintegration and hydrolysis, MW irradiation accelerates the rate and extent of AD (Park et al., 2004; Saha et al., 2011). MW irradiation effectively disrupts the slowly hydrolyze compounds, also extracellular polymeric substances (EPS) of organisms and the network of divalent cations within biosolids (Eskicioglu et al., 2007). There are several factors such as power, target temperature, irradiation time that affect the effectiveness of MW irradiation on the breakdown of WAS and releasing components such as soluble oxygen demand (sCOD), proteins, and sugars (Ya-Wei et al., 2015). For example, when the biosludge was subjected to a long irradiation time, i.e., a short heat ramping rate of one-three °C/min, the release of sCOD, protein, and sugars increases compared to the rate of 11 and six °C/min (Koupaie et al., 2015; Toreci et al., 2010). It was reported that a possibility of caramelization and Maillard reactions occurred when the sludge was irradiated at a high temperature of more than 80 °C (Eskicioglu et al., 2007).

In this study, WAS pretreatment with MW irradiation was investigated at a target temperature lower than the boiling point. By the disintegration of WAS at a target temperature of 80 °C, it will be achieved less energy consumption than at higher temperatures, no caramelization and Maillard reaction, and more CH_4 can be produced due to the higher number of organic compounds that can be utilized by the organisms in AD process. Therefore, this study aimed to investigate the effect of low target temperature and prolonged exposure to irradiation on the release of organic and inorganics from the biosludge, as well as the effect on physical properties of biosludge such as dewaterability and particle size distribution.

MATERIALS & METHODS

Waste Activated Sludge Sampling

WAS samples were collected from the return line from the sedimentation tank to suspended biological reactor at Municipal Biological Wastewater Treatment Plant in Sivas, Turkey. After drawing WAS from WWT plant, to avoid microbial activities, which caused the change of characteristic of samples were immediately transferred to Environmental Engineering Laboratory of Faculty of Engineering at Sivas Cumhuriyet University.

Microwave Disintegration

WAS was disintegrated using a MW (Model Milestone ETHOS EASY), which has a magnetron with a total power of 1,800 W and a pressure of 35 bar. MW contained 44 PTFE containers, each with a capacity of 100 mL, and could be programmed to reach the target temperature at different heat ramping rates by adjusting the run time via the digital display. The energy consumption during the WBS disintegration in MW was calculated according to Eq. (1) (Passos et al., 2013).

MW was operated at a low ramping heat rate of 2 °C/min to reach a temperature of 80 °C and a holding time of 5 min was applied at the target temperature. MW was operated the power of 900 W and the ramp rate and retention time at the target temperature was determined as the optimal condition of MW operation for WAS disintegration in our previous studies considering the disintegration degree and sCOD concentration (Aslan, 2023). MW disintegrated samples were cooled to the room temperature of about 20±5 °C and then stored at four °C for analysis.

$$\text{Energy consumption (kJ/kg TS)} = \frac{\text{MW power (kW)} \times \text{t (hours)}}{\text{Sample weight(Kg)}}, \quad (1)$$

where *TS* is total solids and *t* is heating time.

Analytical Method

WAS samples were centrifuged at 4,100 RPM by a laboratory centrifuge (NF 800 NÜVE) and the supernatant was filtered using 110 mm "fast" black quantitative filter paper. For sugar and protein analysis, samples were filtered using 0.45 µm, 47 mm radius white filters. Analyzes of sCOD, TS, suspended solids (SSs), and alkalinity were performed according to standard methods (APHA, 2005). Sugar was tested using the anthrone method (Raunkjær et al., 1994). NH^{+}_{4}-N (Merck 14752), PO_4-P (Merck 114848), protein (Merck 1103070500) and sugar were measured using a MERCK PHARO 100 instrument. pH and electrical conductivity (EC) of the samples were determined using Hanna pH and EC meter. Sludge filter resistance (SFR) was calculated based on Eq. (2) according to Wu et al. (2023).

Scanning electron microscopy (SEM) particle size analyzes were performed at Central Laboratory for Advanced Technology Research and Applications of Cumhuriyet University. The d-values (d10, d50, and d90) are indeed commonly used metrics when describing particle size distributions. These values represent the intercepts for 10.00%, 50.00%, and 90.00% of the cumulative mass or volume distribution, depending on the context. They provide a concise way to characterize the particle size distribution of a sample (Baig et al., 2018).

$$\text{SFR (m/kg)} = \frac{2 \times \text{P}(N/m^2) \times f(\text{s/m}^6) \times \text{A}^2(\text{m}^2)}{\mu\ (\text{N.s/m}^2) \times \text{W(Kg/m}^3)}, \quad (2)$$

