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Published by the Faculty of Engineering, Khon Kaen University, Thailand

Improving anaerobic digestion of various sludge types through alkaline pretreatment

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Received 30 March 2024
Revised 20 September 2024
Accepted 30 September 2024

Abstract

This study investigates the impact of alkaline pretreatment on the anaerobic digestion of waste activated sludge (WAS) with varying types and total solids (TS) concentrations. Using NaOH, we optimized pretreatment conditions with a pH of 8.5 and a contact time of 2 hours. Our key findings demonstrate that this pretreatment significantly enhances sludge solubilization, evidenced by a notable increase in soluble chemical oxygen demand (SCOD). Specifically, SCOD increased by 57.5% for domestic wastewater (Plant A) and by 12.1% for industrial wastewater (Plant B) at a 0.5% TS concentration. Higher TS concentrations showed similar trends, with improved solubilization and higher methane production rates. Additionally, pretreatment elevated ammonia nitrogen (NH_3-N) and phosphorus concentrations, with greater releases at higher TS levels. These improvements resulted in enhanced anaerobic digestibility, higher total solids (TS) and volatile solids (VS) destruction, and increased specific methane production. The study underscores the effectiveness of alkaline pretreatment in optimizing anaerobic digestion processes, offering practical implications for wastewater treatment efficiency.

Keywords: Waste Activated Sludge (WAS), NaOH pretreatment, Solubilization, Anaerobic sludge digestion, Biogas productivity

1. Introduction

The activated sludge process stands as the most prevalent biological method for treating domestic wastewater. Throughout aerobic biological treatment, organic pollutants undergo mineralization into carbon dioxide and water, resulting in the production of excess bacterial biomass commonly referred to as waste activated sludge (WAS). The management, handling, and disposal of this surplus sludge account for up to 60% of the overall operating costs of wastewater treatment plants [1, 2]. The substantial volume of waste sludge, comprising refractory and non-biodegradable cellulose compounds, poses challenges for sludge disposal [3, 4].

Anaerobic digestion serves as a conventional sludge treatment process utilized to stabilize organic matter. Key features of the process include mass reduction, methane production, and improved dewatering properties of the treated sludge. However, a drawback of anaerobic digestion is its slow degradation rate, necessitating retention times in conventional digesters of approximately 20-30 days. Particularly, the organic fraction of waste activated sludge (WAS) with low concentrations often results in anaerobic digestion failures, as solubilizing particulate matter in sludge with low organic content proves more challenging than in sludge with higher organic content. The decrease in organic matter concentration in sludge observed in many countries is attributed to the implementation of Biological Nutrient Removal (BNR) processes [5]. Consequently, biological sludge requires pretreatment to enhance digestibility. The objective of such pretreatment is to rupture the cell wall and membrane, facilitating the release of intra- and extracellular matter into the aqueous phase for subsequent degradation an operation referred to as sludge disintegration. These pretreatment methods encompass physical techniques (e.g., ball milling, ultrasonication) [6, 7], chemical methods (e.g., ozone, hydrogen peroxide, acid and base treatments) [6-10], thermal processes, and biological approaches (e.g., enzymatic hydrolysis) [6].

Among these methods alkaline pretreatment has emerged as a favored method for sludge pretreatment due to its simplicity, ease of operation, and high efficiency [11]. This process involves splitting complex polymers into smaller molecules, thereby increasing solubilization levels and enhancing the subsequent performance of anaerobic digestion [12-16]. Sodium hydroxide (NaOH) is commonly preferred as the reagent due to its reported higher solubilization efficiency compared to calcium hydroxide ($Ca(OH)_2$) [15]. Research by [17] demonstrated that alkaline pretreatment performed at pH 12 and 30 minutes of contact time using various alkaline agents at ambient temperature increased soluble chemical oxygen demand (COD) values by approximately 39.8%, 36.6%, 10.8%, and 15.3% for NaOH, KOH, $Mg(OH)_2$, and $Ca(OH)_2$, respectively. Similarly, Research by [9] reported an increase in the percentages of soluble COD to total COD (SCOD/TCOD) from 3.31% to 36.3% following alkaline pretreatment. However, research has also explored other pH levels and durations, indicating that the effectiveness of pretreatment can vary based on sludge composition and specific operational conditions. Sodium hydroxide at low concentration is essential for the methanogenic bacteria presumably because it is

