

Viscosity and Total Organic Carbon Removal in High-Solid Anaerobic Digestion of Sewage Sludge

Ahmed M. Aboulfotoh^a, Hanan A. Fouad^b, Ahmed Y. Bakry^c, Rehab El-hefny^d

^a Associated Professor, Environmental Engineering Department, Faculty of Engineering, Zagazig University.

^b Professor, Environmental Engineering Department, Faculty of Engineering at Shubra, Benha University, Egypt.

^c Assistant Lecturer, Environmental Engineering Department, Faculty of Engineering at Shubra, Benha University, Egypt.

^d Egypt Associated Professor, Environmental Engineering Department, Faculty of Engineering at Shubra, Benha University, Egypt.

Abstract. A batch test was done to study the effect of anaerobic mono-digestion of sludge and pretreatment on the sludge properties like viscosity and total organic carbon removal. After the Biomethane potential test of the first and second sets ended, samples of anaerobically digested sludge were analyzed for TOC removal. Set one and set two ended after 22 days. Set 1 shows that the best TOC removal was caused for the concentration of 11% for the pretreated and blank reactors and it was 33.92% and 27.25% respectively. The minimum removal ratio occurred at 16% concentration for the pretreated and blank reactors. Set 2 shows that the best TOC removal was caused for the reactor at a pre-treatment temperature of 180°C and it was 35.83% and which was higher than the removal of the blank reactor which was 27.50%. The maximum biogas produced was 2475 ml at 11% TS with an improvement of 55.56% and in terms of TOC removed, it was 189.19 mLbiogas/gTOCremoved. In set 2, The maximum Biogas produced was 3645 ml at 11% TS with an improvement of 60.57% and pretreatment temperature of 180°C and in terms of TOC removed, it was 169.53 mLbiogas/gTOCremoved. Sludge viscosity is enhanced after thermal pretreatment and improved the mixing of the high solid sludge.

Keywords: Digestion; Anaerobic digestion; Sewage sludge, viscosity, Total Organic Carbon.

ABBREVIATIONS

| | | | |
|-----|----------------------------------|------------------|--------------------------------|
| VS | Volatile Solids | NH ₃ | Ammonia |
| TP | Thermal Pre-Treatment | H ₂ S | Hydrogen Sulfide |
| TS | Total Solids | CO ₂ | Carbon Dioxide |
| TOC | Total Organic Carbon | H | Hydrogen |
| COD | Chemical Oxygen Demand | HSAD | High-Solid Anaerobic Digestion |
| TS | Total Solids | AD | Anaerobic Digestion |
| CAD | Conventional Anaerobic Digestion | | |

1. INTRODUCTION

Biomass energy generation from sewage sludge is gaining popularity around the world since it eliminates contamination from untreated sludge while also reducing dependency on fossil fuels [1]. Treatment of sewage sludge before AD has the ability to improve AD efficiency. Thermal pretreatment of SS can improve digestibility in the digester while also improving methane generation [2]. Landfills, agricultural utilization, composting, burning, reuse as a building material, and anaerobic digestion are all common sewage sludge disposal methods. The utilization of anaerobic sludge digestion is of high relevance. However, this treatment is well-known and can result in a reduction in sludge volume while also producing methane, which can be considered a renewable power resource [3],[2],[4]. All of these control techniques have practical limitations in terms of human and environmental health [5]. Among the treatment options, anaerobic digestion is regarded as an effective, environmentally friendly, and cost-effective technique. AD reduces the total solids in sludge before delivering to the disposal facility, stabilizes the sludge, kills pathogens, and minimizes odor releases [2],[6]. The following are the main disadvantages of SS AD: (1) the sluggish hydrolysis of bacterial aggregation leads to poor reaction rates; (2) the generation of (H_2S) and volatile silicon components, which obstruct methane production and use; (3) Process sensitivity and lowering the resistance to inhibitor buildup (e.g., NH_3); Other drawbacks include a large buffer needed for pH adjustment, ineffective treatment of mixed waste, and higher heavy metal concentrations in the ADS [6].

