



PRELIMINARY INVESTIGATION OF ANAEROBIC CO-DIGESTION POTENTIAL OF PRIMARY AND SECONDARY SEWAGE SLUDGE FRACTIONS WITH FRUIT WASTE

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ABSTRACT: Co-digestion feasibility of primary (PS) and secondary (SS) sewage sludge fractions produced in a municipal wastewater treatment plant (WWTP) was investigated by the mixing of fruit waste in sequential batch reactors. Toxic threshold level for pH was exceeded as fruit waste digestion produced a high level of volatile fatty acid (VFA) concentration. Alkali (NaOH) addition provided an optimum level of pH at 7.95-8.05 and 8.16-8.25 in the co-digestion of PS and SS, respectively, and efficient conversion of VFA to methane gas eliminating VFA accumulation. Buffered co-digestion resulted in 55% (+227-271 mL) and 400% (+501-546 mL) increase in biogas production compared to solely PS and SS stabilization. Fruit waste digestion's positive effect was obtained at a higher level in SS digestion. pH rather than VFA proved to be the key parameter and NaOH highly effective as an alkali source producing double buffering effect by converting carbonic acid produced to bicarbonate alkalinity. High carbohydrate content of fruit waste exhibited a beneficial potential to neutralize high pH occurring in PS and SS digestion, thus, minimize free ammonia toxicity balancing hydroxide alkalinity formed from protein hydrolysis. Optimization of fruit waste loading rate based on pH will enable surplus energy gain and stable operation in sewage sludge and protein-rich wastewater digestion.

Key Words: Sewage sludge, co-digestion, organic waste, biogas, energy.

Primer ve Sekonder Arıtma Çamur Fraksiyonlarının Meyve Atığı ile Anaerobik Çoklu Besiyeri Çürütmesi Ön Çalışması

ÖZ: Kentsel atıksu arıtma tesislerinde (AAT) oluşan primer (PÇ) ve sekonder (SÇ) arıtma çamurlarının meyve atığı ile birlikte anaerobik çürütme çalışması ardışık kesikli reaktörlerde yürütülmüştür. Proseste meyve atığının parçalanması ile yüksek seviyede uçucu yağ asitleri (UYA) oluşarak toksik pH eşiği geçilmiştir. Alkali kaynağı olarak NaOH ilavesi sonrasında pH PÇ ve SÇ için sırasıyla 7.95-8.05 ve 8.16-8.25 olarak alkali seviyelerde gerçekleşmiş ve UYA'nın etkin metana dönüşümü ile biyogazda sırasıyla %55 (+227-271 mL) ve %400 (+501-546 mL) artış elde edilmiştir. Meyve atığının aynı alkali şartta SÇ için daha yüksek verim ve proses stabilitesi sağladığı gözlenmiştir. Proses için UYA'dan ziyade pH'nın anahtar parametre olduğu belirlenmiştir. NaOH, alkali kaynağı olarak proseste oluşan karbonik asiti bikarbonat alkalinitesine çevirdiği için çift kat etki sağlamıştır. Meyve atığının karbonhidrat içeriği, PÇ ve SÇ çürütmesinde oluşan yüksek pH'nın nötralize edilmesinde faydalı katkı sağlamıştır. Sonuç olarak meyve atığının özellikle SÇ çürütmesinde ilave substrat olarak enerji eldesini arttıracakları belirlenmiştir. Organik yükleme hızının pH bazında optimize edilmesi ile alkali ilavesiz arıtma çamurlarının ve proteinli atıkların çoklu çürütmesinin kararlı işletimle gerçekleştirilmesi de mümkün olacaktır.

Anahtar Kelimeler: Arıtma çamuru, anaerobik, çürütme, organik atık, biyogaz, enerji.