Table 1. Characteristics of raw & disintegrated WAS

Parameter	Unite	Raw WAS	Disintegrated WAS
pH	-	7.310	7.210
EC	ms/cm	1.401	1.513
TS	mg/L	11,840.000	11,092.000
SS	mg/L	10,760.000	9,230.000
sCOD	mg/L	88.500	2,380.000
Protein	g/L	0.440	1.180
Sugar	mg/L	5.500	66.500
NH_4-N	mg/L	10.000	20.400
PO_4-P	mg/L	11.000	16.300
Alkalinity	mg/L	440.000	422.000
SFR	m/kg	1.02×10^{13}	2.15×10^{14}
Specific energy	kj/kg TS	-	376,200

where P is pressure, A is filter area, f is V, V/t is slope of the graph, μ is viscosity, and W is ratio of cake weight to permeate volume. The 'f' value in the formula is the coefficient of x in the equation that appears on the graph created by writing T/V values on the y-axis and V values on the x-axis.

Experiments and analyses were performed at least three times and average values were presented.

RESULTS & DISCUSSION

Solids Reduction

While TS and SS concentrations of raw WAS were about 11,840 and 10,760 mg/L, after MW irradiation, declining by the ratio of 6.00% and 14.20%, decreased to about 11,091 and 9,230 mg/L in the disintegrated sludge, respectively (**Table 1**). Uma Rani et al. (2013) reported that after applying MW irradiation for 12 minutes, SS decreased by 15.30%. Ebenezer et al. (2015) reported the primary relation for SS removal was the correlation between COD concentration and breakdown of particulate organic compounds by MW irradiation into soluble organic matter. In another investigation, experimental results showed that just 5.00% of TS could be removed, whereas reductions in SS were found to be between 22.00% and 37.00% (Dogruel & Ozgen, 2017). Results indicated that that the distribution of solid particles in biosludges was altered with the pretreated.

Soluble Oxygen Demand Protein & Sugar Release

After MW irradiation, the concentrations of sCOD, protein, and sugar increased from 88.5 mg/L, 0.44 g/L, and 5.5 mg/L to 2,380.0 mg/L, 1.18 g/L and 66.5 mg/L, respectively. The release rate of sCOD, proteins, and sugars from raw WAS were about 2,640.00%, 1,680.00%, and 1,220.00%, respectively. A substantial release of organic materials with MW irradiation on the biosludge flocs was determined.

It was also observed that sCOD (Eskicioglu et al., 2007; Zheng et al., 2009), protein, and sugar (Eskicioglu et al., 2007) release increased between 35-90 °C MW temperatures, but there was a decrease in protein and sugar concentrations due to Maillard reactions at 96°C (Eskicioglu et al., 2007). Koupaie and Eskicioglu (2016) achieved the maximum dissolution rate for sCOD, protein and polysaccharides at the ramping rate of three °C/min, where the irradiation time was prolonged.

Table 2. Variation of metals in raw & disintegrated WAS

Metals (mg/L)	Raw WAS	Disintegrated WAS
Na	128.700	83.000
Ca	222.600	106.800
Mg	24.700	17.800
Zn	0.040	0.200
Al	0.008	0.002
K	23.500	50.800

The release of proteins, sugars and sCOD was at an energy consumption of 376,200 kJ/kg TS for MW pretreatment. Therefore, if AD process is applied after MW disintegration, the difference in CH_4 yield in AD between disintegrated and raw biosludge, which is used as the substrate, should be considered to calculate the energy efficiency.

NH_4-N & PO_4-P Releasing

Nitrogen is a vital component of amino acids, proteins, and nucleic acids. The improvement in the release of NH_4-N from biosludge by MW disintegration under the operational condition was significantly low compared to the improvements observed in sCOD, sugars, and proteins. The concentration of NH_4-N and PO_4-P increased from about 10 mg/L for raw biosludge to about 20 mg/L about 16 mg/L in the disintegrated biosludge, respectively. Bohdziewicz et al., (2011) determined also disintegrating the biosludge under the conditions of temperature at 70 °C with 900 W of MW power for 3.5 minutes of irradiation time, leads to increase in the release of both NH_4-N and PO_4-P. Although low amount of NH_4-N and PO_4-P improvement was observed in this study, about 30.00% decrease of NH_4-N concentration was reported after the heat treatment at temperatures ranging from 80-120 °C for 30 min due to the formation of NH_3, which was the volatile form of nitrogen. Also decrease of PO_4-P concentrations might be occurred due to the phosphate ions reacting with other compounds to form insoluble precipitates or stable complexes (Kumar et al., 2020).

