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Sustainable Low-Thermal Pretreatment Enhances Substrate Solubilization and Biogas Production: a Case Study with Potato Peel Waste

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Abstract- Low-thermal pretreatment has been recently reported as an environmentally sustainable, energy efficient and chemical free method for pretreatment of complex substrate for anaerobic digestion. In the present study, the effect of low-thermal pretreatment as a sustainable mean was evaluated on potato peel waste (PPW) for enhancing the biomethane production by anaerobic digestion, with special emphasis on accelerating solubilization of COD and carbohydrates. Various temperatures in the mild range (60, 80 and 100°C) with varying pretreatment times (20, 40 and 60 min) at individual temperatures have been thoroughly evaluated to maximize biomethane yield. Results indicated that the low-thermal pretreatment of PPW facilitated the organic matter solubilization in terms of carbohydrates and COD by up to 26.51 and 21.91%, respectively. The low-thermal pretreatment enhanced biomethane production with112 %increase in yield. The maximum of 2.12 fold increase in methane yield was observed at the pretreatment conditions of 100°C and 60 min.
Key words- agro-industrial waste; potato peel waste; low-thermal pretreatment; substrate solubilization; biomethane

I. INTRODUCTION

The potato peel waste (PPW) is one of the agro industrial wastes (AIW) generated from the various potato processing industries. It contributes to 15-40% of total potato waste and is considered of zero importance for further processing [1]. The PPW is mainly composed of 30-35% of carbohydrates (dry weight basis), 20-25% of lignin and suberin, 17-25% proteins, and 3-5% lipids [2]. Due to this suitable biochemical composition with high content of polysaccharides and proteins, PPW is considered as a promising feedstock for anaerobic digestion [1]. The mitigation of the potato waste is becoming a great challenge for the industries, and a huge amount of the waste is being disposed of as municipal waste or sewage. Moreover, the management and treatment of the PPW attracts additional cost to the potato processing industries since it is also known to be responsible for membrane chocking during wastewater treatment.

Anaerobic digestion (AD) is of particular interest in PPW treatment since it can serve as the dual purpose of nutrient recycling and co-generation of energy through methane production [3]. Anaerobic degradation of the PPW is hampered due to its rigid structure and lignified cell wall, protecting the intracellular components [2, 4]. Hence, hydrolysis of the PPW requires longer retention time and has been identified as the rate-limiting step. To enhance the rate of hydrolysis and performance of AD, the pretreatment processes to increase the degree of solubilization of the organic matter for accelerating the AD through waste disintegration are being evaluated. Increasing the degree of the solubilization by pretreatment enables less waste production, solids reduction, and enhanced biogas production compared to the classical AD [5]. A number of pretreatment methods have been reported to increase the rate of hydrolysis of various substrates [3, 6, and 7]. However, very few reports are available for the pretreatment of PPW. These PPW pretreatment methods include dilute alkaline [4], steam explosion [6], and thermal pretreatment [8, 9]. The steam pretreatment was studied for potato pulp, potato peel pulp and potato fruit water and observed that a maximum biogas production of 332, 377 and 323 lN (kg-1 VS), respectively [10]. Rani et al. (2012) have observed that 103% enhancement in biogas production after low temperature thermo-chemical pretreatment (50-80 °C) of dairy waste. Appels et al. (2010) have reported that the low thermal pretreatment (70-100 °C) of sludge resulted in enhanced biogas production up to 10 folds as compared to control. Chandra et al. (2014) have observed that 40-56% enhancement in bio-methane content after thermal pretreatment (100-150 °C) of defatted algal biomass. Beszédes et al. (2009) have reported that the acidic microwave pretreatment of canned maize resulted in 25-fold of increase in biogas production, while the combined pretreatment (ozone/microwave) showed only 10 folds increment [11]

Interestingly, low-thermal pretreatment which can have significant effect on PPW for the organic matter solubilization, solids reduction, and enhancing the susceptibility for the AD has not been reported. The major advantages of low-thermal pretreatment are that these methods are chemical free, cost effective and do not require any specialized reactors. Further, the operational cost of low-thermal pretreatment is lower than that of any other pretreatment method such as acid, alkaline and microwave pretreatment etc. [3]. Furthermore, it has been reported via comprehensive Life Cycle Assessment (LCA) that, the low-thermal (<110 °C) pretreatment is the most sustainable pretreatment process in terms of efficiency, energy balance, economic and environmental sustainability, and operational and maintenance costs in comparison to other pretreatment methods such as mechanical, chemical, and biological pretreatment methods [12].

