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Effect of Alkaline Pre-Treatment on Cassava Pulp for Optimum Biogas Production

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Abstract*: (Being produced in vast quantity as one of by-product from cassava starch processing chains, cassava pulp has great potential for energy recovery by harnessing biogas through anaerobic digestion (AD). This study aims to enhance biogas production by comparative investigation in batch mode digestion. 5%TS w/v of cassava pulp mixed with mill effluent were pre-treated with 10 molar potassium hydroxide (KOH), sodium hydroxide (NaOH), and calcium hydroxide ($Ca(OH)_2$) solution for 6 hours contact time. Effects of different alkaline pre-treatment on cassava substrate were assessed in total dissolved solid (TDS), soluble chemical oxygen demand (SCOD), Volatile Fatty Acids to Alkalinity ratio (VFA/TA), and reducing sugars. Daily accumulated biogas yield was taken as final indicator of the effect of different pre-treatment. KOH pre-treatment in pH 11 resulted highest dissolved solid 13.07 mg/L, and improved soluble chemical oxygen demand (SCOD) formation up to 75.61% (480,000 mg/L) than control substrate. The experiment revealed peak biogas production by KOH pre-treated substrate was found at day 6 after digestion executed, and achieved 546 ml. The finding proves out of different pre-treatment method applicable to cassava pulp, KOH pre-treatment could realistically increase biogas yield for cassava mills. Biogas production increased up to 101%, 92%, and 70% using KOH, $Ca(OH)_2$ and NaOH respectively. However, when future provision to the technology for AD system and design is concerned, the choice of highly reactive alkali could lead to complication in the system.

Keywords: Cassava, Anaerobic Digestion, Biogas, Pre-treatment, Alkaline.

I. INTRODUCTION

Climate change and global warming link to detrimental impacts of the massive fossil fuel consumption across the globe. This has to be reconsidered to avoid mounting environmental problems and dwindling resources. Therefore, it is mandatory to tap environmentally friendly energy to lessen dependence on fossil energy [1]. Renewable energy is one of the substitutions in the energy transformation. In comparison to its counterparts, biomass gains renewed interest because of its inherent advantages in large scale renewability, availability, storage capability, and adaptable to produce different bioenergy from diverse biomass [2].

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Biogas or bioethanol could be produced from agro-industrial by-products generated from production chains. Based on market demand and the dimension of agricultural sector, every country has unique potential to produce biomass as feedstock material for bioenergy production. Cassava or tapioca related foodstuff is one of the top agriculture products of Thailand as well as in the region. Thai Tapioca Starch (TTSA) reported the cassava production reached more than 33.94 million tons in 2016, and the figure is increasing. As of Thailand's Alternative Energy Development Plan (AEDP) 2015 which is to foster cutting greenhouse gases (GHGs) emission and boost carbon credit rating, out of biogas energy potential of 657 MW, it was targeted to increase from current figure of 312.95 MW to 600 MW by 2036 [3]. Though the target is achievable, technological challenges remain as major hindrance that the AEDP goal is to be met [4].

Biogas technology in its generic term is the anaerobic digestion (AD) process in which any digestible biomass is subjected for conversion into biogas from fermentation through symbiotic action of bacteria under strictly anoxic condition along four common stages (i.e. hydrolysis, acidogenesis, acetogenesis and methanogenesis) [5],[6]. The process of biogas could be divided into three divisions which are pre-digestion, digestion and post-digestion. While pre-digestion phase is related to processing feedstock material and digestion phase is associated with environmental variables and digester configuration, post-digestion is linked to proliferation of raw biogas into highly energy calorific values [7]. The advantages of pre-treatment to biomass prior to initiate AD process improves biodegradability of cellulosic material of biomass producing fermentable sugars, amino acids and volatile fatty acids as the products of hydrolysis stage [8]. Pre-treatment offers more food source from specific loading for hydrolytic bacteria and subsequently all microorganisms within AD system as whole for more biogas yield. Se Hoon Kim & Holtzapple (2005) reported that during fermentable sugar generation under calcium hydroxide pre-treatment for 0.5 g $Ca(OH)_2$ for 4 weeks contact time with up to 55° C temperature to corn-stover biomass, the overall yield of glucose and xylose were 91.3 and 51.8 at 15 FPU/g cellulose [9]. Similarly, Chang et al., (2001) recommended that $Ca(OH)_2$ of 0.1 g/dry mass for effective digestibility and better reducing sugar yield and more lignin solubility [8]-[10]. Likewise, calcium hydroxide, Sodium hydroxide has been widely used in different biomass pre-treatment.

