

Enhancement of Thermophilic Digestion of Food Waste (FW) via Trace Element Supplementation

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Abstract: In this study, the role of trace element (TE) supplementation and performance characteristics of a thermophilic anaerobic digester fed by food wastes (FW) is investigated in the long run, and a representative operational data set for field application is reported over the whole experimental period. Continuous feeding of food wastes with a dry matter of 5% for 150 days was carried out using a 100 L pilot-scale CSTR type anaerobic digester under thermophilic operation conditions. Hydraulic retention time (HRT) and organic loading rate (OLR) were kept around 28 days and less than 3.0 kg oDM m³ day⁻¹, respectively. Volumetric biogas production values were reported to be 0.32 m³ m⁻³ day⁻¹ during the period where there is no TE supplementation; on the other hand, biogas production was doubled (0.69 m³ m⁻³ day⁻¹) following TE supplementation. This corresponds to an average unit biogas production of 317 and 443 L kg⁻¹ oDM, during TE supplementation and no TE supplementation periods, respectively. Statistical analysis indicated that Co, As, Se, and Al were the most significant trace elements affecting the digester performance.

Keywords: Anaerobic digestion, biogas, food waste, thermophilic digestion, trace element.

INTRODUCTION

Anaerobic digestion (AD) has been one of the widespread and oldest processes for bioconversion of organic matter into biogas that is one of the most sustainable renewable energy types [1-4]. AD is also a promising future as an alternative technique that may be used to cope with environmental problems such as global warming, energy security, and waste management [5, 6]. Biogas in the AD process is mainly produced from high biodegradable organic fractions of municipal, agricultural, and industrial organic wastes [1, 7-9].

Food waste and its management are becoming a critical issue due to over consumerism all over the world. The exponential growth in food waste is not only imposing serious threats to our society in terms of environmental pollution and health risk but also causing distress in landfills, which have limited storage capacity [10]. The organic fraction of municipal solid waste (MSW) varies depending on the economic development level of the nations, and it is reported that food waste constitutes 40% in developed countries and up to 75% MSW in developing countries [3, 8]. The generation of municipal solid waste, which is composed of more than 50 % FW, exceeds 30 million tons per year in Turkey [11]. It is also reported by Menon et.al. [12] that approximately 36.4 and 89 million tons of FW are generated annually in the USA and EU,

respectively. The amount of food wasted is expected to increase by 44% globally between 2005 and 2025 [13, 14].

Even though a vast amount of literature reports are available for the anaerobic digestion of food wastes, the majority of these studies focus on mesophilic digestion. On the other hand, the thermophilic AD of food wastes, which has high energy potential, has become an important research topic in recent years [5, 15]. Another trending topic for the enhancement of biogas production from FW is the exploitation of the advantage of trace element supplementation during AD of processes. The balanced availability of various nutrients coupled with the provision of ideal growth conditions is essential for well-working anaerobic digesters. Disruptions to one, or more, of those factors may disturb the activity of specific groups of organisms and impair digester performance. The requirement for feedstock well balanced in carbon, nitrogen, and phosphorus (C/N/P) is well understood for optimal AD operation. However, the role of TE in mesophilic but also thermophilic digesters has been relatively neglected by researchers and operators. The first studies on the importance of TE in AD were done in the 1980s, with the work of Callander and Barford [16, 17] and Speece *et al.* [18]. Those studies shifted the view of heavy metals, such as nickel or cobalt, as dangerous substances to essential elements supporting microbial growth in AD. Callander and Barford [16, 17] also introduced the notion of chemical speciation and availability as fundamental concepts in assessing the role of TE on microbial activities.

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In this study, it is aimed to study the long-term effect of both thermophilic operation mode and trace element supplementation at a pilot-scale digester continuously fed under thermophilic conditions for 150 days.

MATERIAL AND METHODS

Source of Food Wastes (FW) and Inoculum

FW used in this study was kindly provided by the Municipality of Izmir Metropolitan City, Solid Waste Management Department in Izmir, Turkey on a weekly basis. FW was first ground and diluted down to 5% Dry Matter (DM, on the average) by adding water before its use in the digester. The thermophilic inoculum was supplied from an industrial AD plant treating wastewater from an alcohol distillery. This inoculum was adapted to FW for 1 month before use.

Analytical Methods

The parameters such as pH, DM%, oDM%, COD, TN, free NH_3 and $\text{NH}_4\text{-N}$ were measured according to "Standard Methods for the Examination of Water and Wastewater". All chemical solutions were prepared with deionized water (Milli-Q® Ultrapure Water Purification System, Millipore Corp.). Biogas production in the digester was measured by wet type gas meter (Ritter). Methane content of biogas was analysed by using a Gas Chromatograph (6890N Agilent) with a flame ionization detector and a DB-FFAP 30-m x 0.32-mm x 0.25-mm capillary column (J&W Scientific, USA) [19]. Trace elements were analyzed by ICP-MS in accordance with APHA 3125 standard.