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doi: 10.14456/easr.2024.63

important for the formation of ATP or oxidation of NADH. However, high concentration of sodium ion inhibits the activity of the microorganisms and interferes with their metabolism. The level of inhibition depends on the concentration found in the sludge. The optimal growth condition of hydrogenotrophic methanogens occurs at concentration of 350 mg Na^{+}/L. Moderate inhibition is at the concentrations between 3,500 and 5,500 mg/L, whereas the concentration about 8,800 mg/l is strongly inhibitory to methanogenic bacteria during mesophilic digestion [18, 19].

Despite these findings, there is limited information available on the mechanisms underlying the enhancement of solubilization and methane potential of excess sludge with different types through alkaline pretreatment, especially at lower pH levels and extended treatment durations. This study, we investigate the effects of alkaline pretreatment on anaerobic digestion using waste activated sludge (WAS) with varying compositions. We specifically explore the use of NaOH at a pH of 8.5 and a contact time of 2 hours, aiming to evaluate its impact on sludge solubilization and the subsequent performance of anaerobic digestion. These parameters were selected based on preliminary tests that indicated potential benefits in terms of enhanced solubilization at lower pH levels and longer contact times, which may be more practical for full-scale operations compared to higher pH levels. Additionally, variations in total solid concentration in WAS during alkaline pretreatment will be analysed. Furthermore, the impact of pretreatment on anaerobic digester performance will be assessed to evaluate its effects on solid reduction and biogas production.

2. Materials and methods

2.1 Waste activated sludge sample

Samples of waste activated sludge (WAS) were obtained from two different sources: domestic wastewater treatment (Plant A) and an industrial wastewater treatment plant (Plant B). At Plant A, the sludge was collected from the drying bed, while at Plant B, secondary sludge was obtained from the sedimentation tank. Following collection, the sludge samples were stored in a cold room at 4°C to maintain their integrity. Typically, WAS from secondary sedimentation tanks has a concentration ranging from 8,000 to 12,000 mg/l. To standardize the samples, the sludge was prepared by evaporation and blending. The concentration of WAS was varied between 0.5-2.0% of dry solids, and various parameters including soluble chemical oxygen demand (SCOD), total solids (TS), volatile solids (VS), and elemental composition were analyzed with the standard methods (American Public Health Association, 2017). The WAS samples were then subjected to alkaline pretreatment and biochemical methane potential (BMP) tests. The characteristics of the raw sludge are outlined in Table 1. The BOD/COD ratio values are approximately 0.10-0.12 for plant A and 0.10-0.16 for plant B, indicating a predominance of slowly biodegradable constituents. The majority of the WAS comprises particulate organic matter, as evidenced by the SCOD/COD ratio and TSS/TS ratios. Sludge from wastewater treatment facilities typically contains organic compounds with large molecular.

2.2 Alkaline pretreatment

The experiments were conducted by adjusting the pH to approximately 8.5 and maintaining a contact time of 2 hours under anoxic conditions at ambient temperature. Chemical pretreatment was carried out using NaOH 1 N at ambient temperature. The evaluation of sludge disintegration efficiency was based on the solubilization of chemical oxygen demand (COD) into the aqueous phase. Soluble chemical oxygen demand (SCOD) and total COD (TCOD) of the untreated sludge were analyzed before pretreatment, and the SCOD of the pretreated sludge was also analyzed. The increase in SCOD was calculated as a percentage increase compared to TCOD. A comparison of the percentage of SCOD before and after the run was performed to clarify the effectiveness of the pretreatments. Statistical analysis was performed using ANOVA to determine the significance of the differences between the treatments, with a confidence level of $p < 0.05$.