By using NaOH up to 5 w/v, it was reported that total reducing sugar was produced 350mg/g in 20.5% NaOH with 120 ˚ C in bioethanol synthesis by kans grass [11]. On contrary, pre-treatment with NaOH alone and even with variable concentration, mixing rate and temperature resulted in low delignification levels of only 5-21% and low saccharification yield of 3-8% in bioethanol production studies [12]. In a comparative investigation among potassium hydroxide (KOH) and calcium hydroxide ($Ca(OH)_2$) of corn stover for bio-gasification study, under same bio-methane potential productivity, the dosage of KOH could be reduced up to 4 folds. By using one particular KOH, with different temperature and contact time settings, Raring et al., (2014) proved that carbohydrate conversion has been achieved 91.8% in switchgrass for sugar generation and subsequent biofuel production [13]. In general, alkaline pre-treatment has been extensively investigated to biomass which are difficult to biodegrade under bioconversion process. But with regard to the research related to cassava tubers and its related residues has not been much investigated as its counterparts.

This study fills this gap highlighting optimization of biogas yield from comparative alkaline pre-treatment in the absence of thermal effect to cassava pulp in batch rectors by three commercially available alkali namely caustic potash (KOH), caustic soda (NaOH), and slaked lime ($Ca(OH)_2$). The results of biogas yield from different alkaline pre-treated substrates were compared against alkali free control sets. This study aims to achieve ultimate biogas yield of alkaline pre-treatment to cassava pulp for biogas plants in cassava industry and promote the capability of enhancing biogas generation than conventional practice applied in biogas plant in the region.

II. MATERIALS AND METHODS

A. Substrate and Inoculum Characteristics

Fresh cassava pulp was collected from Korat Starch Factory located in Nakhon Ratchasima province in north-eastern Thailand. Upon proximate analysis, fresh cassava pulp of all variety was found having moisture contents between 55-70% on wet basis, 60-65% of starch and 15 - 18% of fibre respectively (AOAC, 1990) [14]. Wastewater from the starch mill had been chosen for hydrolysis and stored under 4˚C in cold storage until used. Hydrolysing for 15 mins was carried out prior to alkaline pre-treatment to ensure homogeneity of the bulk cassava pulp and wastewater.

Table 1: Characteristic of substrate and Inoculum

Parameter	Unit	Substrates	Inoculum
pH	-	4.3 ± 0.2	7.8 ± 0.3
TS	% (w/v)	1.5 ± 0.5	4.2 ± 0.3
VS	g VS kg^{-1}	68.5 ± 0.2	2.73 ± 0.2
Total COD	mg/L	831,500 ± 500	762,500 ± 500
SCOD	mg/L	18,500 ± 500	173,900 ± 500
Total VFA	mg/L	182.22 ± 20	128 55 ± 20

Triplicate analysis was carried out to each parameter.

APHA standard methods (1995) was applied to analyse substrate characteristics. Laboratory samplings were done prior to start anaerobic digestion in batch reactors. Characteristic of substrates and inoculum were shown in Table 1. Inoculum was taken from covered lagoon type biogas plant operated in parent cassava starch factory, acclimatized at 35 ˚C up to 3 hours, and executed AD process within 6 hours of collection time.

B. Alkaline Pre-treatment Experiments

Cassava pulp substrates were made in 15% TS (w/v) by mixing solid cassava pulp with 1000 ml effluent wastewater from starch mill. 20 N chemical solution of each potassium hydroxide (KOH), sodium hydroxide (NaOH), and calcium hydroxide ($Ca(OH)_2$) were prepared for alkaline pre-treatment and used to adjust desired pH ranging 9-11. To obtain the ultimate contact time, prepared samples were left stirring for alkali attack to lignocellulosic material of cassava pulp for 6 hrs. Finally, hydrolysed samples were neutralized back to pH value of 7.5 for digestion with concentrated hydrochloric acid (HCL). $NaHCO_3$ were added for buffer agent. Control sample which was not subjected to chemical pre-treatment were prepared in its original condition with same hydrolysis contact time for 6 hrs, and was compared against all other comparative alkaline pre-treated sample sets.