To best of our knowledge no studies have yet been reported for the low-thermal pretreatment as an effective pretreatment option for organic matter solubilization and subsequent biogas production from PPW. Therefore, the objective of the present study was to evaluate the effect of low-thermal pretreatment of PPW for biogas production by facilitating the solubilization of organic matter [carbohydrates, soluble carbohydrate (SC); COD and soluble COD (CODS)], and subsequent anaerobic digestion.

II. MATERIALS AND METHODS

2.1 Feedstock and inoculum

The potato peel waste (PPW) sample was collected over a 2 h period in April 2018 from a stream of local potato processing plant at Mysore, Karnataka, India. The PPW was thoroughly mixed and stored at -20 °C in a plastic container until further use. The inoculum was freshly collected from the effluent of biogas digester located at Department of Microbiology and fermentation, CSIR-CFTRI, Mysore, Karnataka, India. The physico-chemical characteristic of the PPW and inoculum were done as per the standard methods and represented in Table 1. The chemicals used in the present study were procured from Sigma and Himedia Pvt. Ltd., India.

2.2 Low-thermal pretreatment of PPW

Low-thermal pretreatment of PPW was performed in an open reflux COD (Chemical Oxygen Demand) digester (Model: Bio-Technics) equipped with air-cooled condensers [3]. Three temperatures (60 °C, 80 °C, and 100 °C) were selected for thermal pre-treatment of PPW. The digester was fed with 100 ml of 5% PPW and subjected to each of the pretreatment temperatures individually. The pre-treated samples were withdrawn at various time intervals (20, 40 and 60 min). Post-treatment, the samples were placed in the cooled water bath to ensure rapid cooling. During the treatment, the samples were gently mixed to avoid the temperature gradient. An untreated sample was also analyzed as the reference for all the treated samples.

Table 1 Characterization of PPW and inoculum

Parameter	Substrate	Inoculum
TS (mg/L)	49419	24932
TSS (mg/L)	38170	16627
TDS (mg/L)	11249	8305
Ash (%)	0.8	0.4
VS (% of TS)	84.8	84.1
COD (mg/L)	35200	-
Carbon (%)	40.48	-
Nitrogen (%)	3.374	-
C/N ratio	14.80	-
pH	6.2	7.5

Average of two sets of data with SD < 5 %

2.3 Anaerobic digester setup and analysis

The fresh anaerobic sludge obtained from a biogas digester located at Microbiology and Fermentation department, CSIR-CFTRI, Mysore, Karnataka, India was used as inoculum. The inoculum was degassed to deplete any residual biodegradable organic material present in it. Pre-incubation was done at the same temperature from which the inoculum was sourced. Degassing was continued until no significant gas production was evidenced. During pre-incubation, the necessary micro/macronutrients/vitamins were supplied for optimal performance of the inoculum to maintain the inoculum in an active state.

Biogas experiments were carried out in serum bottles of 125 ml capacity. The assay bottles were flushed continuously with N_2 before transferring the substrate and inoculum. Pretreated PPW serving as the substrate (80 ml) was transferred to the bottle and nitrogen was purged continuously to remove any traces of oxygen.

Subsequently, 16 ml (20%) of inoculum with 20 g/L of volatile solids (VS) was added to the bottles with the substrate. The bottles were closed with thick butyl rubber stoppers and crimp sealed to maintain strict anaerobic conditions. The bottles were incubated under mesophilic (37±2 °C) condition in a constant temperature BOD incubator (Biotechnics) for 10 days, and the contents were shaken to mix manually for 1 min, twice a day [3].

2.4 Biogas measurements

The volume of biogas produced was measured on a daily basis using a glass cylindrical graduated impinger (250±5 ml, Polltech Industries Pvt. Ltd., India) by water displacement method [3]. Samples were withdrawn at regular intervals and were analyzed for pH to ensure the slightly alkaline conditions for the growth of methanogenic micro-organisms. While methane (CH_4) and carbon dioxide (CO_2) percentages were analyzed using portable gas analyzer equipped with an infrared sensor (OX300B, Aaru Engineers, India) [13].

