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Anaerobic co-treatment of tannery wastewater and cattle dung for biogas production using a pilot scale anaerobic sequencing batch reactor (ASBR)

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Abstract: The anaerobic co-digestion of tannery wastewater and cow dung was investigated in a 100m3 pilot scale anaerobic sequencing batch reactor (ASBR) under mesophilic condition (31±1.50c). The experiment was conducted at five different mixing ratios (100:0, 70:30, 40:60, 20:80 and 5:95) of cow dung to tannery wastewater. The results of the study indicated that the average biogas productions were in the ranges of 18.67 to 24.4m3 per day and the methane yield was ranged from 0.19 to 0.30 m3/kg COD removed. The removal efficiencies was 75-82% for COD, 70-80% for TS and 81-89 for VS. The highest methane yield (0.29 m3/kg COD removed) with COD removal efficiencies of 82 and 81% was obtained at 20:80 and 5:95percent cow dung to tannery wastewater ratio by volume while the lowest (0.22 m3/kg COD removed) was observed at 100:0 percent cow dung to tannery wastewater ratio.

The results of this study showed that co-digestion of tannery wastewater with cow dung up to 80%by volume is possible to produce biogas with high methane content and methane yield.

Keyword: Anaerobic, ASBR, COD, methane yield, tannery wastewater

1. Introduction

Tanning is one of the oldest industries in the world. It is almost a wet process that consumes high amounts of water (about 30-40 L of water/kg of hides or skin processed) and also generates about 90% of the water as wastewater (IFC, 2007). Tannery effluents, which are discharged without proper treatment into water bodies or open land, contaminate surface as well as sub-surface water and soils (Andualem and Seyoum, 2011). Tannery wastewaters, which percolate into ground water for a long period, seriously affect the groundwater. Chromium and sulfide from the effluent pollute groundwater permanently and make it unfit for drinking, irrigation and general consumption. A single tannery can cause the pollution of groundwater around the radius of 7–8 km (Mondal *et al.,* 2005). Other negative effects include the loss of land productivity, retardation of the germination of plants and seeds (Bernel*et al.*, 2006). Now days, there is a growing environmental pressure against the leather processing activity because of the rise in salinity and heavy metals in the soil and groundwater (Saravanabhavan *et al.*, 2005 and Bernel *et al.*, 2006). In Ethiopia, modern tannery industries started in the mid 1920's (Favazzi, 2002). Currently, there are more than 30 tanneries under operation and this tannery produce 11,312m^3 wastewater daily which are disposed to the surrounding without proper treatments (LIDI, 2010). This wastewater is characterized by a high load of contaminants that require treatment before it can be discharged into a body of water. However, the treatment systems developed and used by most industries are frequently regarded as regulatory obligation that increase capital and operational costs and ultimately yield negative economic returns. Compliance to environmental legislations should not necessary lead to the creation of additional costs, but can instead provide a secondary source of income. One possible source of increased revenue available to industries is through taking advantage of anaerobic treatment. Anaerobic treatment is considered as sustainable method of reducing pollution from domestic, agricultural and industrial operations (Rao *et al*, 2005**;** Farhadian *et al.*, 2007). It consumes little energy as no aeration is needed and produces renewable energy in the form biogas. It also produces little sludge and efficient at removing organic matter and endure high loading rates which also reduces its space requirement **(**Cakira and Stenstromb, 2005**).** However, anaerobic digestion of tannery wastewater might be hindered due to the presence of toxic compounds for anaerobic microorganism. Anaerobic co-digestion of two different types of organic wastes is a cost-effective waste treatment method (Azaizeh and Jadoun, 2010). Co-digestion establishes positive synergism in the digestion substrate; dilutes toxic compounds and improve nutrient balances (Minale and Worku, 2014). This can support to maintain a reliable and stable performance of digester that can steadily produce a high volume of biogas and methane content. Different researchers obtained satisfactory results from co-digestion of industrial sludge and municipal solid waste (Nuri and Teresa, 2007), dairy manure and food waste (El-Mashad and Zhang, 2010), olive mill wastewater and swine manure (Azaizeh and Jadoun, 2010), cattle manure with food waste and sludge (Maranon *et al.*, 2012), cattle slurry and cheese whey (Comino *et al*., 2012), water hyacinth and beverage wastewater (Lay *et al.*, 2013), food waste and cattle manure (Zhang *et al.*, 2013) and sanitary wastewater and kitchen solid waste (Minale and Worku, 2014).