1. INTRODUCTION

Sewage sludge produced as primary (PS) and secondary (SS) sludge fractions in municipal wastewater treatment plants (WWTP) are mixed, thickened and fed to anaerobic digesters to obtain useful methane/energy and volatile solid (VS) reduction. Sewage sludge stabilization and disposal usually make up more than half the overall operational costs of the WWTP (Mininni *et al.*, 2015). An approximate 40-50% of the VS in the mixed sludge is degraded producing a biogas composed of mainly methane (65%) and CO₂ (35%) which is converted to electrical energy via regenerators (Metcalf and Eddy, 2003). The energy obtained has a potential to recover 40 (in case of solely PS digestion) to 70% of the WWTP's total daily operation costs where 100% recovery is possible depending on the rate of solid loading and/or organic content. Each sludge fraction owns its specific content and ability to biodegrade. PS is the sum of settleable solids separated in the first settling tank on the main line of a municipal WWTP and consists of raw organic material as proteins and lipids with a high degree of degradability. SS is the settled bacteria grown in the biological treatment unit (activated sludge process) and resistant to anaerobic degradation. As the sludge age applied increases in nutrient removing activated sludge systems, the bacteria get older and gain further resistance to anaerobic biodegradation and methane yield drops in such WWTPs. Conversion rate to methane is faster for PS because SS is composed of viable microorganisms and extracellular polymeric substances for which hydrolysis is the limiting step of anaerobic digestion which proceeds at a low rate. Lab-scale studies investigated pre-treatment methods' efficiency to improve biodegradability such as thermal, ultrasonic and wet oxidation prior to anaerobic digestion for which cost-efficiency has not been proved yet for full-scale application (Carrere *et al.*, 2010). There is also a contrast in the pre-treatment of SS as high dewaterability is largely reduced leading to higher cost for chemical in the decanter unit (Erdirencelebi *et al.*, 2017).

Anaerobic co-digestion of sewage sludge with organic wastes of food and agricultural origin is an attractive alternative which helps increase methane production and is advantageous over composting with useful energy recovery, increased anaerobic degradability and stabilization in a shorter time scale (Demirer *et al.*, 2001). Anaerobic biotechnology is a very valuable process in the renewable energy field as the most economic method to stabilize organic wastes for which a predicted production of 2.2 billion ton worldwide is given by United Nations' FAO until 2025 (Ariunbaatar *et al.*, 2014; 2015).

In the present paper, a preliminary lab-scale anaerobic co-digestion study was conducted with PS and SS fractions as parallel batch and sequential batch feeding sets with multiple substrate as fruit waste and process performance was investigated on biogas and volatile fatty acid (VFA) production and pH/HCO₃⁻ balance as the key parameters of the anaerobic stabilization.

2. MATERIAL AND METHODS

2.1. Batch Study

The study was started for PS in two 1000 mL volume glass and special-made reactors with gas outlets in the first part of the study. Control reactor received 700 mL anaerobic inoculum sludge and 100 mL PS whereas the second reactor received an additional fruit mix at 100 mL volume. The reactors' incubation temperature was adjusted to 35(+/-0.5)°C. The process was monitored for 17 d and evaluated with biogas production and than the second part's operational setup was configured.

2.2. Sequential Batch Study

Eight 250 mL special-made glass reactors were used in paralel for PS, SS and fruit waste digestion. Two paralel reactors were set for each feeding condition where control/reference reactors were set in paralel to compare the process performance with multiple substrate digestion. The control reactors received 100 mL of inoculum sludge, 25 mL of PS or SS and others an additional 25 mL of fruit mix. The reactors' incubation temperature was adjusted to 35(+/-0.5)°C. Four subsequent feedings were

implemented at durations of 16-22 d depending on the biogas production. pH, HCO_3^- and VFA were determined in the end of feeding periods.

2.3. Sludge and Substrate Characteristics

Inoculum, PS and SS samples were obtained from the anaerobic sludge digester outlet, primary settling tank outlet and recycle line of the main-line biological treatment unit of the municipal WWTP, respectively. Winter fruit waste was collected from the market place and blandered to pieces smaller than 3 mm and stored at 4°C. Volatile (VS) and total solid (TS) concentrations were measured at ranges of 12465-35810 mg VS/L and 18270-48700 mg TS/L for PS and 2620-10385 mg VS/L and 5315-13040 mg TS/L for SS.