2.5 Component analysis

The PPW samples were analyzed for the below mentioned components: total solids (TS), total suspended solids (TSS), total dissolved solids (TDS), volatile solids (VS), chemical oxygen demand (COD), elemental analysis (CHNS) and carbohydrates and were presented in Table 1. The TS content was determined by drying at 105 °C for 24 h. The TSS and TDS were estimated by filtering the well-mixed sample through a Whatman filter paper No.1. The solids retained on the paper (TSS) and the filtrate collected through the paper (TDS) was dried at 105 °C till a constant value was obtained. The VS content was determined by the weight loss of TS at 550 °C. All the components were analyzed as per the standard methods. The C and N contents of the PPW were determined by an elemental analyzer (ELIII, Vario, Germany). The total and soluble carbohydrate of PPW was determined by phenol-sulfuric acid method. For soluble carbohydrate (SC) the PPW sample was centrifuged at 8000 rpm for 15 min followed by filtration through 4 µm filter paper [14]. The aqueous fraction collected after filtration was used for the quantification of COD_S as followed by Alvarado-Lassman (2017). The total and soluble COD of PPW was estimated by dichromate method. For soluble COD, the PPW sample was centrifuged at 8000 rpm for 15 min followed by filtration through 4 µm filter paper [14]. Throughout the article, the term "soluble" represents the analysis performed to the supernatant and the term "total" represents un-centrifuged PPW.

The degree of solubilization of COD and carbohydrate was calculated by the following equation:

$$Degree\ of\ solubilzation\,(\%) = \frac{\text{soluble concentration}_{\text{treated}} - \text{soluble concentration}_{\text{untreated}}}{\text{total concentration}_{\text{treated}}} * 100 \qquad (1)$$

III. RESULTS AND DISCUSSIONS

3.1 Effect of low-thermal pretreatment on PPW solubilization

In this study, low-thermal pretreatment of potato peel waste (PPW) was performed to improve the hydrolysis efficiency in terms of substrate solubilization and to facilitate enhanced methane production during anaerobic digestion. The pretreatment was performed at three different temperatures (60, 80 and 100 °C) and treatment time (20, 40 and 60 min). The untreated PPW was considered as the control to compare it with all the pretreated PPW. The effect of low-thermal pretreatment of PPW was an enhancement of insoluble material, COD solubilization (COD_S), carbohydrate solubilization (CS), solids reduction, and biogas production. The effect of various pretreatment conditions on these parameters is detailed in following sections.

Table 2 Concentration and solubilization of organic components from untreated and pretreated PPW

Parameter	Untreated	60 °C			80 °C			100 °C		
		20 min	40 min	60 min	20 min	40 min	60 min	20 min	40 min	60 min
COD_t (mg/L)	35200	35200	38253	40001	40000	48000	52800	40000	48000	56000
CODs (mg/L)	850	1936	2561	3258	2310	3584	4987	4985	5989	8564
Solubilization (%)	0.00	3.09	4.86	6.84	4.15	7.77	11.75	8.91	14.60	21.91
Carbohydrates (%)	21.06	21.49	22.29	22.65	22.83	23.72	23.81	24.25	24.97	26.04
Soluble carbohydrates (%)	4.39	4.69	5.62	6.17	5.94	6.92	8.19	7.29	9.90	9.97
Solubilization (%)	0.00	1.45	5.87	8.45	7.39	12.05	18.05	13.76	26.15	26.51