The fundamental steps of AD include hydrolysis (solubilization), acidogenesis, acetogenesis (produce acetic acid), and methanogenesis [7]. The rate-limiting phase here is hydrolysis, in which bacteria' extracellular enzymes break down complex organic compounds into smaller, smaller molecules [8]. It is useful to use several pretreatment technologies in this case, such as chemical, mechanical, thermal, or biological to the substrates to accelerate the rate of solubilization, dissolve organic molecules for increasing their degradability, increase methane production, and to tribute to the stability of AD [9],[10],[11],[12],[13],[14]. The use of thermal pretreatment to accelerate AD hydrolysis [2],[15],[16]. Pretreatment at high temperatures disinfects the sludge and degrades the extracellular polymeric substances (EPS) inside as well as on the face of the flocs, dissolving the floc structure and enhancing the bioavailability of the materials that make up the cells as well as SS dewaterability. Furthermore, cell damage caused by pressure variations during the thermal process may lead to an improvement in the rate of degradation [17],[18]. For AD improvement, low-temperature pretreatment uses temperatures below 100 °C. Thermophilic bacteria are activated, organic compounds are hydrolyzed, and the degradation rate is improved [19],[16]. Low-temperature pretreatment at 70 °C can also be used to kill pathogens in sludge [20]. The optimal parameters for low-temperature

thermal pretreatment of urban wastewater sludge were reported by Nazari et al. [16], according to their findings, the best temperature, time, and pH for pretreatment were 80°C, 5 hours, and pH 10 correspondingly. Soluble COD improved to 19 % under these conditions, but VS declined to 27.7 % [16].

Pretreatment at high temperatures usually causes physical degradation and solubilization of organic material [21],[22]. Thermal processing at temperatures ranging from 125 to 175 °C made WAS biodegradable quickly [23]. Thermal pretreatment causes the cell wall and membrane to separate, making proteins more accessible to degradation [24]. Liao stated that thermal pretreatment for 30 minutes at 70°C is recommended. The accumulative methane generation can be improved by more than 10%, and the digestion period can be greatly reduced, due to the rapid hydrolysis and lower viscosity of thermal degradation sludge. According to Bougrier, results obtained at 190°C were superior to those obtained at 135°C. COD removal increased from 52 % to 64 % after a thermal pretreatment at 190 °C. Thermal pretreatment has been proposed as a viable strategy for speeding up hydrolysis during conventional AD (CAD) of low-solid sludge, reducing digestion duration and increasing methane production [25],[26]. Because CAD and high-solid anaerobic digestion (HSAD) are founded on the same fundamental metabolic mechanisms, applying heat pretreatment to HSAD makes sense. Thermal pre-treatment can improve the hydrolysis of sludge particles and supramolecular organic compounds while also lowering the viscosity of high-solids sludge, according to theory. In fact, as the temperature of the sludge rises, it becomes more liquid [27],[28].

In this research, Viscosity, Durability, pH, and total organic carbon (TOC) after pretreatment processes and Anaerobic Mono-Digestion of HSAD of sludge were investigated. The study was two sets. The first set was studying the effect of different concentrations at a pretreatment temperature of 180°C for thirty minutes. The second set was studying the effect of the different temperatures of 100, 120, 140, 160, and 180°C for a certain concentration of 11%.

2. MATERIALS

The sludge utilized in the batch models was a drying sludge (raw and digested) from El-Berka and El-Gabal El-Asfar wastewater treatment plants located in Cairo, Egypt as shown in table 1. In order to study the viscosity and total organic carbon of high solid digestion of sewage sludge, a series of experiments were performed at different temperatures and different solid concentrations as shown in table 2.

TABLE 1. Characteristics of inoculum sludge and raw sewage sludge.

| Parameter | | COD | TOC | TS | VS | VS/TS | pH |
|-----------|--|------------|--------------|-----------|-------------|-------|------|
| Unit | | g/L | g/L | g/L | g/L | % | — |
| Value | SS | 150 ± 4.1 | 60 ± 1.5 | 160 ± 2.5 | 101 ± 1.5 | 63.12 | 6.12 |
| | inoculum sludge (From digester) | ----- - | ----- --- | 30 ± 0.5 | 22 ± 0.5 | 73.3 | 8 |
| | inoculum sludge (From Belt press filter) | ----- - | ----- --- | 210 ± 2.5 | 109.2 ± 1.5 | 52 | 6.5 |

2.1 BMP Test

The BMP tests were two sets. The first set was studying the effect of different concentrations of TS 10,11,12,14, and 16 % at a pretreatment temperature of 180°C for thirty minutes. The second set was studying the effect of the different temperatures of 100, 120, 140, 160, and 180°C for a certain concentration of 11%. The pre-treatment duration was thirty minutes. The digestion process was done at mesophilic temperature (35°C). All reactors were replicated. The used reactors were glass bottles of volume one liter, and the working volume was 0.50 liter. The biogas generated was measured by using the water displacement method. After the batch ended, samples were analyzed for pH and TOC removal. Table 2 is showing the stages of the BMP batch.