Table 1 Characteristic of Waste Activated Sludge from different sources

Parameter	Plant A			Plant B		
	0.5%TS	1.0%TS	2.0%TS	0.5%TS	1.0%TS	2.0%TS
Total COD (mg/l)	7214.90	13684.05	27623.70	5249.15	12604.59	21922.69
Soluble COD (mg/l)	173.02	303.00	514.93	233.98	442.61	571.05
Total Solid (mg/l)	4233	8760	18400	5773	8173	16720
Total volatile Solid (mg/l)	3707	7453	14340	3920	5313	12140
Suspended Solid (mg/l)	3850	8455	17075	4575	7845	15665
Total Dissolve Solid (mg/l)	300	800	1380	750	1470	3100
Total BOD (mg/l)	875	1552	2766	847	1722	2399
% of Soluble COD/Total COD	2.3980	2.2143	1.8641	4.4574	3.5115	2.6048
BOD/COD	0.1213	0.1134	0.1001	0.1614	0.1366	0.1094
VS/TS	0.876	0.851	0.779	0.679	0.650	0.726
TDS/TS	0.072	0.086	0.075	0.141	0.158	0.165
TSS/TS	0.928	0.914	0.925	0.859	0.842	0.835

2.3 Biochemical methane potential assay

The biochemical methane potential (BMP) test was conducted to assess the biodegradability of sludge before and after alkaline pretreatment under ambient temperature conditions in batch anaerobic digesters for approximately 30 days. During the BMP test, 120 ml serum bottles capped with butyl rubber stoppers were utilized. The total working volume of each bottle was 100 mL, with 70 mL designated for inoculum and 30 mL for WAS. Prior to purging with oxygen-free nitrogen gas to eliminate oxygen from the headspace, the serum bottles were filled with the respective volumes. Subsequently, the serum bottles were placed in an incubator until biogas production ceased. The volume of biogas produced daily was measured by inserting a needle attached to a syringe (10 and 25 mL).

Additionally, methane composition was analyzed using a Gas Chromatograph equipped with a packed column. Biogas production serves as a measure of the quantity of biogas generated from the WAS. Gas production is directly correlated biochemically to the amount of volatile solids destroyed and is expressed as the volume of gas per unit total solids added. Samples were collected using a graduated syringe with a volume of 0.1 mL and injected into the sampling column of the Gas Chromatograph for analysis.

3. Results and discussion

3.1 Impact of Alkaline Pretreatment on SCOD Solubilization

The SCOD before and after of different concentration of TS, and sources of WAS are summarized in Table 2. When the alkaline pretreatment was performed at TS of 0.5% and the pH was adjusted to 8.5 using NaOH, an increase of approximately 286 mg/L and 282 mg/L in SCOD was observed after 2 hours for Plant A and Plant B, respectively. With the same reaction conditions at the 2.0% of TS concentration about 655 and 818 mg/l of SCOD increase was observed. The solubilization of WAS at different total solids (TS) concentrations was examined. After alkaline pretreatment at a TS concentration of 0.5%, an increase from 2.40 to 3.79 was observed for plant A, and an increase from 4.46 to 5.00 for plant B, after a reaction time of 2 hours. Similarly, under the same reaction conditions but at a TS concentration of 1.0%, an increase from 2.21 to 2.92 was observed for plant A, and an increase from 3.51 to 5.08 for plant B. The comparison between Plant A and Plant B reveals notable differences in SCOD solubilization efficiency. Plant B consistently demonstrates higher SCOD solubilization percentages compared to Plant A across various total solids (TS) concentrations. This increased efficiency can be attributed to the higher BOD/COD ratio in the WAS from Plant B, which facilitates the transformation of organic particles into soluble forms more effectively. Consequently, the solubilization process is more pronounced in WAS from Plant B, leading to greater SCOD increases after alkaline pretreatment. Comparing different types of WAS, it was found that alkaline pretreatment led to an increase in SCOD solubilization for both Plant A and Plant B. Specifically, at a TS concentration of 0.5%, SCOD solubilization for Plant A improved from 2.40% to 3.79%, representing an increase of approximately 58%. For Plant B, SCOD solubilization improved from 4.46% to 5.00%, reflecting an increase of approximately 12%. These results indicate that while Plant B exhibited a higher overall SCOD solubilization percentage, Plant A experienced a more significant percentage increase under the same conditions. Statistical comparison using ANOVA revealed significant differences between the Raw Sludge and Alkaline Pre-treatment of excess sludge from WAS of Plant A and Plant B at different concentrations, with a 95% confidence level ($p < 0.05$). The Alkaline Pre-treatment condition demonstrated a higher %SCOD/COD compared to the non-pretreatment condition. This difference can be attributed to the higher BOD/COD ratio in the WAS from Plant B, which indicates a greater proportion of readily biodegradable organic matter. A higher BOD/COD ratio has been associated with more efficient breakdown of particulate matter into soluble forms, thereby enhancing the solubilization process [20]. Alkaline pretreatment can increase the levels of solubilization by splitting complex polymers into smaller molecules, more readily biodegradable molecules, thereby improving the performance of subsequent anaerobic digestion [14].