Average of two sets of data with SD < 5%

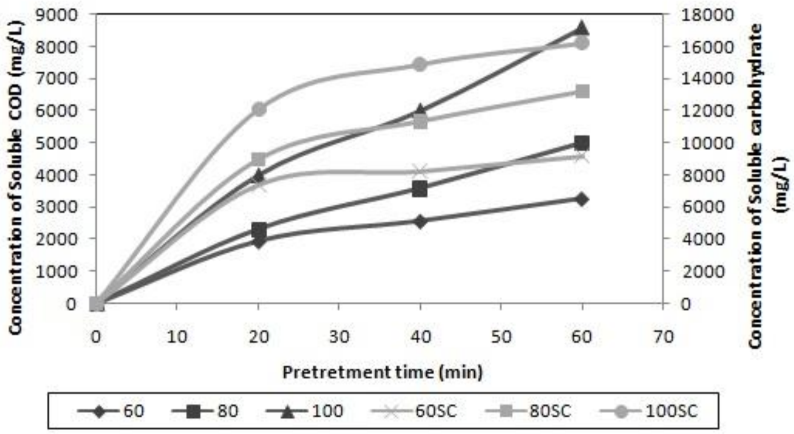


Figure 1 Degree of solubilization of COD (CODs) and carbohydrates (CHOs) in liquid phase of PPW at various pretreatment conditions

3.2 COD solubilization (COD_S)

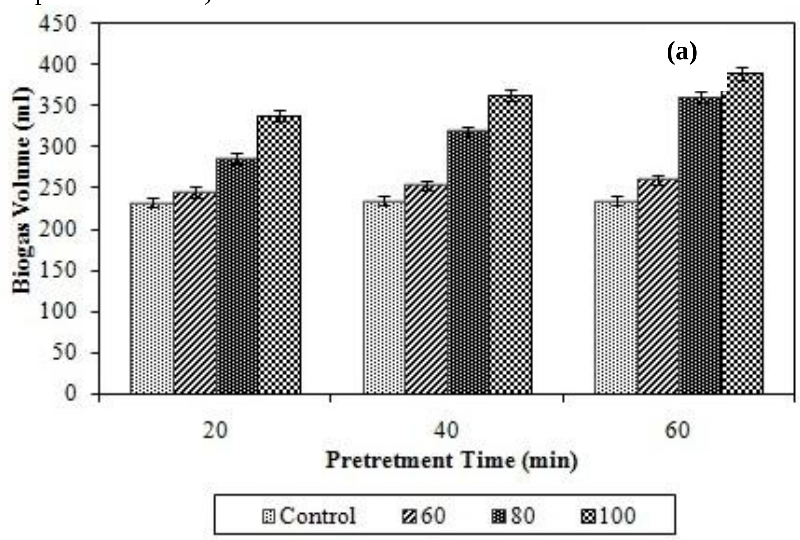
The primary goal of any pretreatment is to hydrolyze the complex organic substrate into a simpler form and further improves the bioavailability of substrate particulate matter. Several studies have highlighted the importance of pretreatment for enhancing the bioavailability of soluble organic matter which facilitates the anaerobic digestion of complex waste material [14, 15]. The thermal pretreatment of waste was reported to be one of the efficient pretreatment methods for anaerobic digestion [5]. It aids in the solubilization of the organic matter by disrupting the chemical bonds in the cell wall and cell membrane [3]. The COD_S was considered as the major parameter for evaluating the PPW particulate matter, and it also enables evaluation of the maximum degree of solubilization. The increase in COD_S is considered as an indicator of the substance that is readily available for methane production during anaerobic digestion [14]. The COD_t and the COD_S (in the aqueous phase) at various pretreatment conditions are represented in Table 2. In the present study, the COD_S of the pretreated PPW have increased with an increase in pretreatment condition (temperature and time). A maximum COD_S of 8564 mg L^{-1}, with a degree of solubilization of 21.91% was observed at a temperature of 100 °C and treatment duration of 60 min (Figure 1). It is evident from the Table 2 that, as the duration of the pretreatment increased from 20 to 60 minutes, an increase in COD_S was observed in the range of 4985 to 8564 mg L^{-1} with a degree of solubilization of 8.91 to 21.91% (Figure 1).This may be attributed to the disruption of chemical bonds in the cell wall and cell membrane due to thermal treatment that releases the organic matter in the liquid phase and thus enhances the COD_S. It was also observed that the increase in the COD_S and degree of solubilization of the PPW was limited (6.84 % solubilization) at 60 °C when compared to the higher temperatures. At this temperature (60 °C), even at higher pretreatment time (60 minutes), the value of the COD_S was only quadrupled. However, at higher temperatures (80 and 100 °C), up to 10 fold increase of COD_S was observed. Similar results were reported for low-temperature thermo-chemical pretreatment of dairy waste activated sludge; where at a higher pretreated temperature (80 °C) resulted in maximum COD solubilization of 25% [5]. Further, Chandra et al. (2014) have also reported thermal pretreatment of defatted algal biomass in the temperature range of 100-150 °C and time 20-60 min, which resulted in an increase in solubilization of total organic carbon (TOC) up to 6.2 % only. Similarly, the thermal pretreatment of waste activated sludge at an optimal temperature of 90 °C with pretreatment time of 60 min resulted in the solubilization of the COD up to a maximum of 17.81% [14]. The thermo-chemical pretreatment (pH 10, 55 °C) of municipal organic waste resulted in an enhancement of solubilization of organic matter especially the soluble COD [15]. The concentration of soluble COD was increased by approximately 50%, from 14.6 to 21.9 g/L. The COD solubilization has been considered as the reliable indicator of pretreatment efficiency for various substrates, and an increase in COD solubilization represents one of the main predictors of anaerobic digestions efficiency [16].