Experimental Set-Up

The custom made pilot-scale digester used in this study had a total volume of 100 L (liquid volume 70 L). The temperature and mixing conditions were automatically controlled throughout the experiments. The content of the bioreactor was continuously mixed at 50 rpm using an anchor agitator propelled by an overhead stirrer. The temperature was kept constant at 55°C ($\pm 1^\circ\text{C}$) controlled by an external water heater system and thermostat.

Biogas production was measured by Ritter wet type gas meter on a daily basis. Ground and diluted FW (5% DM) was fed daily into the digester in a way to keep the hydraulic retention time (HRT) and organic loading rates (OLR) to be around 30 days and less than $3 \text{ kg oDM m}^3 \text{ day}^{-1}$, respectively.

TE stock solutions used in the study were prepared according to the prescription of the authors' previous

studies (unpublished work). Content and concentration of TE stock solution was as follows; *Fe* (17 mg L^{-1}), *Co* (20 mg L^{-1}), *Cu* (29 mg L^{-1}), *Mn* (48 mg L^{-1}), *Mo* (7 mg L^{-1}), *Ni* (1.5 mg L^{-1}), *Se* (49 mg L^{-1}), *W* (20 mg L^{-1}), *Zn* (61 mg L^{-1}), *B* (17 mg L^{-1}), *Al* (3.2 mg L^{-1}).

Statistical Analysis

The statistical analysis was performed to determine the effects of different trace elements, statistically according to biogas production potentials. The concentrations of the effluent trace elements were grouped into 3 or 4 according to concentration intervals, and the groups were compared with ANOVA and further post-hoc test, Tukey, by using PAWS statistics 18.0.

RESULTS AND DISCUSSION

Characterization of Food Waste (FW)

Characterization of FW used in the study is given in Table 1. The C/N ratio of the FW was in the range of

Table 1: Characteristics of FW Examined in this Study [11]

Constituent	Value
pH	4.5-4.6
DM %	10.5-12.3
oDM %	80-92
TCOD (g L^{-1})	84-101
$\text{NH}_4^+\text{-N}$ (mg L^{-1})	100
TN (g L^{-1})	3.3-3.6
C/N	23-30
Sb (mg L^{-1})	0.0018-0.0037
Pb (mg L^{-1})	0.031-0.098
Cr (mg L^{-1})	0.055-0.096
Zn (mg L^{-1})	2.292-2.680
Hg (mg L^{-1})	0.011-0.014
Cd (mg L^{-1})	0.006-0.008
Cu (mg L^{-1})	1.382-1.746
B (mg L^{-1})	1.741-1.825
Ni (mg L^{-1})	0.197-0.237
Sn (mg L^{-1})	0.009-0.0141
As (mg L^{-1})	0.035-0.036
Fe (mg L^{-1})	17.150-20.190
Na (mg L^{-1})	125.2-240.6
Ag (mg L^{-1})	0.0130-0.022
Co (mg L^{-1})	0.099-0.111
Ba (mg L^{-1})	0.468-0.807
Mn (mg L^{-1})	1.511-2.952
Se (mg L^{-1})	0.012-0.043
Al (mg L^{-1})	9.413-10.410

23-30, which is quite amenable for AD. The total nitrogen concentration was found to be in the preferable ranges.

Performance Evaluation of Anaerobic Digester (AD)

Continuous feeding of a 100 L pilot-scale CSTR type anaerobic digester using FW mixture with a dry matter of 5% was carried out under thermophilic operational conditions ($55^{\circ}\text{C} \pm 1^{\circ}\text{C}$) during the first period of the study (first 40 days of operation) without TE supplementation. The rest of the study was carried out with TE supplementation using the TE recipe given in Table 1. Hydraulic retention time (HRT) and organic loading rate (OLR) were kept around 30 days and less than $3.0 \text{ kg oDM m}^{-3}\text{day}^{-1}$ in each study period, respectively. pH was stable in the digester, i.e., around 7.5-8.0, hence there was no need to provide external alkalinity throughout the study.

Figure 1 shows the effect of TE supplementation on the biogas production and enhancement of anaerobic degradation of FW under thermophilic conditions. As seen in Figure 1a, volumetric biogas production performance is enhanced by almost 212% and biogas production value of $0.33 \text{ m}^3 \text{ m}^{-3} \text{ day}^{-1}$ during the period of no TE supplementation increased up to $0.7 \text{ m}^3 \text{ m}^{-3} \text{ day}^{-1}$ upon TE supplementation on the average. Similarly, a significant enhancement in unit gas production was observed, as seen Figure 1b in which unit gas production increases up to $443 \text{ L kg}^{-1} \text{ oDM}$ from a value of $317 \text{ L kg}^{-1} \text{ oDM}$. These results also clearly indicate that TE supplementation resulted in 40% better degradation of volatile organic matter. Statistical analysis conducted by ANOVA followed by the TUKEY test also proved that the results are statistically significant. Methane content did not vary significantly which were between 55-65% through out the experiments.