The objective of this study was to investigate the biogas production potential of tannery wastewater co-digested with cow dung at different mixing ratio in pilot scale Anaerobic

Sequencing batch Reactor (ASBR). Anaerobic sequencing batch reactor (ASBR) has high degree of process flexibility in terms of cycle time and sequence; possible elimination of equalization tanks and secondary clarifiers as well as relatively simple operations. Anaerobic sequencing batch reactor operated with four distinct phases per cycle (Zhang and Dugba, 2000; Andualem and Seyoum, 2011).

2. Material and Methods

2.1. Experimental device

The pilot-scale ASBR with a total volume of $100m^3$ was used in this study (Figure.1). The digester system has different accessories which include digester (1), control panel (2), agitation hydraulic pump (3), feeding system (4), gas pipe (5), gas meter (6), moisture trap (7), biogas storage bag (8), gas valve (9), gas blower (10), sulfur scrubber (11), generator (12) and gas line to the kitchen (13). The control panel was located in a closed box and included the electric system controls required for the functioning of the digester and collection of analytical data. A hydraulic mixer system was used to stir the systems. It was operated under mesophilic condition (31^0c) and the temperature was maintained by circulating hot water heated with solar panel.

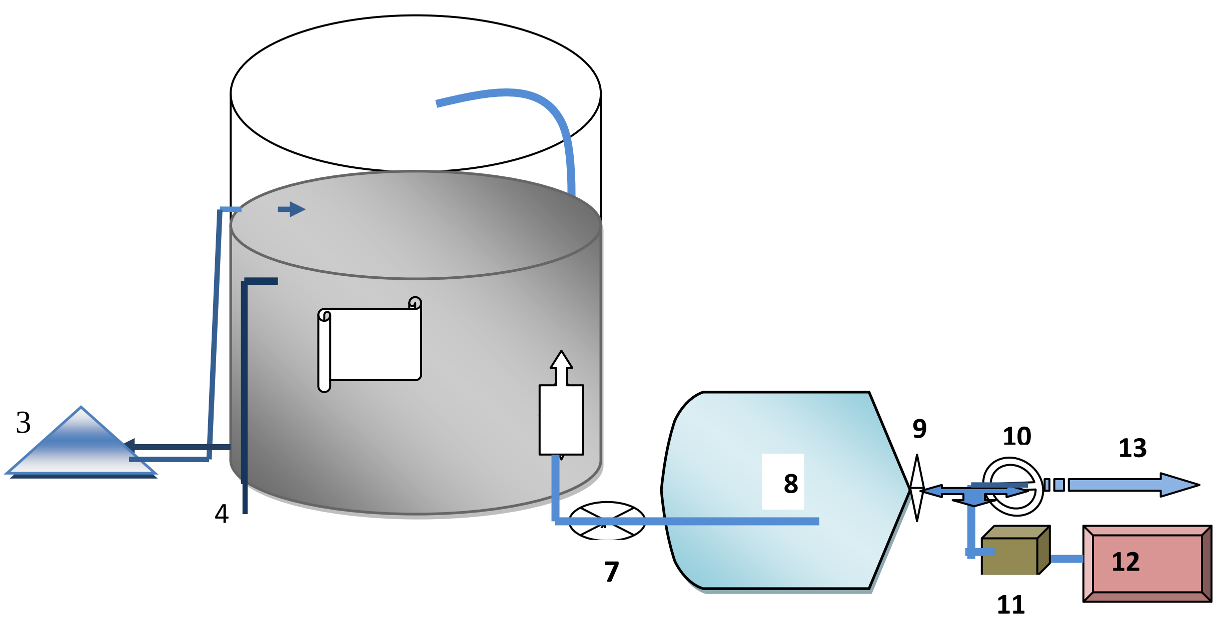

Figure 1: Schematic diagram of the pilot scale Anaerobic Sequencing Batch Reactor.