3.3 Carbohydrate solubilization (CS)

Release of the organic components into the aqueous medium upon cell lysis is the first stage of particulate matter solubilization. Carbohydrates are the principal organic components of biomass, and they contain carbon, which is a common organic component like hydrogen, oxygen, and nitrogen. Several studies have been reported that the thermal pretreatment enhances the solubilization of carbohydrates in the aqueous phase, which further facilitates the bioavailability of soluble carbohydrates for the effective AD [15]. Hence, it is important to determine the soluble carbohydrate content prior to anaerobic digestion. Table 2 represents soluble carbohydrates in the aqueous fraction of PPW at various pretreatment conditions. In general, it is evident from the study that the carbohydrate solubilization was in a positive correlation with the pretreatment time and temperature. Nevertheless, only marginal increase in the soluble carbohydrate was noted at low temperature (60 °C) even at higher pretreatment durations (60 min). Further, it was observed that the shorter pretreatment time of 20 and 40 minutes at higher temperature (80 °C) had no significant effect on the carbohydrate solubility. However, when the pretreatment time of 60 min was tested, the solubility of carbohydrate was doubled with a degree of solubilization being 18% (Figure 1). Similar results were reported for thermal and mechanical pretreatments of secondary sludge on biogas production, where thermal pretreatment at 116 °C for 38 min resulted in 383% increment of FVS/TVS ratio. Whereas, at the same temperature and prolonged time (78 min) showed an enhanced increment of 429% of FVS/TVS ratio [18]. This observation clearly indicates that at low-thermal pretreatment temperatures, time plays a dominant role than the pretreatment temperature [14]. However, at higher pretreatment condition (100 °C, 60 min) the SC was found to be in the range of 7 to 9 % with 26% degree of solubilization (Figure 1). Rani et al. 2012 have also reported that increase in the pretreatment severity on the sludge resulted in an increase in the soluble carbohydrate content. Similar results were also reported by others, where the increase of the pH leads to enhancement of the carbohydrate content of activated sludge [19]. This might be due to the degradation of extracellular polymer substances (EPS) and cell lysis during the thermal treatment. The carbohydrates are mainly localized in the EPS, and its degradation resulted in the decomposition of carbohydrates and subsequent release in the aqueous phase [20]. It was also visually confirmed that the color of the supernatant post-treatment was turned from transparent to slightly brown.

3.4 Effect of low-thermal pretreatment on biogas generation potential of PPW

The effect of low-thermal pretreatment of PPW on biogas production is represented in Figure 2. It was observed that the low-thermal pretreatment effectively enhances the biogas production during the subsequent anaerobic digestion. At the pretreatment temperature of 60 °C, the biogas production was marginally increased. On the other hand, at higher pretreatment temperatures of 80 °C and 100 °C, a significant enhancement of biogas volume (70%) was observed when compared to control (Figure 2). As indicated earlier, a large fraction of organic matter (carbohydrates and COD) was also solubilized at the 80 °C and 100 °C pretreatment temperatures. Similar results were also observed by Appels et al. (2010), where the thermal pretreatment of sludge showed low biogas volume at low pretreatment temperatures. Chandra et al. (2014) have studied the thermal pretreatment of defatted algal biomass and observed that the higher pretreatment condition of 150 °C for 60 min yielded a high biogas volume (up to 2 fold higher as compared to control).