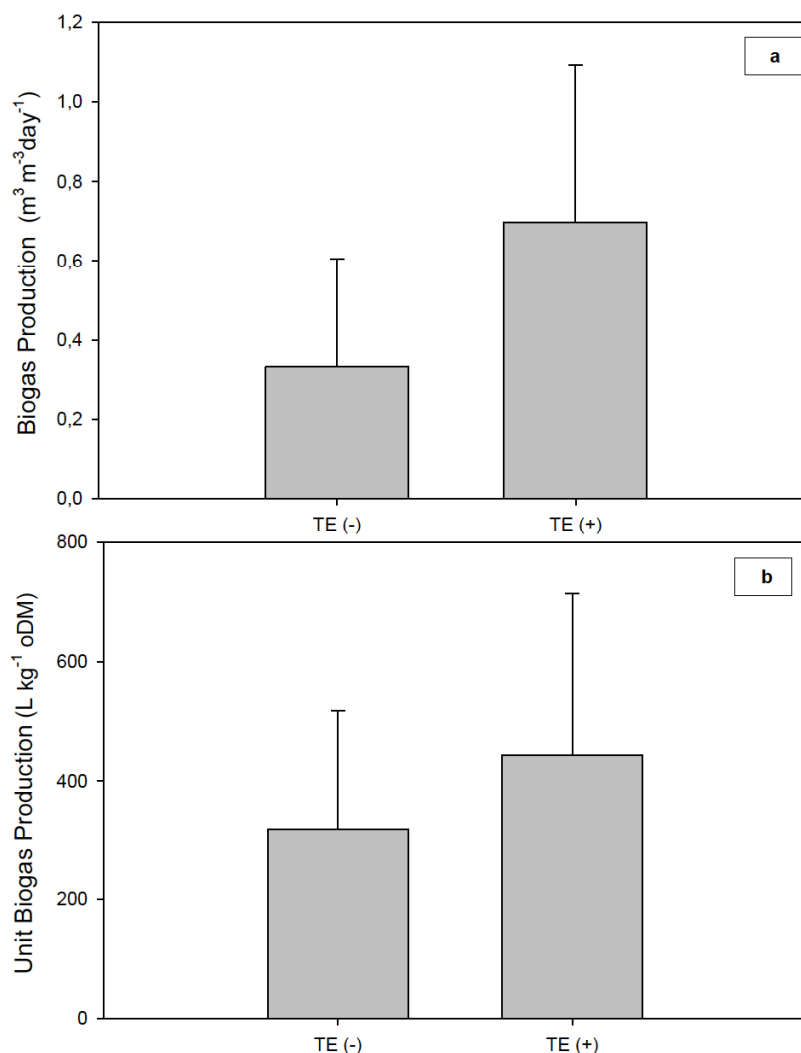


Figure 1: Effect of TE supplementation on biogas production performance **a)** $\text{m}^3 \text{ m}^{-3} \text{ day}^{-1}$, **b)** $\text{L kg}^{-1} \text{ oDM}$.