2.2. Operation of ASBR

The Anaerobic sequencing batch digester was inoculated with fresh rumen fluid collected from the nearby slaughter house and it was acclimatized for more than two months. At the end of the start-up phase, co-digestion of cow dung (CD) and tannery wastewater (TW) was started. The experiment was conducted in five subsequent phases with different mixing ratio. In the first phase of the experiment only cow dung mixed with tap water was used at OLR of $1.2 kg.m^{-3}.d^{-1}$ and HRT of 15.4 days. In the second phase, the experiment was carried out in the proportion of 70% cow dung and 30% tannery wastewater at OLR of $1.2 kg.m^{-3}.d^{-1}$ and HRT of 12.6 days. The third experiment was conducted using 40% cow dung and 60% tannery wastewater at OLR of $1.4 kg.m^{-3}.d^{-1}$ and HRT of 9 days. In the fourth phase 20% cow dung and 80% tannery wastewater was used for feeding at OLR of $1.3 kg.m^{-3}.d^{-1}$ and HRT was 6.7days. In the last phase, the proportion of the cow dung and tannery wastewater was 5 and 95%, respectively. The OLR and HRT used in the study were $1.1 kg.m^{-3}.d^{-1}$ and 5 days, respectively. In all the experimental phases of operation, the feeding operations were performed on daily bases.

2.3. Chemical Analysis

Chemical oxygen demand (COD), Total nitrogen (TN), Ammonium-Nitrogen (NH_4+ -N), Sulphides (S^{-2}), total phosphorous (TP) and sulfate (PO_4^{-3}) were measured colorimetrically using spectrophotometer (DR/2010 HACH, Loveland, USA) according to HACH instructions. Total solid and volatile solid were also measured according to the methods described in standard methods (APHA, 1998). pH, total dissolve solids (TDS) and salinity were measured using a pH/TDS/salinity meter (CON 2700). The biogas production was measured using wet gas meter and the biogas composition was determined using biogas meter (Biogas meter Geotechnical instruments, UK, England).

2.4. Statistical Analysis

Statistical analysis was performed with R package, EXCEL and Origin 8.0 software. Mean, Standard deviation and Analysis of Variance (ANOVA) were analyzed using R statistical package. The graphs were drawn using Origin 8.0 software. The comparison between mean was performed at 95% confidence interval.

3. Result and Discussion

3.1. Characteristics of cow dung and raw tannery wastewater.

The characteristics of cow dung and tannery wastewater used in the study are given in Table 1. The average pH of cow dung was 7.05 ± 0.07 and this pH of the manure was found to be suitable for anaerobic digestion. The tannery wastewater was alkaline in nature with pH value of 8.38±0.27. This high pH in the tannery wastewater was due to the different chemicals used in tanning processes. Seyoum (2004) and Adey *et al.* (2014) had also reported higher pH value.

Tables 1. Characteristics of cow dung and tannery wastewater used in the study

	unit	cow dung	tannery waste water	literature for tannery wastewater
pH	-	7.05±0.07	8.38±0.27	10.08±0.08[a], 10.40±0.3[b]
EC	mS	3.459±1.2	8.63±1.29	14.33±1.182 [a]
TDS	$g\ l^{-1}$	3.123±1.15	7.8±1.15	6.65±0.57 [a]
Salinity	$g\ l^{-1}$	3.434±1.1	9.1±1.4	9470.50±1335[b]
TS	-	12.5±2.3	7.95±0.24	-
VS	% based on TS	85.0±1.3	68.26±1.37	-
COD	$mg\ l^{-1}$	24,280±430	4546.5±667.5	11123±563 [a], 2547.50±3910[b]
TN	$mg\ l^{-1}$	1070±142	433±115.9	1330±182.1 [a], 245.25±76[b]
NH_4^+	$mg\ l^{-1}$	710±37	256±72.13	122.2±8.3 [a]
TP	$mg\ l^{-1}$	106±67	25.6±6.4	15.33±1[b]
S^{-2}	$mg\ l^{-1}$	-	264.4±29	630.4±67.0 [a], 55.50±6[b]
SO_4^{-2}	$mg\ l^{-1}$	-	440±85.4	502.0±82.0 [a], 800±505[b]

[a] Seyoum *et al.*(2004); [b] Adey *et al.* (2014)

Tannery wastewater contains high level of electrical conductivity (8.63±1.29mS), total dissolved solids (7.8±1.15g/L) and salinity content (9.1±1.4g/L). High levels of conductivity and salinity indicate the presence of inorganic substances and salts. This might be resulted from the chemicals used in the soaking and beam house operation (Lefebvre *et al.*, 2006). The levels of TS, VS, COD and total nitrogen in cow dung were higher than the tannery wastewater. The comparable COD value was also found by Seyoum (2014). On the other hand sulfate and sulfide were higher in the tannery wastewater. This high level of sulfide and sulfate might be due to the chemicals used in the beam house operation and from the sulfide from the hires and protein of the skins.