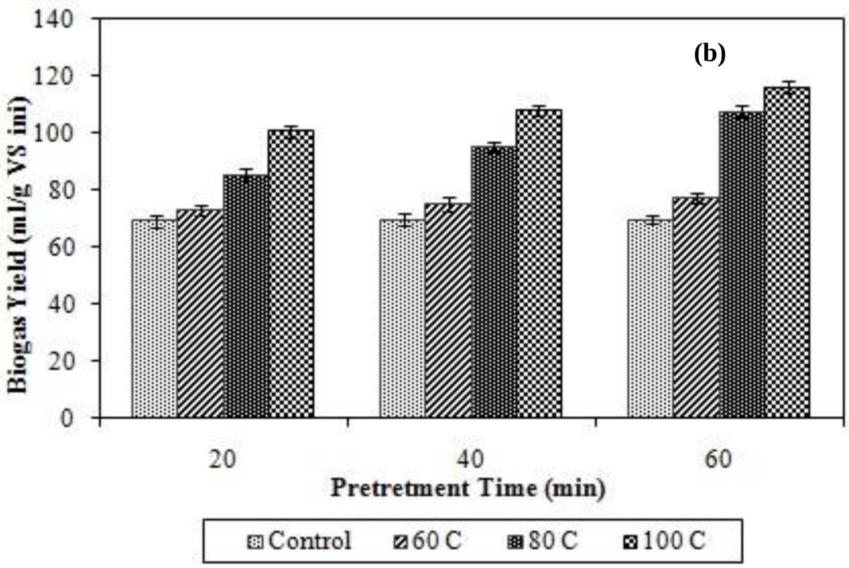


Figure 2 Biogas volume (a) and biogas yield (b) obtained at various pretreatment conditions

In the present study, the CH_4 content of biogas of untreated samples was observed to be in the range of 38–40% (v/v). The methane content was increased up to 50% (v/v) at a pretreatment temperature of 100 °C and duration of 60 min, while, at low pretreatment severity conditions (60-80 °C), methane was found to be around 45% (v/v). Biogas with higher CH_4 content (≥50%) is advisable for combusting purpose [3]. It is evident from the Figure 3 that the increase in temperature and pretreatment time has a significant effect on the methane generation. Compared to untreated PPW, higher methane yields were obtained from pretreated PPW. Methane yield for untreated PPW was 28.33 ml g^{-1} VS_{ini}, while the pretreated PPW at the temperature of 100 °C and 60 min the value was 60.08 ml g^{-1} VS_{ini}, with an enhancement factor of 2.12 fold (112%). However, at low pretreatment conditions, the CH_4 production enhancement was minimal (10-15%). It was also observed that the cumulative biogas and methane volumes were observed to be in the range of 50-100 ml at first four days of the incubation, later it was increased up to 10 folds. The maximum methane content of 67% was observed at 8^{th} day for the experimental sample of 100 °C and 60 min as shown in Figure 4. Similar results were also reported by Liang et al. (2015) where the biomethane content of pre-fermented PPW was increased up to 60-70% after 8-10 days of AD. In another study, the maximum methane content of 49.8% was observed at the 15^{th} day of HRT [18]. Kryvoruchko et al. (2009) have also observed that the slower increase in CH_4 and biogas production was probably due to low initial hydrolysis rate of organic matter which results in accumulation of high organic matter leading to methanogenesis inhibition (Liang et al., 2015). Further, the thermal pretreatment of PPW reported at 122 °C for 35 min has resulted in an enhanced biogas yield of 35.7% when compared to control [8]. Santos et al. (2013) have also reported that the thermal pretreatment of PPW showed an increase of 29% of biogas yield over control PPW sample. Similar results were also reported for anaerobic digestion of lignocellulosic biomass where 50-60% of CH_4 content was produced at higher treatment conditions [22]. The higher thermal pretreatment conditions (150 °C and 60 min) of defatted algal biomass resulted in an enhancement of CH_4 content was up to 56% [3]. Appels et al. (2010) have also reported that at 80 and 90 °C, a significant amount of biogas has been produced. Thermo-chemical pretreatment of sludge at 60 °C resulted in an enhancement of 51% of biogas when compared to control [5].