C. Analytical Method, Biogas Potential Assay and Statistical Analysis

The results of pre-treatments during hydrolysis were analysed by using APHA standard methods (1995) in several key parameters, such as total dissolved solid (TDS), soluble chemical oxygen demand (SCOD), total alkalinity (TA), Volatile Fatty Acids (VFAs), and reducing sugars (RS). To obtain optimum gas yield from the batch reactors of each set, batch reactors were set up in glass bottle of 1000 ml content. The head space was largely eliminated by adding 500ml of prepared substrate and 500 ml of inoculum. The inoculum to substrate ratio (F/M) was set at 1 and anaerobic digestion was maintained under 33 ˚C (+/- 2) in thermo-control room. Digestion period (HRT=SRT) was set 2 weeks (14 days). Mixing was conducted in 150 rpm for 2 min/12 hours. Biogas was collected in water displacement system and the total biogas yield was measured on daily basic before mixing to investigate accumulated biogas production rate and end of digestion when food source exhausted. To avoid possible interference, the variances between each sample during laboratory investigation of control parameters were eliminated by triplicating each individual sample set.

III. RESULTS AND DISCUSSION

A. Effect of Pre-treatment on Biomass Conversion

Complex insoluble organic matters, polymers are converted into soluble organic compounds, monomers with the help of hydrolytic bacteria during hydrolysis [15]. In term of solids analysis, among dissolved solid and suspended solid, only dissolved solids is accessible by bacteria for bioconversion processes.

Therefore, the total dissolved solids (TDS) represents digestible food source generated from Total Solid (TS). As a result of pre-treatment, the higher the formation of TDS indicates higher potential of foods source for microorganism. The results from the experiments revealed that KOH pre-treated substrates yield higher TDS yield under the same pH range. Kp-11 was found producing highest dissolved solubility at 13.07 mg/L, while NaOH and $Ca(OH)_2$ pre-treated substrates' TDS were ranging between 12.74 to 13.81 mg/L regardless of pH values. In comparison to control sample (Ctrl), due to the alkaline pre-treatment, TDS could be enhanced from 40% to 67%. In term of comparative performance among three alkalis, while NaOH and $Ca(OH)_2$ were found very identical biomass conversion capability, KOH was found having best TDS generation under same condition (Fig. 1). Although higher pH of each alkaline solution resulted better TDS formation, presumptive experiments indicated that pH value than higher than 11 required more alkaline concentration, and this surpasses inhibition threshold for microorganism to execute further biogas conversion process [6]. Therefore, separation of digestion face among hydrolysis phase and remaining AD phases is highly recommended in order to avoid inhibition caused by highly alkaline condition.

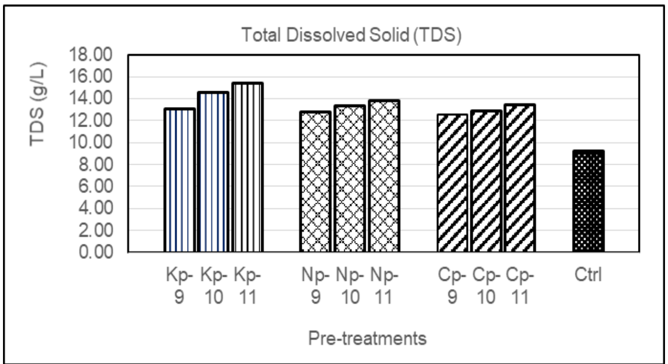

Figure 1: Total Dissolved Solids of Pre-treatments

B. Effect of Pre-treatment on SCOD

Soluble chemical oxygen demand (SCOD), which is being common carbon food source for microorganism during AD process is another indication of effect of pre-treatment. The original pH of substrate which is the mixture of cassava pulp and mill effluent was found at approximately pH 4.3- pH 4.5. The benchmark SCOD of the substrates which is control (Ctrl) samples was found 27333 mg/l. 200 times dilution was required in order to conduct very high SCOD by APHA's prescribed analytical method. The experiment discovered that KOH pre-treated substrates generated more SCOD within same pH of other alkaline pre-treatment. With the exception of Kp-11, the formation of SCOD in remaining substrates were found directly proportional to those of TDS formation for SOCD (Fig. 2). SCOD was observed highest in Kp-10, which was pH 10 pre-treated substrates by KOH. In comparison to control set, SCOD production was increased 26.83% to 56.1% in NaOH pre-treatment, 24.4% to 48.78% ($Ca(OH)_2$) pre-treatment, and 24.15% to 75.61% KOH pre-treatment respectively. Although COD in soluble form was considered as common food source for microorganism in AD processes, total chemical oxygen demand (TCOD) in which insoluble organic carbon can also behaves as food source for certain type of bacteria to speed up fermentation such as [16]. Nevertheless, the rate of bio-methane fermentation also depends on food to microorganism ratio (F/M). While low F/M ratio may lead imbibition to bacteria to initiate AD process, high F/M ratio can occur food exhaustion for microorganism within short digestion period [17].