Table 2 The COD and SCOD of before and after alkaline pretreatment

Source	Condition		Parameter (mean ± S.D)		
			Total COD (mg/l)	**Soluble COD (mg/l)**	**%SCOD/COD**
WAS from Plant A	Raw Sludge	0.5%TS	7214.90 ± 728.18	173.02 ± 15.35	2.398
		1.0%TS	13684.05 ± 1479.20	303.00 ± 47.14	2.214
		2.0%TS	27623.70 ± 345.35	514.93 ± 26.51	1.864
	Alkaline Pre-treatment	0.5%TS	7538.10 ± 180.03	285.71 ± 12.57	3.790
		1.0%TS	14374.65 ± 460.11	420.17 ± 129.42	2.923
		2.0%TS	27166.95 ± 1474.95	655.02 ± 27.56	2.411
WAS from Plant B	Raw Sludge	0.5%TS	5249.159 ± 445.73	233.98 ± 38.28	4.457
		1.0%TS	12604.59 ± 570.01	442.61 ± 30.61	3.511
		2.0%TS	21922.69 ± 270.89	571.05 ± 45.51	2.605
	Alkaline Pre-treatment	0.5%TS	5635.90 ± 338.99	281.91 ± 59.26	5.002
		1.0%TS	13376.85 ± 120.21	679.55 ± 16.26	5.080
		2.0%TS	22775.60 ± 466.12	818.33 ± 91.36	3.593

The findings suggest that as the percentage of total solids (TS) concentration increases, the rate of solubilization decreases when the pH adjustment in alkaline pretreatment of WAS remains constant. Research by [13] conducted a study using NaOH for pretreating industrial WAS collected from southern Taiwan. They observed that as the NaOH content during pretreatment increased to 30 meq/l, the solubilization of WAS, or the SCOD value, increased with higher total suspended solids (TSS) concentration. [13] also suggested that considering the ratio of soluble chemical oxygen demand (SCOD) to total chemical oxygen demand (TCOD), a TSS concentration of 1% exhibited the highest SCOD solubilization compared to TSS concentrations of 0.5% and 2%. In this study, the optimal SCOD solubilization was observed at 0.5% of TS concentration for domestic WAS and 1.0% of TS concentration for industrial WAS. However, the achieved values were lower than those reported in other studies, possibly due to differences in contact time for alkaline pretreatment.