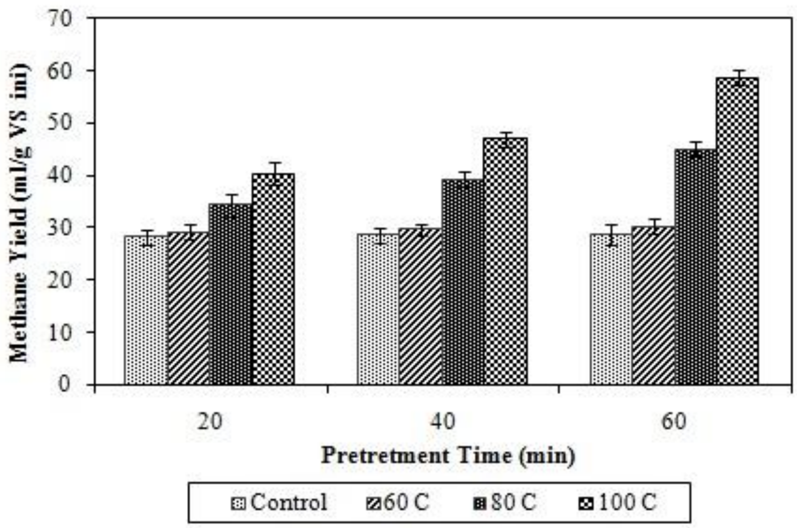


Figure 3 Methane yield (ml/g VS_{ini}) at various pretreatment conditions

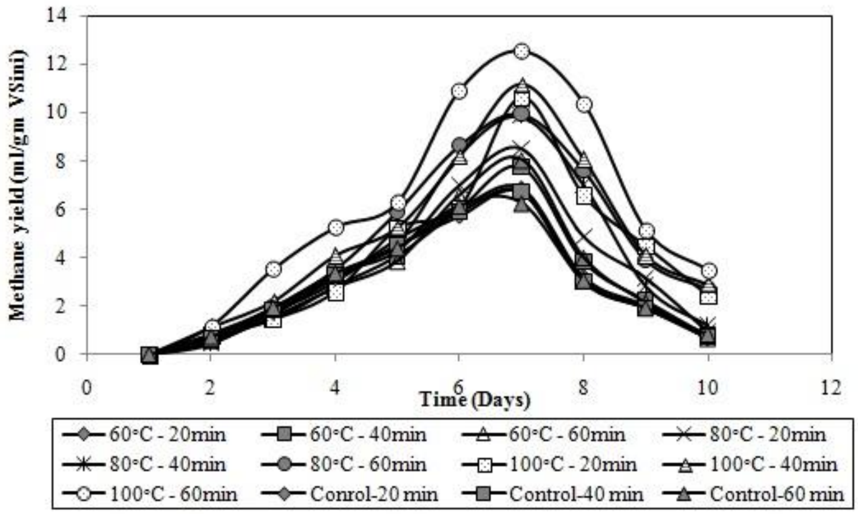


Figure 4 Pattern of methane production for the experimental duration

The observed results have indicated that the low-thermal pretreatment has potential effect on the yield of biogas using PPW. It has been noted that the net biomethane yield has increased by 112 % upon low-thermal pretreatment. Moreover, it may also be considered that the absolute yields of biogas obtained from a complex substrate PPW used in this study are probably on the lower side as compared to previously reported yields of 250 ml g^{-1} VS_{fed} with anaerobic digestion time of 40 days. Since the objective of the present study was limited to evaluate the relative effect of low-thermal pretreatment methods on the biogas yield, the detention time of only 10 days was studied. Therefore, the low detention time of 10 days may be one of the main reasons for lower absolute yields of biogas in this study.

IV.CONCLUSION

The low-thermal pretreatment of PPW (60, 80,100 °C; 20, 40, 60 min) facilitated the solubilization of organic matter in terms of carbohydrates and COD by up to 26.51 and 21.91%, respectively. Higher pretreatment temperature (80-100 °C) and time (40-60 min) were found to be beneficial for the enhanced solubilization of COD and carbohydrates. Low-thermal pretreatment facilitated biomethane yield up to 112 % (2.12 fold) at pretreatment conditions of 100°C and 60 min. The cumulative methane content of biogas was also relatively 30% higher in comparison to untreated PPW.

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