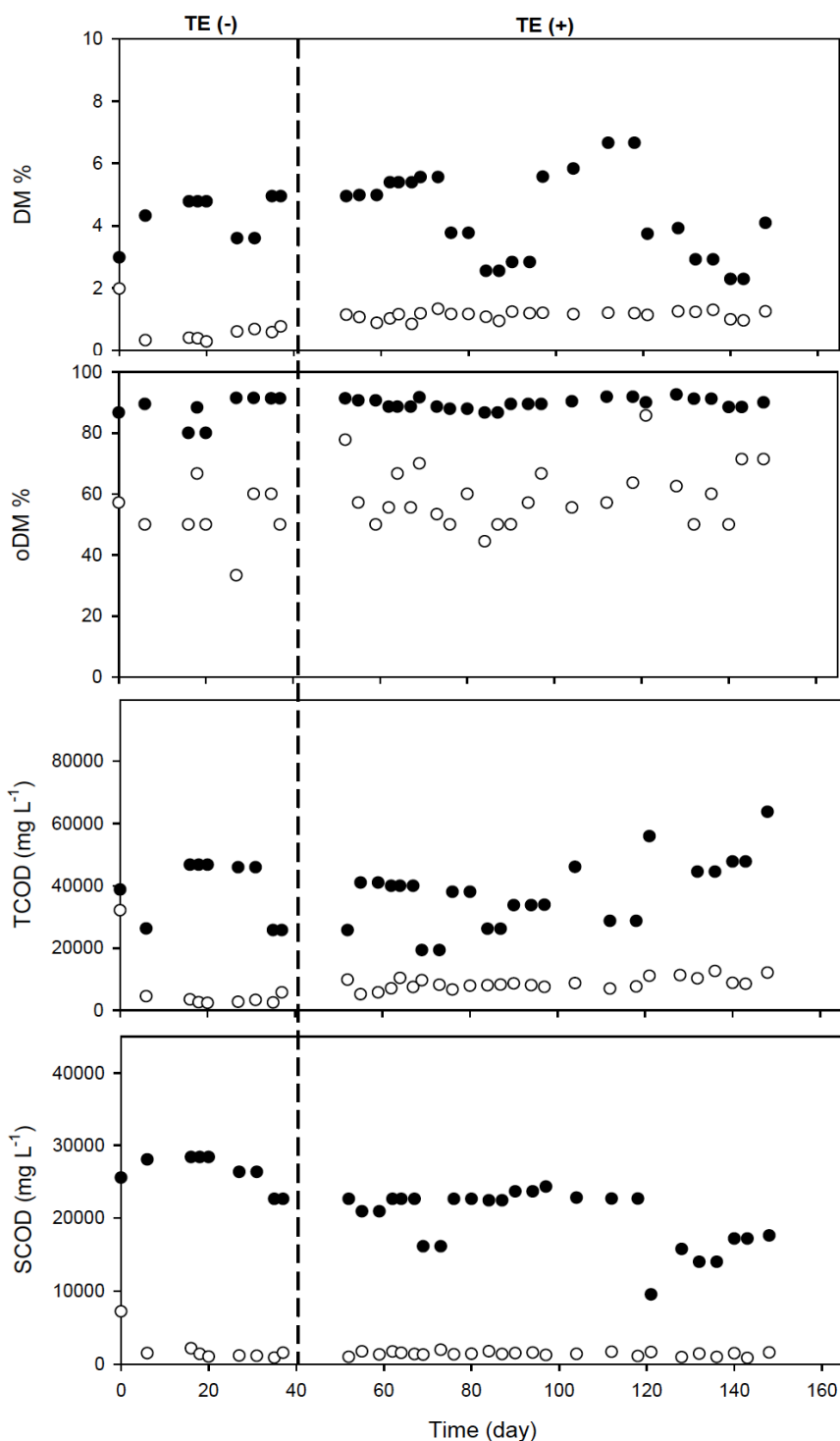


Figure 2: Variation of operational parameters: DM%, oDM%, SCOD and TCOD (● influent, ○ effluent).

Figure 2 shows the variation of operational parameters such as DM%, oDM%, soluble and total COD in influent and effluent. DM values in the influent varied between 3.6-5.0% during the first period of the study (no TE supplementation) and 2.6-6.7% during the TE supplementation period, respectively. DM values in the effluent were measured to be between 0.3-0.8%

and 0.8-1.3%, corresponding to DM removal of 87% and 71% for the period of TE supplementation and no TE supplementation. Similarly, oDM values in the influent varied between 80-91% during the first period of the study (no TE supplementation) and 87-92 % during the TE supplementation period, respectively. oDM values in the effluent were measured to be

between 50-78% and 44-85%, respectively. These values correspond to DM removal of 87% and 80% for the period of TE supplementation and no TE supplementation. Qiang *et al.* [20] studied the effect of Fe, Ni and Co supplementation using high solid food waste (14.3% DM) biogas digester with a volume of 12 L operating at 55°C. In this study, TS removal was reported to be 74% and 71 % for the periods of TE supplementation and no TE supplementation, respectively. They also reported better VS removal values, which are 78% and 72% for the periods of TE supplementation and no TE supplementation periods, respectively.

Narra *et al.* [21] studied bio-methanation of rice straw (RS) in a batch mode at high total solid content (TS) of 25% in outdoor pilot-scale digesters. The performance was monitored for over six months by supplementing Nickel and Cobalt 15 and 10 mg kg⁻¹ RS, respectively, to each of mesophilic and thermophilic digesters for 35 and 21 days retention time. The average biogas production from mesophilic and thermophilic digesters were found varying 310 and 396 L kg⁻¹ TS, respectively. The control values with no TE supplementation were reported to be 225 and 270 L kg⁻¹ TS, which correspond to 37 and 46% higher biogas production by supplementing the micronutrients in mesophilic and thermophilic digesters, respectively. The results demonstrated that TE supplementation resulted in a faster process with having less than 85% water requirement compared to the conventional process. Ariunbaatar *et al.* (2016) [22] studied the effect of supplementing trace elements (Fe, Se, Co, Ni, Zn, Mn, Cu and Mo) on the enhancement of anaerobic digestion of food waste by using batch experiments. According to this study, the most effective trace elements were Fe with an increase of 39.2% of biomethane production, followed by Se (34% increase), Ni (26% increase) and Co (24% increase). In addition, the authors concluded that TE supplementation enhanced volatile fatty acids's consumption, thus yielding a higher biomethane production.