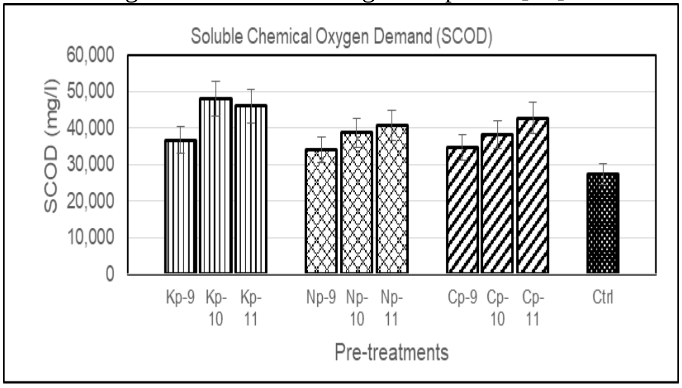

Figure 2. Soluble Chemical Oxygen Demand (SCOD) of Different Pre-treatments

C. Influence of Pre-treatments on Common Intermediary Products of AD Process

Common intermediary products such a volatile fatty acids (VFAs) and reducing sugars are as a result of hydrolysis phase in the AD process. From the results of all pre-treatment, higher pH ended up in lower VFAs values. Controlled sample (Ctrl) was found having highest VFAs values at 133.35 mg/l. Contrary to VFAs, total alkalinity increased as in the increased in pH values because the of the alkaline addition, pH 11 of KOH, NaOH and $Ca(OH)_2$ treated substrates were found reaching TA values of 380, 336.67 and 243.33 mg/L equivalent to $CaCO_3$. As a result of VFA/TA of the pre-treatment, while Control substrate's VFA/TA was found 3.58, those substrates which have been treated to pH 11 of respected alkaline were found having VF/TA under 0.1 – 0.38, which is the ideal range of VFA/TA for AD process [18]. VFA/TA of remaining substrates were found within the range of 0.39-1.1 (Fig. 3). Despite the fact too much higher or lower VFA/TA represents substrates are more likely to digestion failure, the range within 0.1 – 1.5 falls under manageable level by mean of adopting suitable mode of digestion [19]. Since this study employed batch mode which is totally enclosed chamber, the resultant kinetic of VFA/TA at subsequent AD process could not be investigated. Therefore, I could be remarked that highly alkaline pre-treatment is objectionable since it led to VFA/TA gap too high, resulting more likely to occur digestion failure by high alkalinity. Nevertheless, verdict could only be made through daily cumulative gas yield and digester performance at the end of the digestion period.This comparative alkaline pre-treatment of biomass investigation revealed effect of alkaline pre-treatment reduced VFAs formation as increased in pH of respective alkaline s solution. The rate of reduction was very rapid in $Ca(OH)_2$ treated sample while those of KOH and NaOH treated substrates were found slightly decreased. However, in comparison against control sample, VFA production by acetogenesis process increased 100% than control reactor in Cp-9. This study contrasts Fang et. al., (2019)'s work in which NaOH prolong pre-treated of risk husk resulted 72.9% increase in TVFAs generation [20].

37% VFA improvement was achieved by similar study by using sugarcane filter cake [21]. In both cases, VFAs were measured at the mid of AD process of either semi or full continuous reactor, whereas in this study because of batch process, measurement could not be done in the intermediate AD process along digestion period. However, biogas yield improvement in latter discussion is the evidence of VFAs were improved by the effect of comparative alkaline pre-treatment.

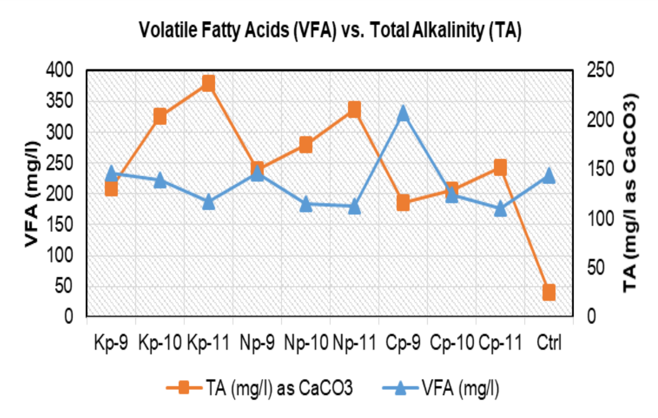

Figure 3: Volatile Fatty Acids (VFAs) and Total Alkalinity in Different Pre-treatments