Release of Metals

The exact composition of biosludge and the effectiveness of thermal pretreatment can vary depending on factors such as the source of wastewater, the treatment processes used, and the conditions of pretreatment (Al Ramahi et al., 2020). Additionally, by the pretreatment of biosludges while some elements like N, P, K, Na, and Ca might be released in significant quantities, others might not be as readily mobilized or formed compounds that were less soluble (Sun et al., 2013).

As can be seen from **Table 2**, except for Zn and K, the concentrations of Na, Ca, Mg, and Al were decreased after MW disintegration under the operational condition. It was thought that during the heat treatment of biosolids, release of the soluble organic compounds was enhance the deflocculation with elements such as Ca, and Mg ions (Tonanzi et al., 2021). Wang et al. (2016) observed the decrease in concentrations of Ca^{2+} and Na^{+} with increasing MW temperature due to the precipitation of carbonate or phosphate forms.

Particle Size Distribution

MW irradiation led to an increase in the specific surface area (SSA) of biosludge, which might cause an increase in the

Table 3. Particle size distribution of raw & before pretreated WAS

Type of sludge	d10 (µm)	d50 (µm)	d90 (µm)	SSA (m^2/kg)
Raw WAS	38.80	228.00	368.00	70.80
Pretreated WAS	14.50	44.60	72.80	235.90

yield of various treatment processes such as adsorption, filtration, and chemical reactions (**Table 3**). An increase of 232.40% of SSA values inclined from 70.80 m^2/kg for the raw sludge to 235.9 m^2/kg for the disintegrated biosludge. MW pretreatment also contributed to reduce the particle size as for the d10, d50, and d90 values decreased from about 39, 228, and 368 µm for raw biosludge to 15, 45, and 73 µm for disintegrated biosludge, respectively. The decrease in particle size was related to the increase in SSA of the pretreated biosludge compared to the raw, and it was considered an indicator of the disintegration efficiency, which was directly proportional to the increase in SSA (Martínez et al., 2015). Liu et al. (2019) observed that the floc size (d10, d50, and d90) of MW-pretreated WAS was smaller than the control sample, indicating that MW irradiation was effective in destroying the biosludge flocs. Furthermore, when the application times of MW increased to 12 or 16 min, the size of pretreated-WAS clumps were smaller than that of the blank sample (Liu et al., 2019). MW or hybrid MW treatment processes caused a significant increase in net negative surface charge. This increase was important because it created electrostatic repulsion, which in turn prevents close contact of sludge particles, especially within the colonic fraction (size ranges from one µm to 100 µm) (Liu et al., 2015). Negative surface charge refers to the presence of an excess of negatively charged ions or functional groups on the surface of molecules or materials. In the context of biosludge treatment, an increase in negative surface charge is beneficial because it enhances repulsive forces between particles. This repulsion helps keep the molecules dispersed rather than clumping or closely packed together (Liu et al., 2015).

The long irradiation time (with slow heating rate) resulted in a significant decrease in particle sizes, especially in particles with a diameter of d90, indicating possible fragmentation of the biosludge into smaller pieces (Peng et al., 2013). However, despite the decrease in particle sizes, the dewatering property of the biosludge deteriorated (Peng et al., 2013).

Sludge Filter Resistance

Dewaterability or biosludge filter resistance, which is an important index used to evaluate the condition of sludge. This index typically shows a positive correlation with the content of soluble organic matters and a negative correlation with the size of sludge flocs (Liu et al., 2019). It has been applied to reflect the physicochemical properties of WAS after MW pretreatment (Liu et al., 2019). SFR value of raw WAS was 2×10^{13} and deteriorated significantly after MW pretreatment and reached 2.15×1,014 (**Table 1**). The dewaterability deteriorates with increasing MW irradiation time and indicated that the floc structure was excessively destroyed by MW irradiation, resulting in the release of many intracellular materials in biosludge (Zhang et al., 2021). Therefore, the viscosity of biosludge also increased, which was also why the increase of SRF in biosludge was treated by MW for more than 180 hours (Zhang et al., 2021). It was consistent with our study in terms of the deterioration of dewaterability as the irradiation time exceeded 180 s. In addition, a previous study noted that the value of biosludge dewatering time increased from 44.3 to 198 seconds when the biosludge was exposed to heat for one hour (Liu et al., 2012). This was probably due to the increased proportion of small particles during biosludge thermal processes, and these small molecular substances could easily clog the filter during biosludge filtration (Liu et al., 2012; Zhang & Yao, 2022).

Scanning Electron Micros Images

A difference of the morphological characteristics of the biosludge with SEM between the raw and disintegrated WAS is presented in **Figure 1**. As shown in left part in **Figure 1**, The raw bio-sludge could be characterized by masses with a relatively rough, compact, and irregular surface, which was an indication of a dense, tightly structure, typical of raw biosludge, also indicated the presence of organic and other compounds in a relatively condensed form.