Figure 2 also shows influent and effluent values for total (TCOD) and soluble (SCOD) chemical oxygen demand values by time. In the first period of the study where there is no TE supplementation, TCOD_{influent} and TCOD_{effluent} were measured as 26-45 g L⁻¹ and 3-6 g L⁻¹, respectively. It was calculated that 90% removal in COD was achieved in the first period of the study. Interestingly, high use of COD did not result in high biogas production in this period, most likely due to the fact that COD consumption was forwarded to the cell

growth but not biogas production. During the second period of the study where TE was supplemented, TCOD_{influent} and TCOD_{effluent} were measured as 20-64 g L⁻¹ and 5-12 g L⁻¹, respectively. In contrast to the period where no TE is supplemented, COD removals during the TE supplementation period were observed to be low (76%). On the other hand, higher volumetric biogas production in this period was measured indicating that TE supplementation was effective in terms of exploiting organic material.

Figure 3 shows the concentration variation of nitrogen species (TN, NH₄-N, NH₃-N) throughout the study. In the first period, TN_{influent} and TN_{effluent} were in the range of 400-740 mg L⁻¹ and 385-690 mg L⁻¹, respectively. In the second period, an increase was observed in TN due to the seasonal change in food waste content (TN_{influent} and TN_{effluent} : 500-1100 mg L⁻¹). In regards to NH₄-N, NH₄-N_{influent} and NH₄-N_{effluent} were 60-180 mg L⁻¹ and 200-370 mg L⁻¹, respectively. The higher NH₄-N values in the effluent indicate solubilization of TN. Similarly, in the second period of the study, NH₄-N_{influent} and NH₄-N_{effluent} were measured to be in the range of 75-225 mg L⁻¹ and 280-550 mg L⁻¹, respectively. It was observed that influent NH₃ values were significantly low in both study periods (5-9 mg L⁻¹ and 7-13 mg L⁻¹). As a function of the pH values of the digester, NH₃ values in the effluent increased up to 44-77 mg L⁻¹ and 116 mg L⁻¹ for both study periods, respectively.

Variations of influent and effluent concentration for Al, Co, As, and Se elements, which were found to be statistically significant results on the biogas production, are shown in Figure 4.

In the first period of the study, Al_{influent} and Al_{effluent} concentrations were found to vary to between 3-19 mg L⁻¹ and 0.6-9.0 mg L⁻¹, respectively. It was observed that Al concentrations in the second period of the study did not change much and Al_{influent} and Al_{effluent} concentrations were in the range of 2.0-17 mg L⁻¹ and 2.5-8.3 mg L⁻¹, respectively. The concentration of Co_{influent} and Co_{effluent} values were found to be very close to each other and varied from 0.03 to 0.7 mg L⁻¹ up to the 84th day of the study, on the other hand, there had been a jump in Co_{influent} concentrations (14-24 mg L⁻¹) during the days of 84th and 104th. Co_{effluent} concentrations during this period were 2.8-10 mg L⁻¹. Following the 104th day of the study, Co values started to descend and stabilized again (Co_{influent}: 0.3-3.0 mg L⁻¹; Co_{effluent}: 2.1-9.5 mg L⁻¹). As values were measured to be significantly low in comparison to other TE, which