D. Effects of Alkaline on Pre-treatment Biogas Yields and Production Rates

Based on the results of AD digestion in batch mode indicated that the performance of reactor in which KOH pre-treated substrates produced highest biogas yield in comparison to all other alkaline pre-treated counterpart. Maximum accumulated gas yield of KOH pre-treated sample was found at Kp-9 with 546 ml and gas yield decreased as increased in pH (Kp-10 and Kp-11). While biogas production was found inactive after day 11 of digestion period, the peak biogas production rate was found at day 5 and 6 in all KOH pre-treated substrates. (Fig. 4). This is due to fact that available food source exhaustion occurred for AD microorganism. The highest rate of biogas production occurred at least 3 days later (day 9) in NaOH pre-treated substrate. Amount of biogas produced in NaOH pre-treated substrates were also found lower as increased in pH. While Np-9 which is the substrate pre-treated with NaOH in pH 9 produced 523 ml at the end of digestion period, Np-10 and Np-11 produced 454.2 ml and 372.9 ml respectively. In contrast to KOH and NaOH pre-treatment, the biogas production rate of $Ca(OH)_2$ were found stable throughout digestion period with approximately about 25 ml per day and the total gas yields of all $Ca(OH)_2$ at the end of digestion with lesser gas yield in all $Ca(OH)_2$ pre-treatments. Nevertheless, the experiments revealed that due to the alkaline pre-treatment, biogas yield could be increased 57% to 101% in KOH pre-treatment, 37% to 92% in NaOH pre-treatment, and 51% to 70% in $Ca(OH)_2$ pre-treatment.

Thomas et. al., (2018) reported 67-227% increase in methane production under co-digestion with cattle manure in lime (CaO) pre-treatment [22]. Under mono-digestion, biogas production increase up to 101%, 92%, and 70% using KOH, $Ca(OH)_2$ and NaOH respectively. At least 100% methane improvement was obtained using NaOH for oil palm residues [23]. By using KOH pre-treatment, 77.5% more methane yield was possible for wheat straw [24]. In the case of rice straw pre-treated with $Ca(OH)_2$, more than 30% methane was enhanced in short duration pre-treatment [25]. This study resemble to this study, in which out of 3 different alkaline pre-treatment, $Ca(OH)_2$ performed lowest process enhancement. However, when the cost of the chemical is a concern, $Ca(OH)_2$ is the most economy to its counterparts. In most literatures, NaOH had been chosen for alkaline pre-treatment. If there is not further modification in the anaerobic digestion by catalytic agents or metal supplementation as nutrient supplementation, KOH and NaOH is more suitable for generating more biogas. But when advanced processes will be employed into the AD system, these tow chemicals can lead to complication with other supplemented chemicals into the system. Therefore, the choice of chemicals for biogas enhancement should be made based on provision in AD system and reactor configuration.

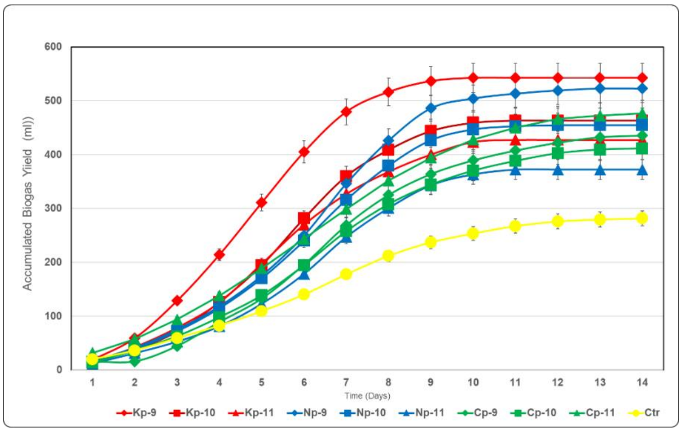

Figure (4): Cumulative Biogas Yield of Different Alkaline Pre-treated Substrates

IV. CONCLUSION

With cassava pulp having fine particles sized and highly carbohydrate residues, out of several pre-treatment methods available, alkaline pre-treatment method proved promising for rapid conversion of cassava pulp substrate for optimized biogas production. In addition to enhance gas yield, the comparative investigation among three alkaline (KOH, NaOH and $Ca(OH)_2$), KOH was proved best substrate degradability and gas yield within specific digestion period. In view of resource and chemicals consumption for alkaline solution preparation, KOH and NaOH demand less alkaline quantity during pre-treatment. Therefore, by adopting the results of the study on alkaline pre-treatment to cassava substrate, significant amount of biogas could be generated in cassava processing plants. However, for future provision into AD system the choice of chemicals could be a concern for the complication within biogas process when reactive alkaline agent is used for biogas enhancement.

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