2.4. Analytical Methods

Biogas was measured by liquid displacement method. VFA and HCO_3^- were determined by a two-point titrimetric method according to Anderson and Yang (1992). TS and VS concentrations were measured via standart methods of 2540-B and C (APHA, 2005). A multi parameter Hach Lange HQ40d was used for pH measurement and titration.

3. RESULTS AND DISCUSSION

The batch study with PS showed a significant inhibitory effect in case of multiple substrate digestion (Fig. 1). Fruit waste feeding produced 62% lower biogas compared to PS digestion only which indicated a strong inhibitory effect. A delaying effect also was observed in the inhibited digestion as inoculum was adapted to PS as a substrate but not to fruit waste. Delaying effect in the biogas production indicated that hydrolysis of fruit waste generated some intermediate products that hindered methanogenesis, thus, methane production. Recovery did start on the 3rd day but stopped on the 12th day. The pattern revealed that inhibitory intermediate products increased in concentration as digestion progressed and were severely effective on methanogenesis.

The biogas yield in PS digestion was obtained at a high level as 0.44 L/g VS_{added} . High anaerobic degradability of PS was supported by the high yield value.

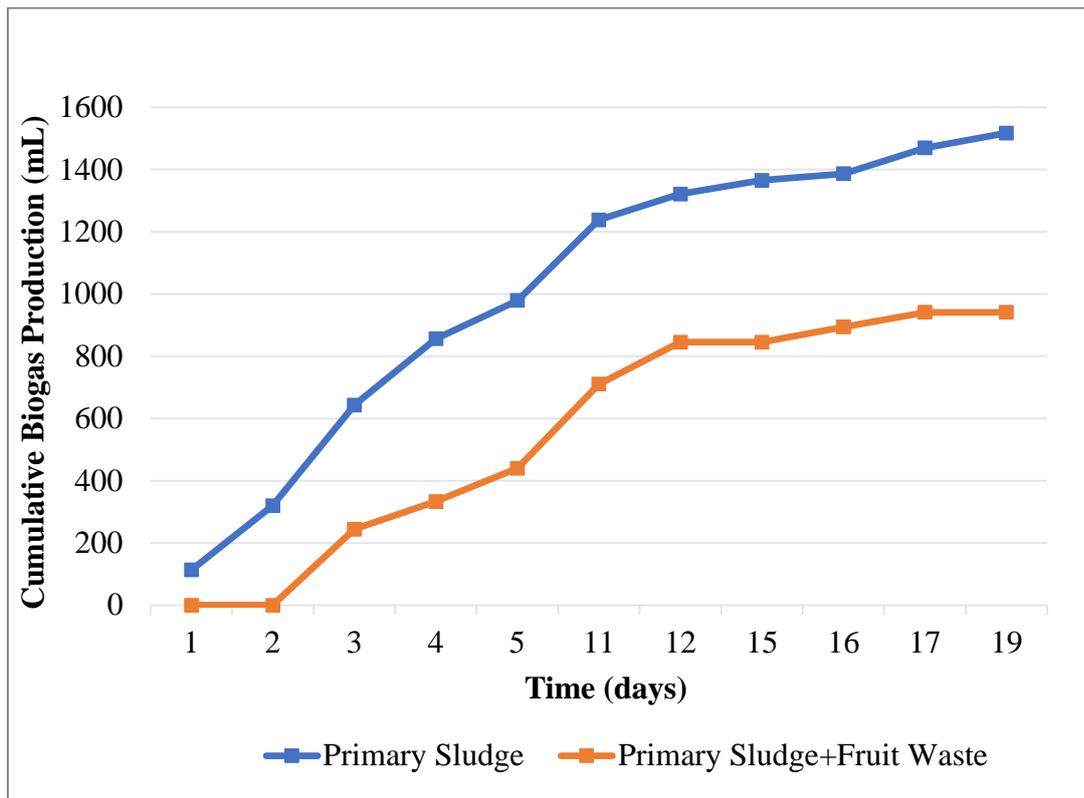


Figure 1. Comparative cumulative biogas production in PS digestion with fruit waste