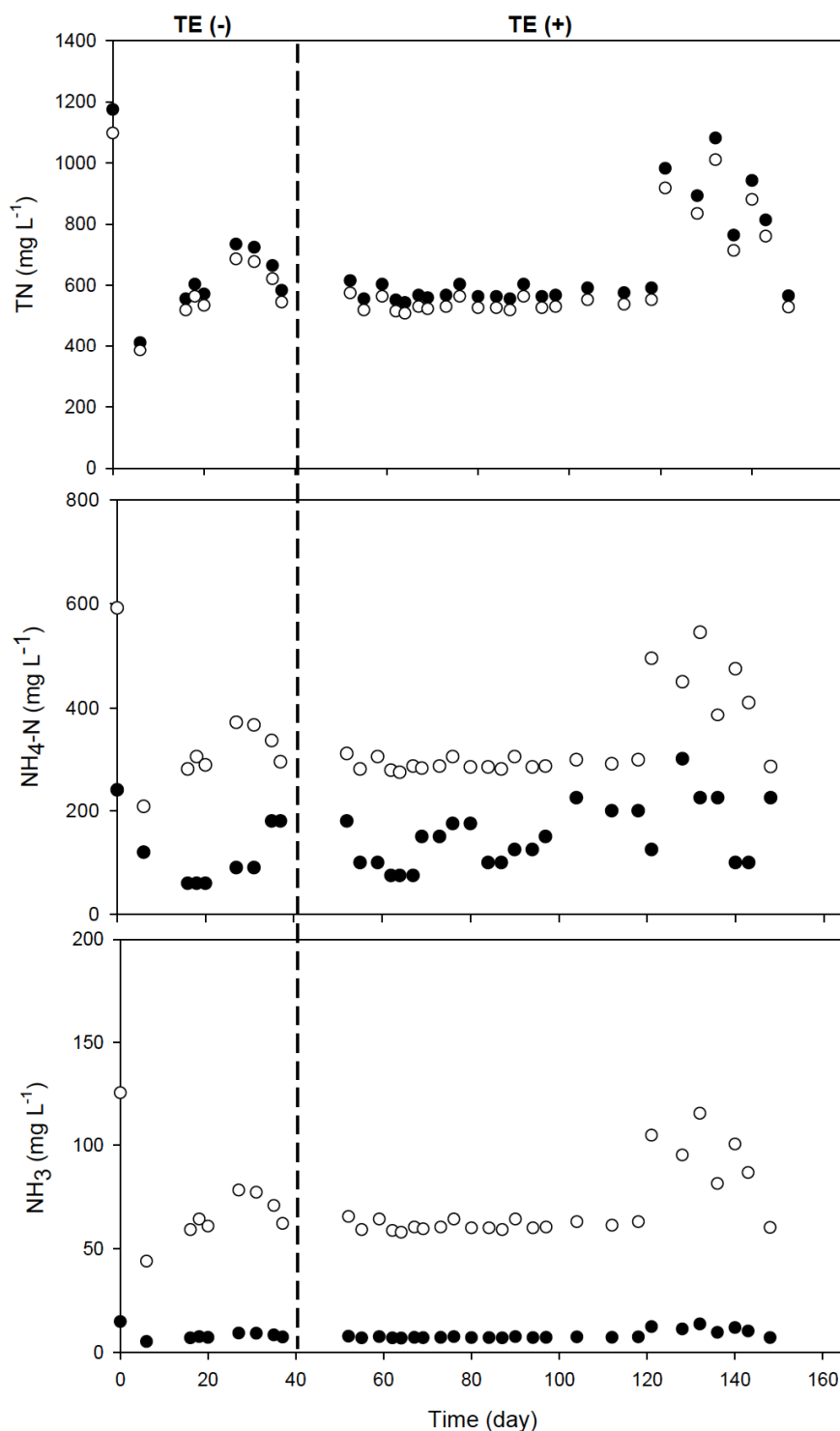


Figure 3: Variation of nitrogen species throughout the study (● influent, ○ effluent).

varied between 0.005-0.08 mg l⁻¹ for both influent and effluent up to day 118. Concentrations both in influent and effluent increased up to 0.6 mg l⁻¹ afterwards. Similar to Co and As, Se values were also low (0.02-0.45 mg l⁻¹) up to the day of 80, extremely high values of Se (29-77 mg l⁻¹) were observed between the days of 80 and 103. These high values then descended and

stabilized (Se_{influent} 0.02-0.2 mg l⁻¹ and Se_{effluent} 0.4-0.7 mg l⁻¹).

Results of Statistical Analysis

The different groups of each trace element, according to concentrations, were compared by