3.2 The impact of alkaline pretreatment on the solid fraction

Alkaline pretreatment has been observed to enhance the hydrolysis step in the anaerobic process, particularly affecting the solid fraction. The total dissolved solids (TDS) and suspended solids (SS) contents of the two source WAS before and after alkaline pretreatment are illustrated in Figures 1 and 2. In this study, solid content is defined in terms of fraction (%) and concentration (mg/l). The TDS ratio is commonly utilized to depict the transformation of particulate organic fractions into the soluble organic fraction, providing insight into the extent of hydrolysis. In our investigation, the TDS/TS ratios increased from 7.2%, 8.6%, and 7.5% to 35.9%, 21.5%, and 23.6% for Plant A at solid contents of approximately 0.5%, 1.0%, and 2.0% of TS, respectively. Similarly, for Plant B, these ratios increased from 14.1%, 15.8%, and 16.5% to 44.7%, 22.8%, and 25.1% for the corresponding TS concentrations. In our investigation, Plant A and Plant B exhibited distinct differences in their TDS/TS ratios across various total solids (TS) concentrations. For Plant A, the TDS/TS ratios showed a significant increase at all TS levels, indicating a notable change in the soluble components relative to the total solids. Conversely, Plant B also experienced an increase in TDS/TS ratios, but the ratios were higher at lower TS concentrations compared to Plant A, suggesting a higher initial solubility of total dissolved solids in the WAS from Plant B. These differences highlight that Plant B waste activated sludge has a greater proportion of soluble components relative to its total solids compared to Plant A. During alkaline pretreatment, the complex activated sludge floc structure is disrupted, releasing intracellular compounds into the soluble phase, thereby increasing TDS and decreasing SS. Previous studies have reported on the solubilization of WAS during pretreatment processes. For instance, the volatile dissolved solids (VDS) to volatile solids (VS) ratio for secondary sludge increased from 0.01 to 0.18 after thermal pretreatment (170°C/120 min) [7]. Similarly, the VDS/VS ratio of food waste was found to increase from 0.60 to 0.70 following thermal pretreatment at 121°C/120 min [17].

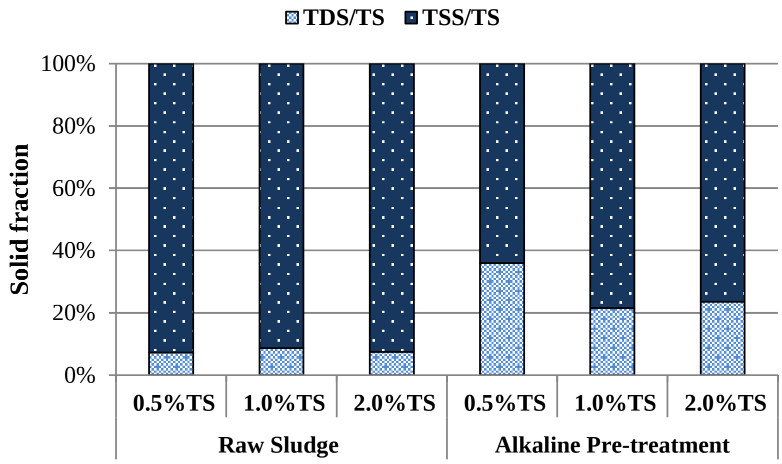


Figure 1 Solid fraction of before and after alkaline pretreatment on WAS of plant A

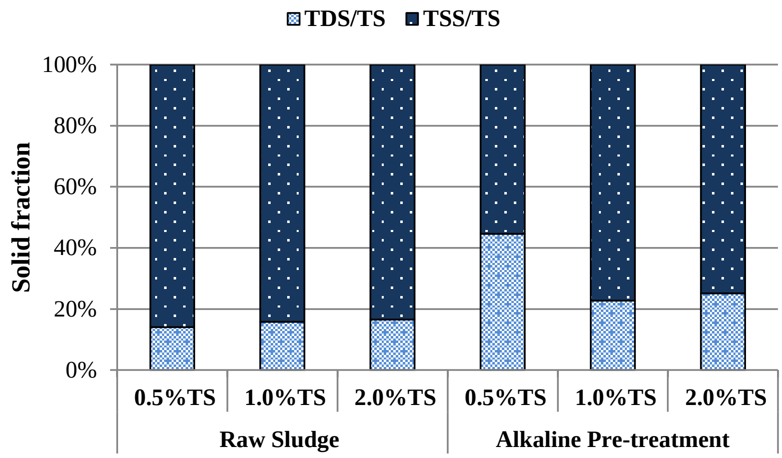


Figure 2 Solid fraction of before and after alkaline pretreatment on WAS of plant B