TABLE 2. Operating settings for BMP tests in the lab

| | Temperature | Ts | Pre-treatment Temperature | Time of pre-treatment |
|---------------------|-------------|----------------------|----------------------------|-----------------------|
| 1 st Set | 35± 2 °C | 10,11,12,14 and 16 % | 180 °C | 30 min |
| 2 nd Set | 35± 2 °C | 11% | 100,120,140,160 and 180 °C | 30 min |

2.2 Lab analysis

TOC and pH were measured at the beginning and end of the batch experiments. Traditional procedures were used to measure the digested sludge samples. The pH readings were measured using a pH meter. During the experiment, the amount of biogas produced was measured by using the water displaced method.

3. RESULTS

The primary objective of this research is to determine the viscosity, durability, pH, and total organic carbon (TOC) after pretreatment processes and anaerobic mono-digestion of HSAD of sludge.

3.1 Viscosity and Dewaterability Improvement

Viscosity, and durability after pretreatment processes of HSAD of sludge were found to be impacted by TP. Sludge viscosity was impacted by TP. Sludge left untreated was a pseudo-plastic fluid. The viscosity of sludge affects its fluid characteristics, which influences stirring performance. In the HSAD of sludge, a substantial energy input for stirring is required to guarantee sufficient mixing. The energy required for stirring would be reduced and the mass transfer would be improved if the viscosity of high-solids sludge could be reduced by TP. Extracellular polymeric substances (EPS) in the sludge surface layer, resulting in high frictional resistance and hence an increased viscosity. Some EPS, like carbohydrates, proteins, and lipids, were separated, liquefied, and even decomposed after TP [6], disrupting the bonds between sludge and lowering apparent viscosity. Farno [28] reported a similar statement. Because of the irreversible degradation of proteins, carbohydrates, and lipids, the impact of TP on sludge viscosity has traditionally been claimed to be irreversible [27],[28].

3.2 Effect of Anaerobic Digestion on pH value

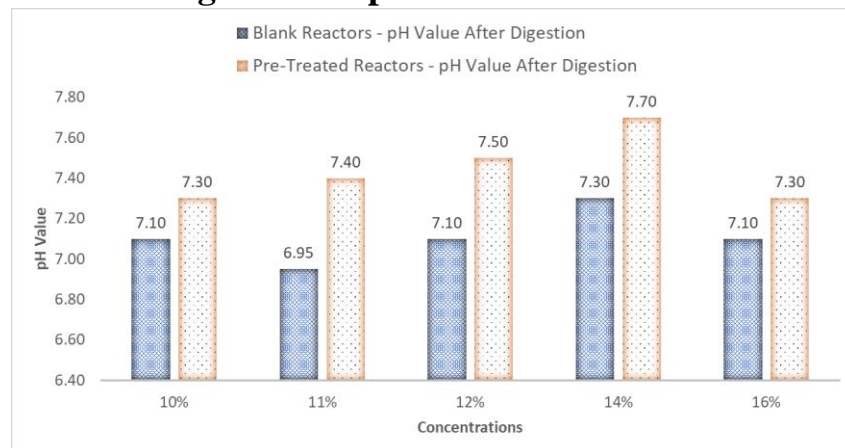


FIGURE 1. pH Values after the batch tests (1st Set)