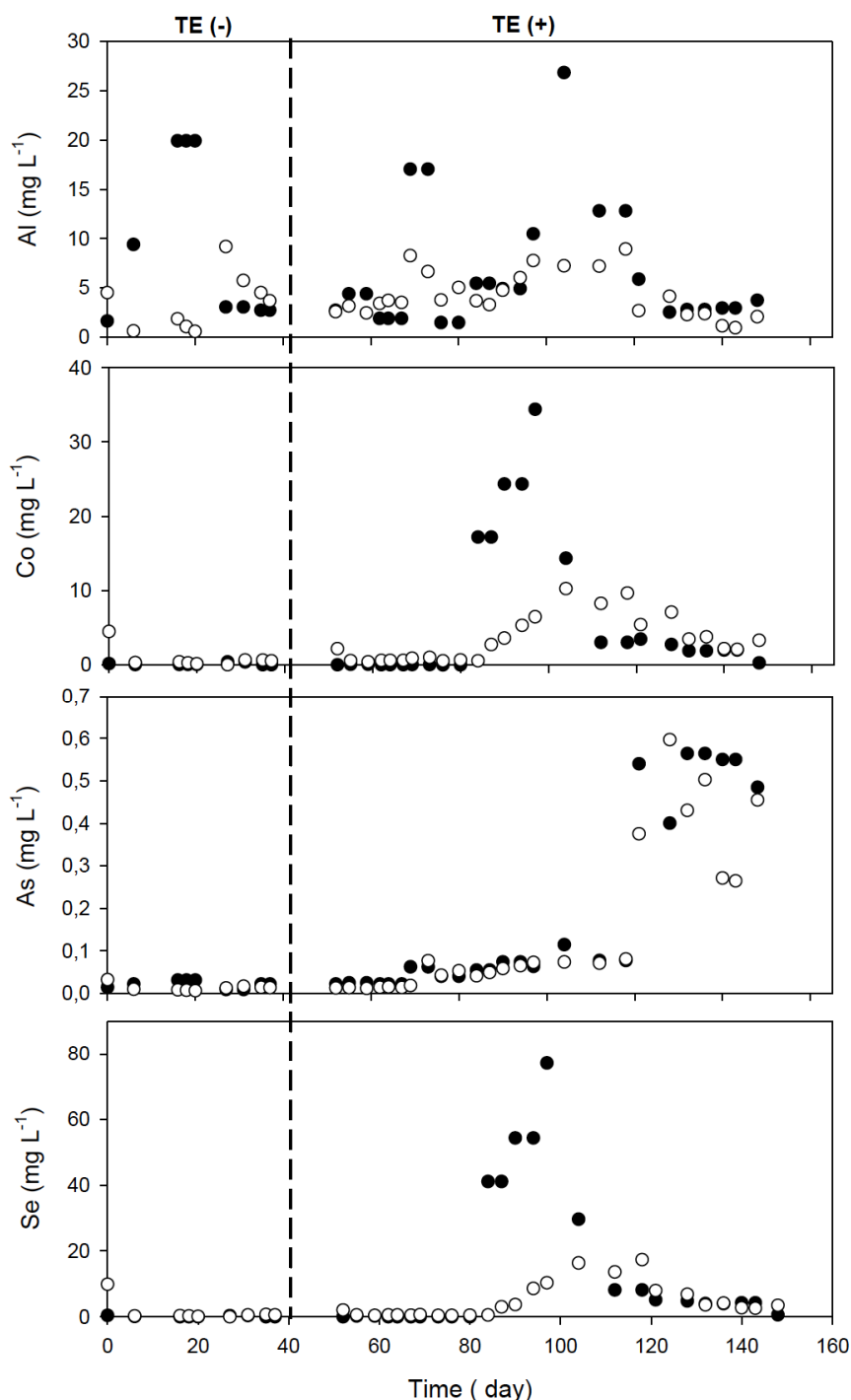


Figure 4: Variations of concentration of Al, Co, As and Se elements (● influent, ○ effluent).

ANOVA and Tukey tests by using PAWS Statistics 18.0. The results of the statistical analysis showed that the trace element concentrations in the effluent are significant.

Statistical analysis for all trace elements was carried out grouping as 3 ranges of concentrations. For example, the concentrations of Al was grouped into 3 concentration ranges as; Group1: 0.3-0.7 mg L⁻¹;

Group2: 3.07-6.14 mg L⁻¹ and Group3: 6.14-9.20 mg L⁻¹ (see Figure 5 for the group ranges for Co, As and Se). Amongst all these TEs studied, the concentrations of Co, Al, Se and As were found to be statistically significant (Table 2). Therefore, Figure 5 only the results of those TEs, which are significant, were included. The difference between groups was found to be significant ($p < 0.05$) for Co, Al, As and Se. Then, a post hoc analysis was also performed to find out which

Table 2: ANOVA Results for Elements ($p=0.05$)

Trace element	F value	p
Co	4.088	0.027
Al	4.738	0.015
As	3.803	0.034
Se	3.834	0.033