3.3 Effect of alkaline pretreatment on soluble ammonia nitrogen and phosphorus release

One of the minor objectives of adjusting the pH to approximately 8.5 during alkaline pretreatment is to render the supernatant of chemically pretreated WAS biodegradable, thus enabling its utilization as a carbon source for denitrification processes. It's important to note the release of nitrogen in the supernatant from chemically pretreated WAS, as this can lead to an increase in nitrogen loading for biological processes [21]. Alkaline pretreatment, particularly with NaOH, promotes the deamination of organic nitrogen compounds. This process breaks down proteins and other nitrogenous organic matter into ammonia and other simpler compounds. The high pH environment facilitates the hydrolysis of these compounds, leading to increased release of ammonia into the solution. Figure 3 presented the levels of ammonia nitrogen and phosphorus in the liquid phase before and after alkaline pretreatment. Ammonia nitrogen (NH_3-N) is also a parameter of interest during sludge disintegration. This study investigated the release of NH_3-N from WAS after alkaline pretreatment and found an increase in NH_3-N concentration post-alkaline treatment. For instance, at a TS concentration of 1.0%, NH_3-N concentration increased from 39.9 mg NH_3-N/l WAS to 43.9 mg NH_3-N/l WAS after alkaline treatment (adjusted to pH 8.5 using NaOH 1 N) for WAS from plant A. [3] reported similar findings regarding the release of total Kjeldahl nitrogen (TKN) and NH_3-N in the supernatant when municipal WAS was pretreated with NaOH under different reaction conditions. The release of TKN and NH_3-N was observed to increase with higher TSS concentrations, with the release at a pretreatment time of 1 hour contributing more than 73% and 20% of the total release, respectively.

The release of phosphorus within the sludge hydrolysate was also notably low. Initially, the concentration of soluble phosphorus at TS concentrations of 0.5%, 1.0%, and 2.0% was measured. After alkaline treatment (adjusted to pH 8.5 using NaOH 1 N), an increase in soluble phosphorus concentration was observed. For example, for WAS from plant A, the concentration increased from 33.9 to 97.9 mg PO_4^{3-}/l at a TS concentration of 2.0%. [12] demonstrated a similar trend, showing that after alkaline pretreatment (at 30 meq/l), soluble phosphorus concentration increased from 11.7 to 11.9 mg PO_4^{3-}/l at a TS concentration of 1.0%.