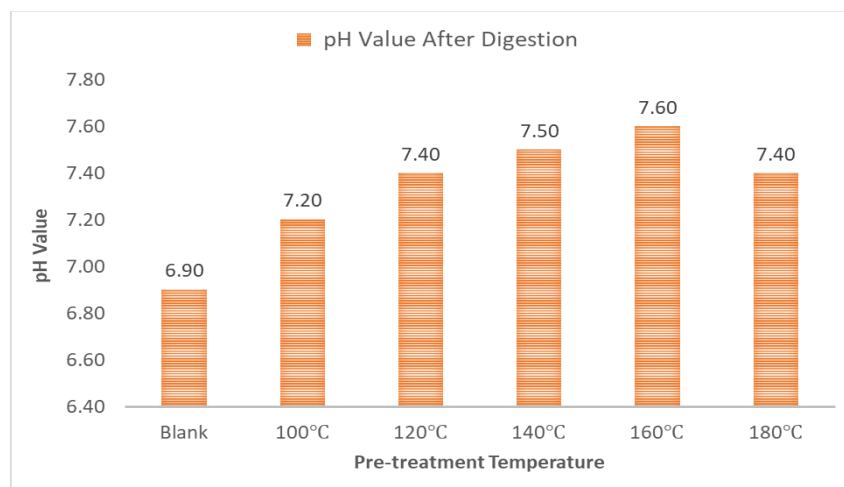


FIGURE 2. pH Values after the batch tests (2nd Set)

pH values increased in the two stages first increased (from 6.12 to 7.5 or 7.6 at 160 °C) and then decreased with a temperature of treatment (to 7.4 at 180 °C). Protein adsorption or acidic chemical evaporation could cause the pH value to rise [28]. The reduction in pH could thus be linked to macromolecule breakdown into acidic chemicals. Because acidogenesis also works at pH near neutrality, and methanogenesis is generally the rate-limiting step in an anaerobic one-step treatment process, the pH should be kept close to neutral. To achieve effective operation and control of the anaerobic digestion, you must control pH and the elements that cause or resist pH change. The compounds that have a strong buffering ability (alkalinity) in the neutral region around 7 are NH_3 , H_2S , $(\text{HCO}_3)_3$, and dihydrogen phosphate. The presence and concentration of buffering substances depend on the structure of the sludge and TOL. Generally, the carbonate balance controls the alkalinity required to control the pH [8]. sludge containing a high amount of nitrogen that is organically bound could create high alkalinity through ammonia production. Because the sulphide system is usually available at trace levels compared to carbonate, its contribution to buffering is normally minimal.

3.3 Effect of Anaerobic Digestion on TOC Removal

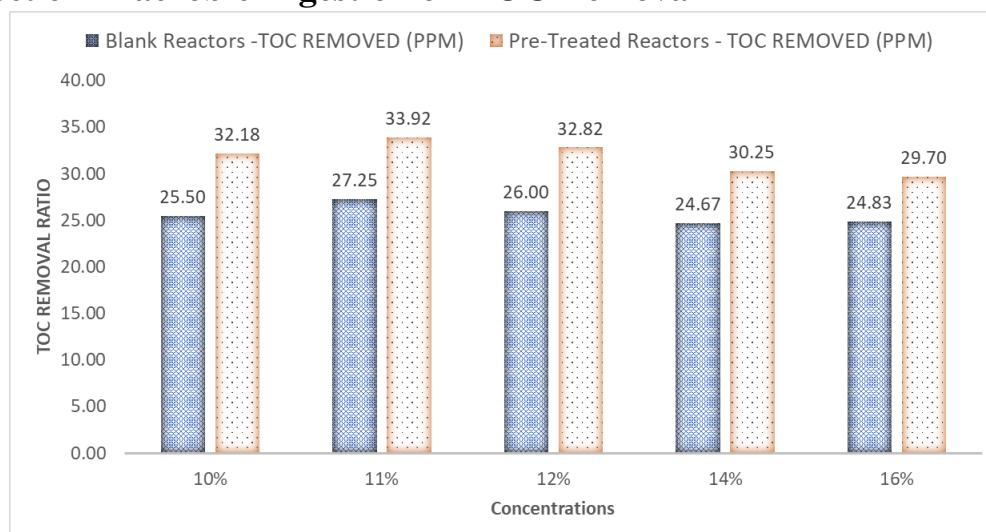


FIGURE 1. TOC Removal Ratio (1st Set)

After the Biomethane potential test of the first and second sets ended, samples of anaerobically digested sludge were analyzed for TOC removal. Set one and set two ended after 22 days.

FIGURE 1 shows the TOC removal ratio of set 1. It shows that the best TOC removal was caused for the concentration of 11% for the pretreated and blank reactors and it was 33.92% and 27.25% respectively. The minimum removal ratio occurred at 16% concentration for the pretreated and blank reactors. **FIGURE 2** shows the TOC removal for set 2. It shows that the TOC removal increases with TP temperature increasing. The best TOC removal was caused for the reactor at

a pre-treatment temperature of 180°C and it was 35.83% and which was higher than the removal of the blank reactor which was 27.50%.