In the sequential batch feeding part, smaller reactors were used as 2 parallel for each digestion style. The same pattern as lower amounts at 50% in biogas production was observed in PS digestion with fruit waste compared to only PS digestion in the first two feedings (Fig. 2). Raw sludge changed and VS content increase induced higher biogas production in the 3. feeding where VS load tripled promoting a similar effect on methanogenesis in PS digestion where VFA at 492 mg CaCO₃/L in one reactor indicated a delay or inhibition effect resulting in lower biogas production. As PS is the sum of most particulate matter in the raw wastewater, its digestion is subject to risks originating from various toxic pollutants as heavy metals, lipids and toxic organic compounds adsorbed on sludge particles which may enter the digesters at varying contents. Any difference is possible between parallel reactors under the same feeding conditions. pH at an alkali level is a natural outcome of PS digestion as ammonia nitrogen produced from the proteinaceous matter hydrolysis is an alkali compound and generates hydroxide alkalinity after conversion to its acidic ammonium form in the process.

In multiple substrate PS digestion, reduced biogas production indicated an inhibited methanogenesis which was evidenced by very low pH at 5.2-5.6 and high VFA concentration at 2926-4970 mg CaCO₃/L. Bicarbonate as the buffering power of the process was consumed significantly and dropped to 1/3-1/4th of the values of the PS digestion.

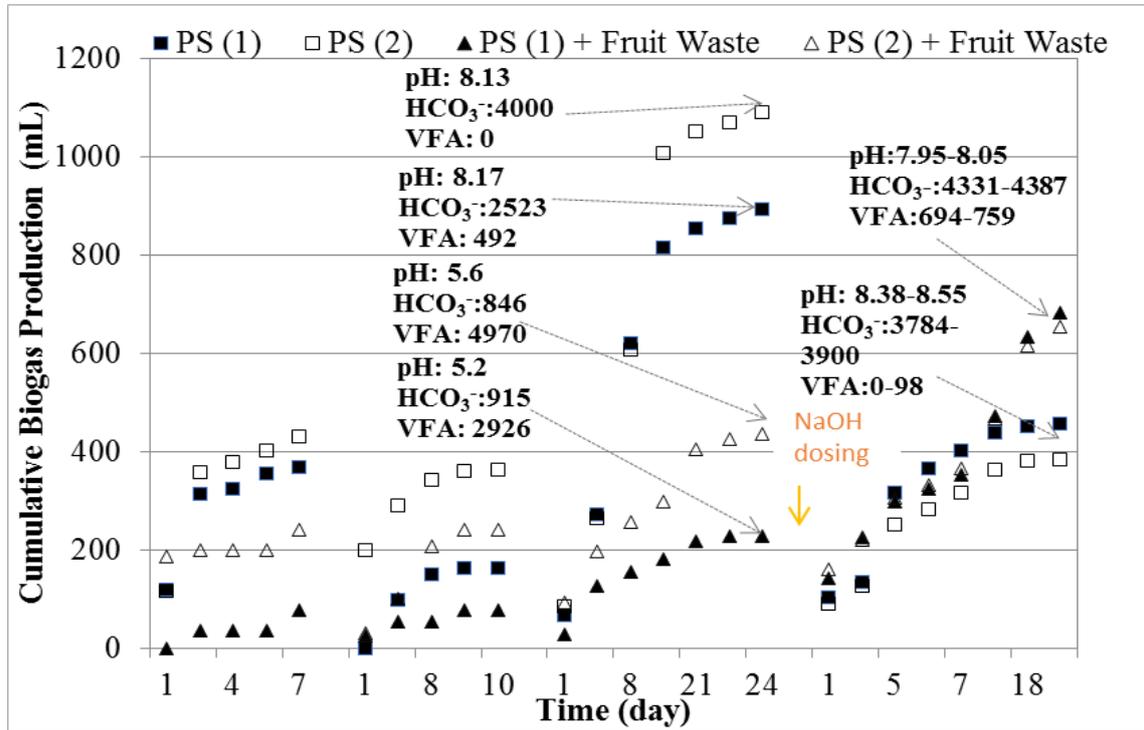


Figure 2. Cumulative biogas production and pH/HCO₃⁻/VFA in PS digestion with and without fruit waste (HCO₃⁻/VFA concentrations in mg CaCO₃/L)