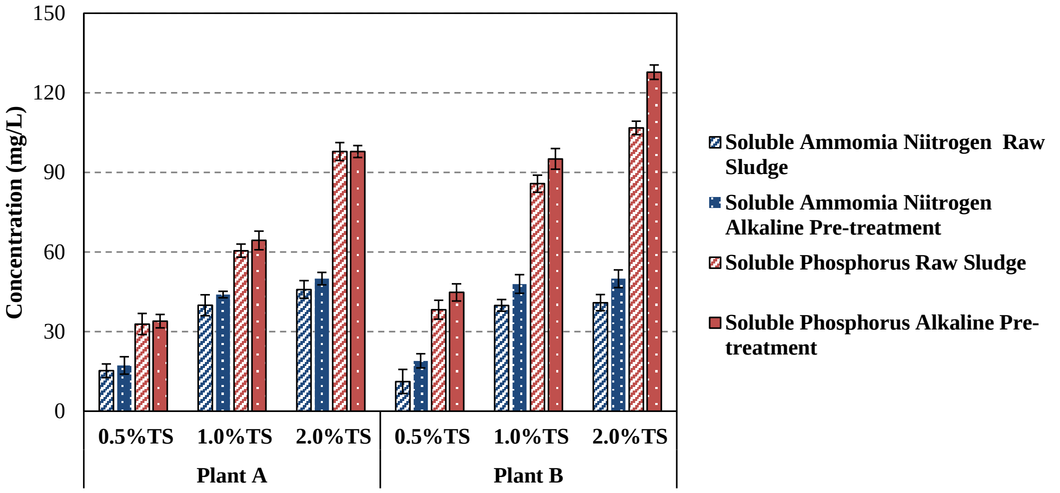
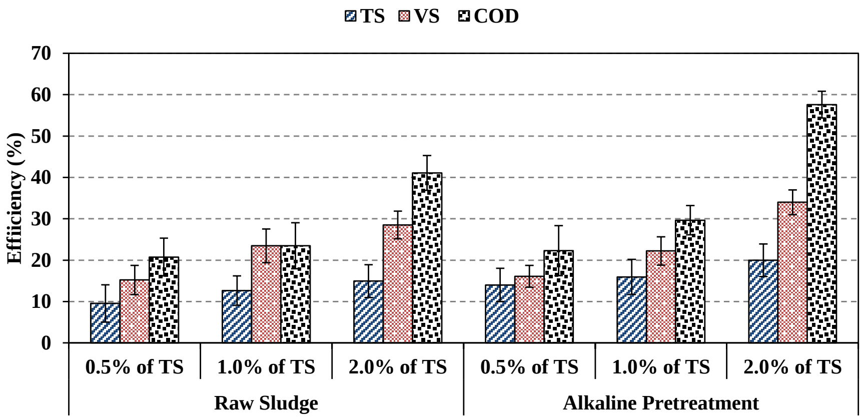
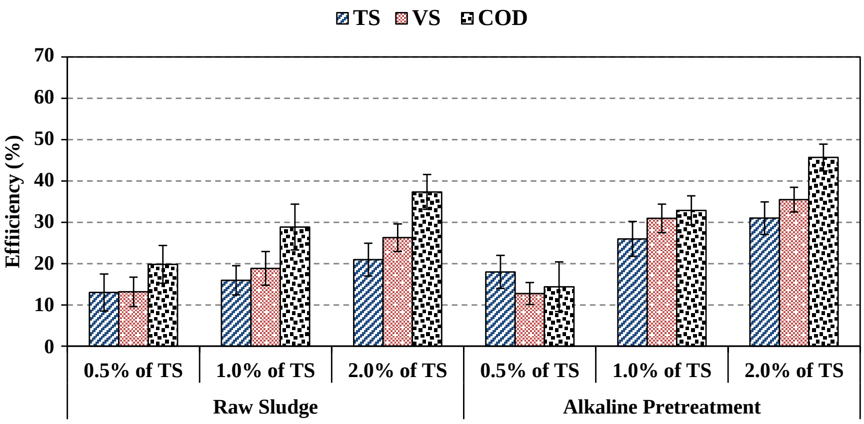


Figure 3 Concentrations of ammonia nitrogen and phosphorus in the soluble phase of the sludge



a) Plant A



b) Plant B

Figure 4 Summaries of efficiency in BMP test

3.4 Biochemical methane potential (BMP) test

The TS, VS, and COD contents before and after digestion, as observed during the experimental runs, are presented in Figure 4. This table illustrates the efficiency of TS, VS, and COD removal in sludge pretreated with different sources and concentrations of TS. Clearly, the removal efficiency of organic compounds increased when the WAS was enhanced by alkaline pretreatment. For WAS from plant A, the variation in TS content had an effect on the efficiency of TS, VS, and COD removal. At a TS concentration of 2.0% in the WAS, higher efficiency was observed compared to lower TS content. For instance, at 0.5% TS, approximately 9.54%, 15.21%, and 20.75% of TS, VS, and COD were removed, respectively. In contrast, at 2.0% TS, the efficiency increased to about 14.95%, 28.51%, and 41.07% for TS, VS, and COD removal, respectively. Alkaline pretreatment improved the characteristics of WAS, resulting in higher efficiency compared to non-pretreated samples. At a TS concentration of 2.0%, removal efficiencies were approximately 19.96%, 33.99%, and 57.60% for TS, VS, and COD, respectively. Similar results were observed for WAS from Plant B, where alkaline pretreatment exhibited higher efficiency compared to non-pretreated samples, with an increase in efficiency observed with higher solid content. For example, at 2.0% TS, non-pretreated samples removed approximately 20.96%, 26.26%, and 37.33% of TS, VS, and COD, respectively. After alkaline pretreatment, these removal efficiencies increased to 31.00%, 35.46%, and 45.67% for TS, VS, and COD, respectively. A study by [13] utilized municipal waste activated sludge (WAS) treated with NaOH to solubilize particulate organic matter and improve digestibility. VS removal efficiency for sludge treated with 40 meq/l of NaOH was reported to be as high as 41% over non-pretreated samples, with COD removal reaching 30% over the control.