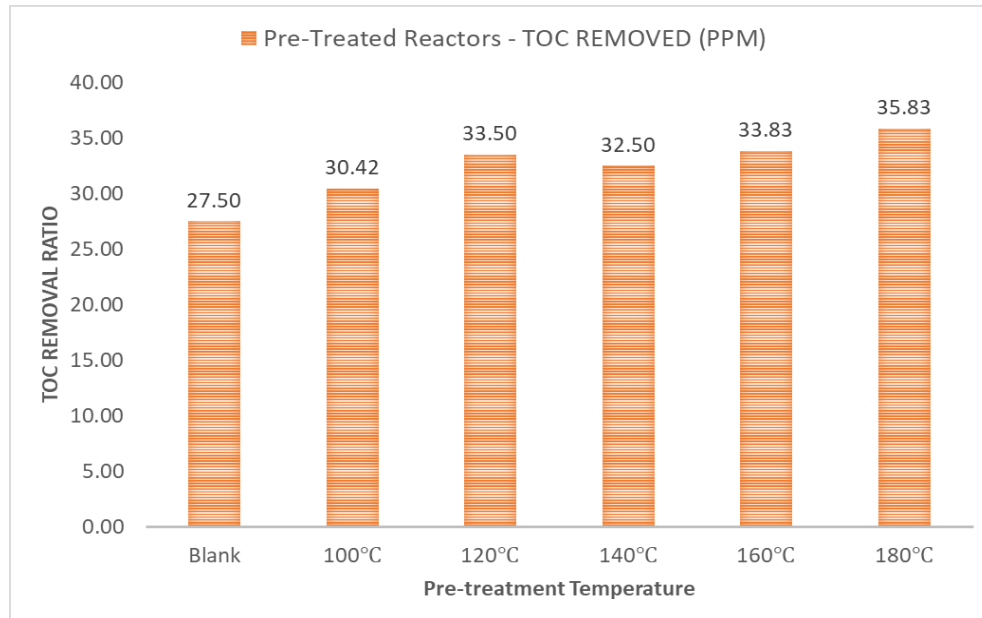


FIGURE 2. TOC Removal Ratio tests (2nd Set)

Table 3. The effect of thermal pre-treatments on sewage sludge anaerobic digestion with high solids (1st set)

| Concentrations | Blank Reactors - Gas Production mL | Pre-Treated Reactors - Gas Production mL | Improvement in gas production % | Blank Reactors- Biogas Yield (mLbiogas/gTO Cremoved) | Blank Reactors- Biogas Yield (mLbiogas/gTO Cremoved) |
|----------------|------------------------------------|--|---------------------------------|--|--|
| 10% | 2440 | 3285 | 34.63 | 159.48 | 170.16 |
| 11% | 2475 | 3850 | 55.56 | 151.38 | 189.19 |
| 12% | 2460 | 3150 | 28.05 | 157.69 | 159.98 |
| 14% | 1575 | 2025 | 28.57 | 106.42 | 111.57 |
| 16% | 1065 | 1370 | 28.64 | 71.48 | 76.88 |

In set 1, It was reported that the gas generation of the reactor of 11% SS was the optimum reactor, and the generation of the biogas was enhanced by (28-55 %) because of the thermal treatment. In set 2, the generation of gas for the various pretreatment temperatures is maintained in Table 4. For the blank reactor, the gas volume was 2270 mL. The reactors with thermal pretreatments at 100, 120, 140, 160, and 180 °C led to an enhancement in the generation of biogas by 20.7, 30.2, 45.4, 50.7, and 60.6 %, respectively. The results achieved are considered within the ranges acquired in the sludge anaerobic digestion which was (30 to 50) % [19]. The highest TOC removal occurred in the reactor of the highest biogas

generation which was the reactor with the concentration of 11% TS as shown in Table 3. The maximum Biogas produced was 2475 ml at 11% TS with an improvement of 55.56% and in terms of TOC removed, it was 189.19 mLbiogas/gTOC_{removed}. In set 2, The maximum biogas produced was 3645 ml at 11% TS with an improvement of 60.57% and pretreatment temperature of 180°C and in terms of TOC removed, it was 169.53 mLbiogas/gTOC_{removed} as shown in Table 4.