A noticeable point was that higher VFA did not produce a worse effect on biogas production in fruit waste fed digesters. pH at 5.2 was the key parameter for the low process performance where pH levels below 5.5 is detrimental on methanogens and can't be tolerated for more than 24 hrs in continuously fed reactors (Speece, 1996). VFA and long chain fatty acids (LCFA)(produced as intermediate products from degradation of lipids and further β -oxidation by anaerobic hydrolytic and acidogenic bacteria, respectively) are in their toxic acid forms at this level. NaOH was added at 0.03 M to raise the pH and supply extra buffering power to multiple substrate fed reactors and new feeding set was started. Hydroxide salts are the most suitable alkali sources in case of intensive acidification because they generate double buffering effect on the system as they convert carbonic acid to bicarbonate and organic acids to their anionic forms that contribute to the alkalinity of the system.

Multiple substrate PS digesters' performances increased in the 4. feeding as they produced biogas at 1.5 time compared to PS digestion where pH occurred at 7.95-8.05 and VFA concentrations were at 1/4-1/7 compared to previous feeding indicating higher conversion to methane. pH control was highly effective on the process, especially on methanogenesis in digesting fruit waste. Higher bicarbonate level in multiple substrate PS digestion proved the efficiency of the alkali source as carbonic acid produced intensely in fruit waste digestion was converted to bicarbonate alkalinity and VFA were kept at their salt form (contributing to alkalinity).

Drop in biogas level in PS digestion was correlated to raw sludge change. The biogas yield obtained in PS digestion was 1.16-1.38, 0.99-1.21 and 0.76-0.90 L/g VS_{fed} in the 1-2., 3. and 4. feedings, respectively. The gradual decrease of the yield indicated also some inhibitory effect's accumulation in the reactors originating from PS or fruit waste content and/or degradation. High pH level at 8.33-8.55 is prone to increase the concentration of toxic free ammonia (FA) in the reactor which is a common problem in the digestion of protein-rich wastes (Speece, 1996; Astals *et al.*, 2014). The synergistic effect of carbohydrate matter in the balancing of pH came out as faster kinetics and improved process performance (Astals *et al.*, 2014). Fruit waste exhibited a potential to overcome possible FA toxicity as a co-substrate to PS digestion but necessitated alkali addition which could be eliminated at lower loading rates in the present study.

The process performance in SS digestion proceeded at a more stable level with biogas yield gradually decreasing from 1.3-1.6 down to 0.5-0.9 L/g VS_{added} in subsequent feedings as raw sludge changed (Fig. 3). pH proceeded at a stable range of 8.5-8.7 and some degree of VFA accumulation at 541-576 mg CaCO₃/L occurred.

In the case of multiple substrate SS digestion, biogas production was augmented up to 2.06, 1.9 and 1.16 times (compared to SS digestion alone) where the gradual decrease was caused by the intense acidification producing a final pH at 5.2-5.3 and VFA at 1812-2000 mg CaCO₃/L. The different pattern compared to multiple PS digestion was that lower pH levels were experienced with lower VFA values at similar methanogenic activity. After NaOH dosing, biogas production jumped to values 4 times the SS digestion with an alkali pH (8.16-8.25) and lower VFA compared to multiple PS digestion.