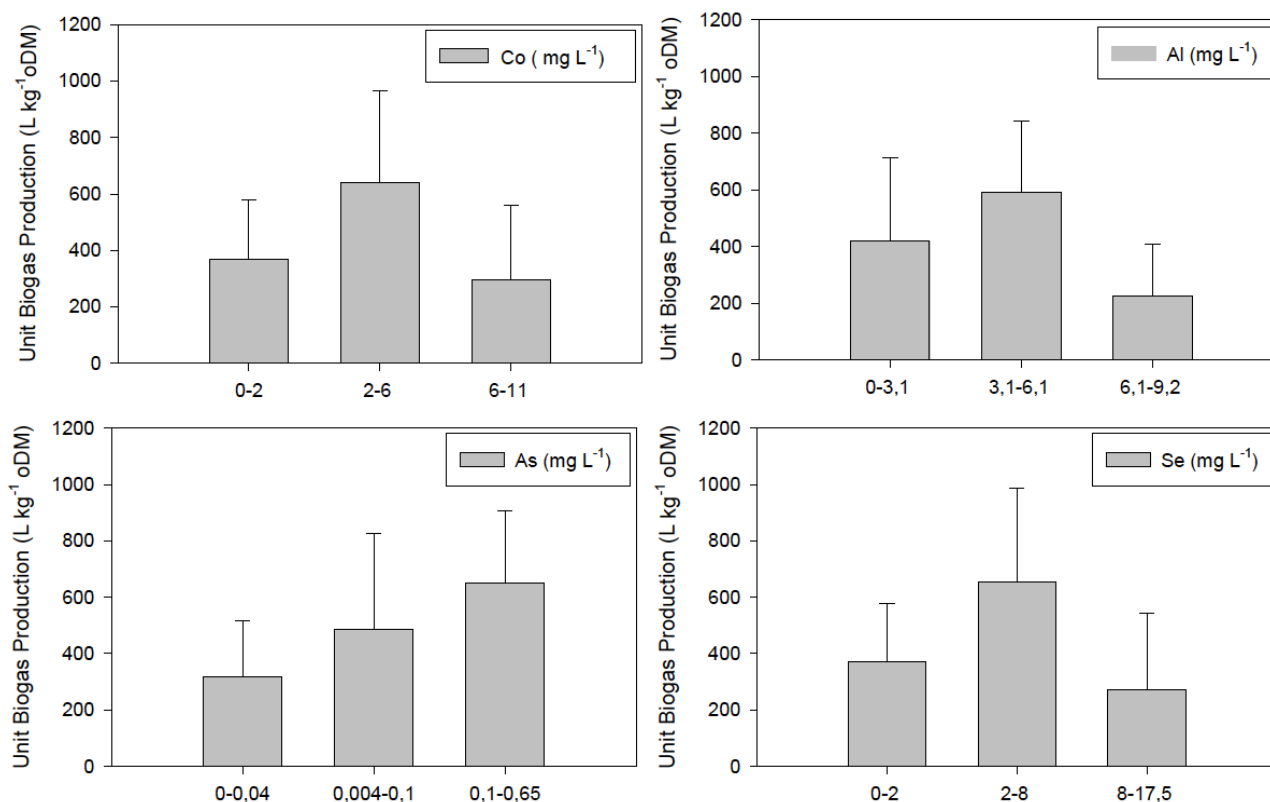


Figure 5: Effect of Co, Al, As and Se on unit biogas production.

groups are significant for each TE. For Al, the concentration range of group 3 (6.14-9.20 mg L⁻¹) was found to be significantly different. Similarly, Co, As and Se concentrations in the effluent were also analyzed after dividing them into 3 groups. The difference between concentrations was found to be significant for all 3 elements as $p=0.027<0.05$; $p=0.034<0.05$ and $p=0.033<0.05$, respectively. A comparison between groups indicated that the statistically different group for Co was found to be 6-11 mg L⁻¹, which resulted in the lowest unit gas production. Statistically different groups for As and Se were as follows; 0-0.04 and 0.1-0.65 mg L⁻¹ for As and 0-2 and 8-17.5 mg L⁻¹ for Se.

Figure 5 shows the effect of selected TEs (Co, Al, As and Se) on the unit biogas production (L kg⁻¹ oDM). Except for As, which had a linear effect on biogas

production, all other TEs (Co, Al and Se) showed an optimum range for TE supplementation. All these three TE (Co, Al and Se) indicated deficiency and overdose effects on the unit biogas production depending on the applied dose. The best TE concentration ranges were determined to be as follows for each TE studied: Co: 2-6 mg L⁻¹, Al: 3.1-6.1 mg L⁻¹, As: 0.1-0.65 mg L⁻¹ and Se: 2.0-8.0 mg L⁻¹.

Lindorfer *et al.* [23] conducted a TE supplementation study on full-scale AD plants where over 1500 anaerobic digesters were sampled for their TEs levels and their performances (60 ADs presenting a low TE level). This study indicated that the TE supplementation decreased the level of VFAs in those digesters, boosted the microbial biomass and enhanced digester performance. They showed an

improvement of the AD performance within the few days following the TE supplementation, and it stayed stable for at least three months after the TE supplementation.

CONCLUSIONS

Anaerobic digestion of FW has a great potential to be used as a renewable and environment friendly energy alternative. This study provides representative operational data for field application of anaerobic digester running on FW. The results also provided precious data in regards to the management of the digestate, especially from the point of residual organics and trace element content.

In order to avoid risks with TE supplementation, it is very important to develop an appropriate dosing strategy, ensuring that each TE concentration is carefully managed between the two critical zones (deficiency and inhibition or even toxicity). Overdosing of at least one TE may result not only in inhibitory effects inside the bioreactor, with a decrease in the reactor performance, but also can lead to an environmental risk once the digestate is discharged into the environment. On the other hand, it is obvious that the right amount of TE supplementation results in significant performance enhancement in digesters.

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