Table 4. The effect of thermal pre-treatments on sewage sludge anaerobic digestion with high solids (2nd set)

| Temperature °C | Gas Production mL | Improvement in gas production | Biogas Yield (mLbiogas/gTOC _{removed}) |
|-------------------|----------------------|----------------------------------|---|
| Blank | 2270 | - | 137.58 |
| 100 °C | 2740 | 20.70% | 150.14 |
| 120 °C | 2955 | 30.18% | 147.01 |
| 140 °C | 3375 | 48.68% | 173.08 |
| 160 °C | 3420 | 50.66% | 168.47 |
| 180 °C | 3645 | 60.57% | 169.53 |

4. CONCLUSIONS

Total solids content has a significant impact on overall digesting efficiency. Pumping and mixing of the sludge become difficult at greater TS content, However, TS content can be increased by up to 25% to (1) reduce storage space within the Wastewater treatment facility and (2) lower transportation costs. Heat applied during thermal treatment reduced viscosity and makes mixing easier. In this research, high solid sewage sludge was thermally pretreated at 100, 120, 140, 160, and 180 °C. The following conclusions can be drawn from the experiment results:

- Sludge viscosity could be reduced by thermal pretreatment.
- The pH should be kept close to neutral to achieve effective operation and control of the anaerobic digestion.
- The best TOC removal was caused for the reactor at a pre-treatment temperature of 180°C and it was 35.83%.
- The maximum Biogas produced was 3645 ml at 11% TS with an improvement of 60.57% and a pretreatment temperature of 180°C.

REFERENCES

1. Ratanatamskul C, Onnum G, Yamamoto K. A prototype single-stage anaerobic digester for co-digestion of food waste and sewage sludge from

- high-rise building for on-site biogas production. *International Biodeterioration & Biodegradation*, 2014 ;95:176-80.
2. Zhen G, Lu X, Kato H, Zhao Y, Li YY. Overview of pretreatment strategies for enhancing sewage sludge disintegration and subsequent anaerobic digestion: Current advances, full-scale application and future perspectives. *Renewable and Sustainable Energy Reviews*, 2017 ;69:559-77.
 3. Hanum F, Yuan LC, Kamahara H, Aziz HA, Atsuta Y, Yamada T, Daimon H. Treatment of sewage sludge using anaerobic digestion in Malaysia: current state and challenges. *Frontiers in Energy Research*, 2019 ;7:19.
 4. Zhu X, Yuan W, Lang M, Zhen G, Zhang X, Lu X. Novel methods of sewage sludge utilization for photocatalytic degradation of tetracycline-containing wastewater. *Fuel*, 2019 ;252:148-56.
 5. Zheng X, Jiang Z, Ying Z, Song J, Chen W, Wang B. Role of feedstock properties and hydrothermal carbonization conditions on fuel properties of sewage sludge-derived hydrochar using multiple linear regression technique. *Fuel*, 2020 ;271:117609.
 6. Appels L, Baeyens J, Degève J, Dewil R. Principles and potential of the anaerobic digestion of waste-activated sludge. *Progress in energy and combustion science*, 2008 ;34(6):755-81.
 7. Wang P, Wang H, Qiu Y, Ren L, Jiang B. Microbial characteristics in anaerobic digestion process of food waste for methane production—A review. *Bioresource technology*, 2018 ;248:29-36.
 8. Deepanraj B, Sivasubramanian V, Jayaraj S. Effect of substrate pretreatment on biogas production through anaerobic digestion of food waste. *International Journal of Hydrogen Energy*, 2017 ;42(42):26522-8.
 9. Caroca E, Serrano A, Borja R, Jiménez A, Carvajal A, Braga AF, Rodríguez-Gutiérrez G, Fermoso FG. Influence of phenols and furans released during thermal pretreatment of olive mill solid waste on its anaerobic digestion. *Waste Management*, 2021 ;120:202-8.
 10. Carrere H, Antonopoulou G, Affes R, Passos F, Battimelli A, Lyberatos G, Ferrer I. Review of feedstock pretreatment strategies for improved anaerobic digestion: From lab-scale research to full-scale application. *Bioresource technology*, 2016 ;199:386-97.
 11. Liu J, Zhao M, Lv C, Yue P. The effect of microwave pretreatment on anaerobic co-digestion of sludge and food waste: Performance, kinetics and energy recovery. *Environmental Research*, 2020 ;189:109856.
 12. Veluchamy C, Kalamdhad AS. Influence of pretreatment techniques on anaerobic digestion of pulp and paper mill sludge: a review. *Bioresource technology*, 2017 ;245:1206-19.