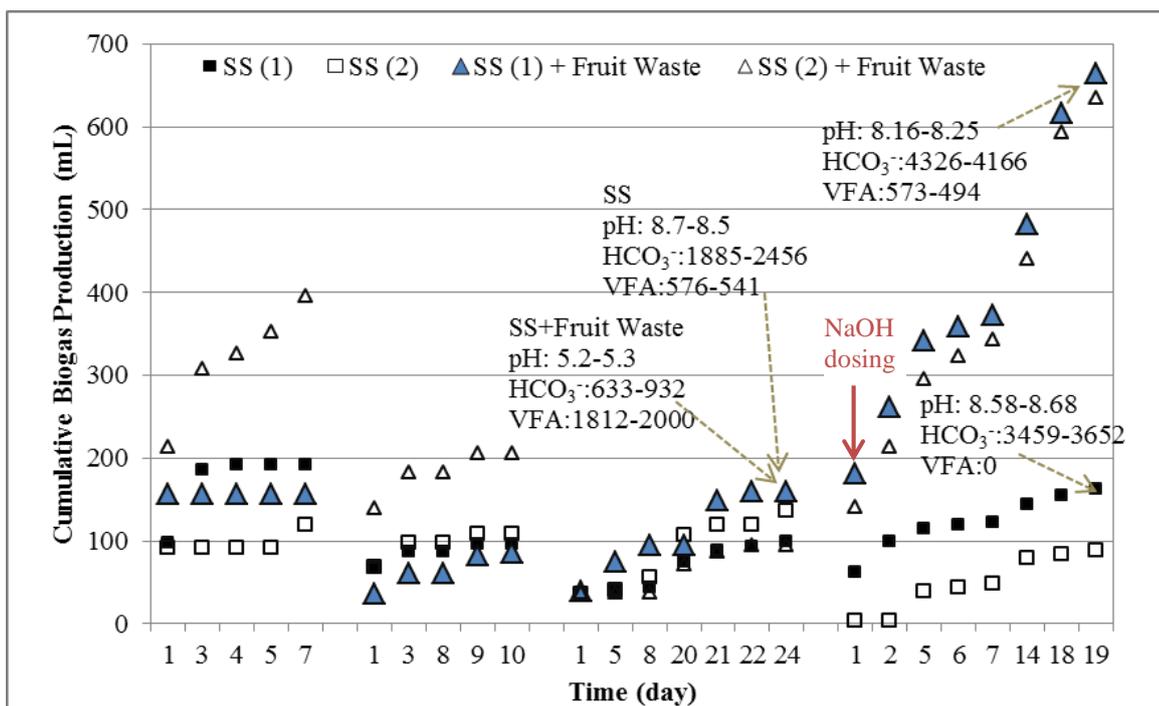


Figure 3. Cumulative biogas production and pH/HCO₃⁻/VFA in SS digestion with and without fruit waste (HCO₃⁻/VFA concentrations in mg CaCO₃/L)

4. CONCLUSIONS

Fruit waste loading applied at equal volume to PS and SS in the sequential lab-scale batch study showed that even this high loading proved feasible under optimum pH control with a suitable alkali source and high VFA concentration is tolerable and can be reduced effectively by conversion to methane gas. pH was determined as the key control parameter of the process.

Co-digestion resulted in 55% ((+227-271 mL) and 400% ((+501-546 mL) increase in biogas production compared to solely PS and SS stabilization. Fruit waste induced a higher degree of VFA accumulation with lower surplus biogas in PS co-digestion and proved its potential and promoting effect as a co-substrate at a significantly higher degree in SS digestion.

Anaerobic sludge digesters are fed at high sludge flow rates and any chemical addition may add considerably to the operational costs. The need for alkali addition can be eliminated via optimization of organic loading rate in case of fruit wastes and its high carbohydrate content will balance high pH range produced in sewage sludge and protein-rich wastewater digestion. Continuous feeding studies with a low-to-moderate mixing is the tool to obtain the optimum flow/load rate of multiple substrate digestion feasible for augmenting energy yield in municipal WWTPs.

Further research on increased biodegradability of sewage sludge fractions with different organic wastes' feeding will also show the high potential of separate PS and SS digestion at full scale towards the amelioration of the sludge line efficiency in municipal WWTPs. Co-digestion can also improve the final stabilized sludge's quality such as nutrient content for soil applications.

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