Due to the alkaline pretreatment with NaOH addition, the performance of anaerobic digesters was assessed through BMP testing. The biochemical methane potential from pretreated WAS was determined based on BMP tests conducted over a period of 30 days. It was observed that BMP (showed in Figure 5), as indicated by total solid feed, increased after alkaline pretreatment. Specifically, alkaline pretreatments resulted in a 30.4% increase in specific gas production for Plant A, from 0.288 L CH_4/g of TS without pretreatment to 0.3755 L CH_4/g of TS with alkaline pretreatment at 0.5% TS. Similarly, for Plant B, a 35.8% increase in specific gas production was observed, from 0.212 L CH_4/g of TS without pretreatment to 0.288 L CH_4/g of TS with alkaline pretreatment at 0.5% TS. Optimal results were obtained at a concentration of 0.5% TS for both sources. This concentration yielded higher % solubilization during the alkaline pretreatment step compared to other TS concentrations. Statistical analysis using ANOVA demonstrated a significant difference in the efficiency of organic matter removal and specific gas production between the Alkaline Pre-treatment and Non-Pretreatment conditions of excess sludge from WAS at Plant A and Plant B, with a 95% confidence level ($p < 0.05$). The Alkaline Pre-treatment condition exhibited superior performance in both organic matter removal and specific gas production compared to the Non-Pretreatment condition.

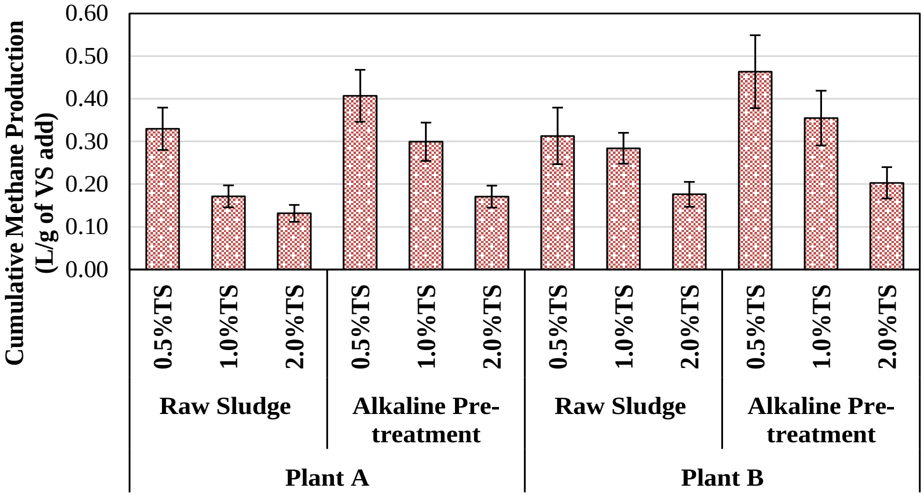


Figure 5 Cumulative methane productivity of Plant A and Plant B

4. Conclusions

Alkaline pretreatment using NaOH to adjust the pH to approximately 8.5 for a duration of 2 hours significantly enhanced the solubilization of SCOD in waste activated sludge (WAS) across various total solids (TS) concentrations. The treatment led to notable increases in SCOD solubilization for both Plant A and Plant B, with Plant B generally showing higher SCOD solubilization percentages. However, Plant A exhibited a higher increase in SCOD solubilization compared to Plant B. The TDS/TS ratios improved considerably, indicating effective conversion of particulate organic matter to soluble forms. Increases in ammonia nitrogen (NH_3-N) and phosphorus concentrations were observed, particularly at higher TS concentrations, reflecting enhanced solubilization and release of these compounds. The pretreatment notably improved the anaerobic digestibility of WAS, with higher efficiency at elevated TS concentrations. For instance, at 2.0% TS, both plants demonstrated significant improvements in efficiency, with higher removal rates for TS, VS, and COD. Alkaline pretreatment also increased specific methane production, enhancing the overall performance of anaerobic digestion. Optimal results were achieved at 0.5% TS, where higher solubilization was observed compared to other concentrations.

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