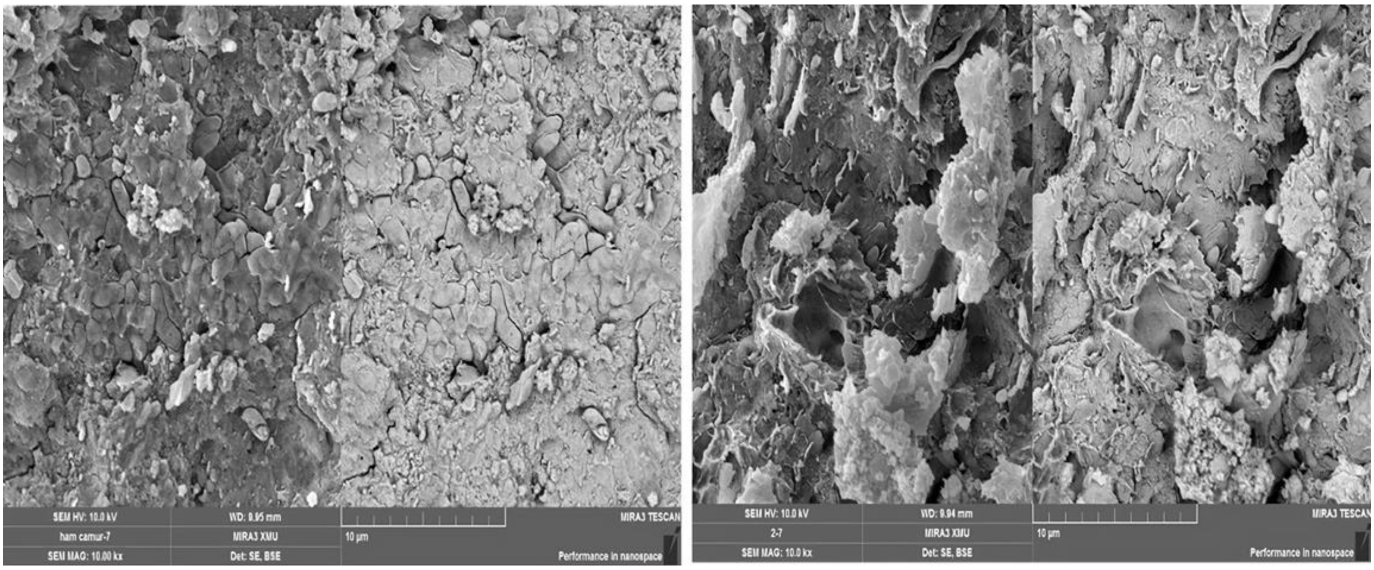

Figure 1. SEM images of raw (left) & MW pretreated biosludges (right) (Source: Field study)

As mentioned before, sCOD concentration was observed in the medium was higher than the raw WAS due to the breakdown of biosludge. The pretreated biosludge, which structure appeared less compact and more granular, due to the breakdown of organic components and the release of trapped fluids, could be characterized as soft, loose, and slightly granular compared to the raw biosludge (right part in **Figure 1**). In a previous study, the control sample appeared to have a smooth, rigid, and compact surface, while MW pretreated sample showed morphological changes on the surface layer, which was characterized by a concave and loosely cracked texture (Veluchamy et al., 2017).

Further examination revealed that many of the healthy cells were tightly integrated or attached to EPS layers in large clusters. With an uneven and sticky structure, the blocks were usually abrasive. The masses were broken down into small particles and distributed uniformly in the liquid phase because of the conditioning of the ultrasonic pretreatment; Thus, the biomolecular polymers were released with the blocks (Bian et al., 2021).

CONCLUSIONS

In this study, MW disintegration of WAS at the target temperature of 80 °C with five minutes retention time was investigated. Results indicated that MW pretreatment had significant effect on the release rate of sCOD, protein and sugars to about 2,640.00%, 1,680.00%, and 1,220.00%, respectively, while a limited improvement of inorganic substances such as ammonium and phosphorus were observed. Compared to the raw WAS, after MW pretreatment, decrease of SSs constituent of 14.00%, whilst SSA increased to about 233.00%. A significant amount of the release of elements from biosludge were not observed except for K and Zn.

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Ethical statement: The authors stated that the study did not require an ethics committee approval. However, all relevant laws, rules and regulations necessary to carry out the study were followed during the study. The study was performed in the environmental engineering laboratories of Sivas Cumhuriyet University, Türkiye.

Declaration of interest: No conflict of interest is declared by the authors.

Data sharing statement: Data supporting the findings and conclusions are available upon request from corresponding author.

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