- 13.Xu H, Li Y, Hua D, Zhao Y, Mu H, Chen H, Chen G. Enhancing the anaerobic digestion of corn stover by chemical pretreatment with the black liquor from the paper industry. *Bioresource technology*, 2020 ;306:123090.
- 14.Zhang J, Li W, Lee J, Loh KC, Dai Y, Tong YW. Enhancement of biogas production in anaerobic co-digestion of food waste and waste activated sludge by biological co-pretreatment. *Energy*, 2017 ;137:479-86.
- 15.Ariunbaatar J, Panico A, Esposito G, Pirozzi F, Lens PN. Pretreatment methods to enhance anaerobic digestion of organic solid waste. *Applied energy*, 2014 ;123:143-56.
- 16.Qi G, Meng W, Zha J, Zhang S, Yu S, Liu J, Ren L. A novel insight into the influence of thermal pretreatment temperature on the anaerobic digestion performance of floatable oil-recovered food waste: Intrinsic transformation of materials and microbial response. *Bioresource Technology*, 2019 ;293:122021.
- 17.Senés-Guerrero C, Colón-Contreras FA, Reynoso-Lobo JF, Tinoco-Pérez B, Siller-Cepeda JH, Pacheco A. Biogas-producing microbial composition of an anaerobic digester and associated bovine residues. *MicrobiologyOpen*, 2019 ;8(9):e00854.
- 18.Cristancho DE, Arellano AV. Study of the operational conditions for anaerobic digestion of urban solid wastes. *Waste management*, 2006 ;26(5):546-56.
- 19.Liao X, Li H, Zhang Y, Liu C, Chen Q. Accelerated high-solids anaerobic digestion of sewage sludge using low-temperature thermal pretreatment. *International Biodeterioration & Biodegradation*, 2016 ;106:141-9.
- 20.De los Cobos-Vasconcelos D, Villalba-Pastrana ME, Noyola A. Effective pathogen removal by low temperature thermal pre-treatment and anaerobic digestion for Class A biosolids production from sewage sludge. *Journal of Water, Sanitation and Hygiene for Development*. 2015 ;5(1):56-63.
- 21.Iglesias-Iglesias R, Campanaro S, Treu L, Kennes C, Veiga MC. Valorization of sewage sludge for volatile fatty acids production and role of microbiome on acidogenic fermentation. *Bioresource Technology*, 2019 ;291:121817.
- 22.Mahdy A, Wandera SM, Aka B, Qiao W, Dong R. Biostimulation of sewage sludge solubilization and methanization by hyper-thermophilic pre-hydrolysis stage and the shifts of microbial structure profiles. *Science of The Total Environment*, 2020 ;699:134373.
- 23.Jo HB, Parker W, Kianmehr P. Comparison of the impacts of thermal pretreatment on waste activated sludge using aerobic and anaerobic digestion. *Water Science and Technology*, 2018 ;78(8):1772-81.
- 24.Aboulfoth AM, El Gohary EH, El Monayeri OD. Effect of thermal pretreatment on the solubilization of organic matters in a mixture of

- primary and waste activated sludge. *Journal of Urban and Environmental Engineering*, 2015 ;9(1):82-8.
- 25.Kim D, Lee K, Park KY. Enhancement of biogas production from anaerobic digestion of waste activated sludge by hydrothermal pre-treatment. *International Biodeterioration & Biodegradation*, 2015 ;101:42-6.
- 26.Liu Y, Li X, Kang X. Effect of volume ratio on anaerobic co-digestion of thermal hydrolysis of food waste with activated sludge. *International Biodeterioration & Biodegradation*, 2015 ;102:154-8.
- 27.Baudez JC, Gupta RK, Eshtiaghi N, Slatter P. The viscoelastic behaviour of raw and anaerobic digested sludge: strong similarities with soft-glassy materials. *Water Research*, 2013 ;47(1):173-80.
- 28.Farno E, Baudez JC, Parthasarathy R, Eshtiaghi N. Rheological characterisation of thermally-treated anaerobic digested sludge: Impact of temperature and thermal history. *Water research*, 2014 ;56:156-61.