3.2. pH, TDS, salinity and ammonium profile in the digester

Figure 2 shows the variation of pH profiles inside the digester over time. As it shown in the Figure, the pH of the digester was low during the initial phase of the operation. This could be due to the high level of volatile free fatty acids (VFA) in the digester that resulted from the activity of acetogenic bacteria. The accumulation of volatile fatty acid can lead to a drop in pH and the continual drop in pH inhibits the methanisation process (Carucci *et al.*, 2005).

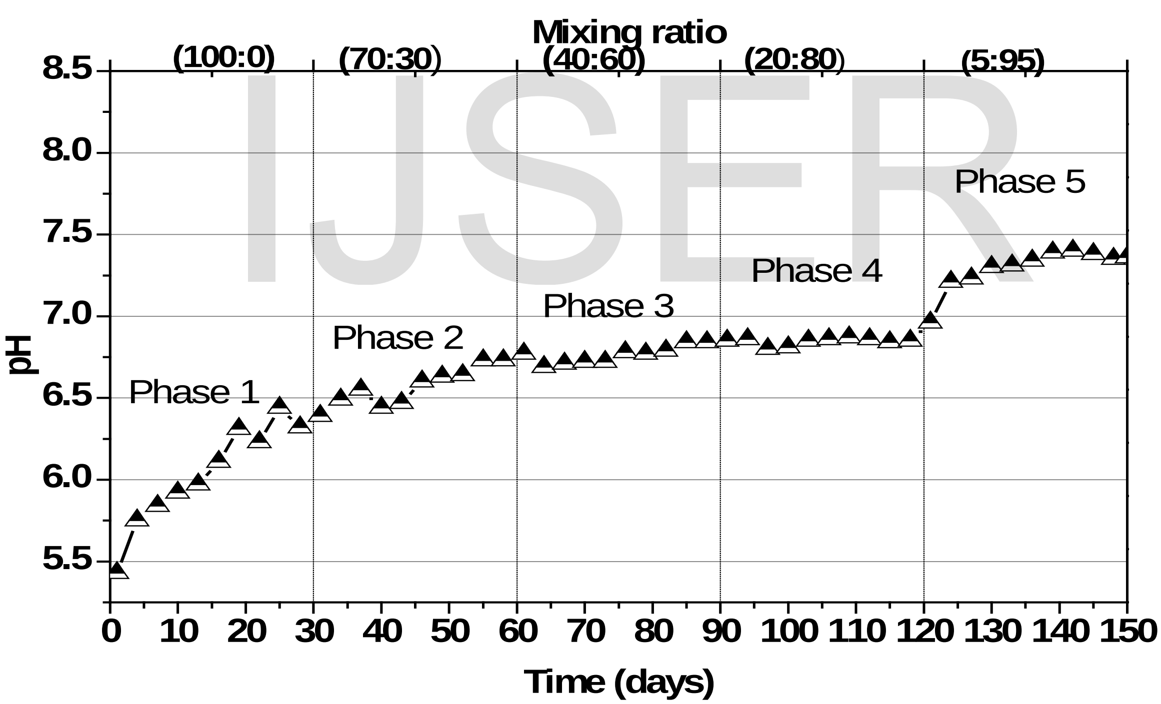

Figure 2: pH profile of the anaerobic SBR

The accumulation of VFA could be due to the presences of macromolecular organic material in the cow dung that are hard to be decomposed directly from the feedstock. The low efficiency of VFA decomposition by acetogenic bacteria could be another factor for the VFA accumulation (Mshandete *et al.*, 2004). The level of pH increased with increasing amount of tannery wastewater and time of operation. The highest pH was recorded in the final phase of operation. This might be partly attributed to acclimatization of methanogic bacteria and partly the amount high alkaline tannery wastewater used in the feeding. The pH observed in the initial phase of the reactor was suitable for hydrolysis and acidogenesis bacteria. The optimum pH for hydrolysis and acidogenesis are in the range of 5.5-6.5 (Kim *et al.*, 2003; Dobre *et al.*, 2014). The pH in all the remaining phases of the operation was suitable for methanogenic bacteria. The optimum pH for methanogenisis process is also in the range 6.5– 8 (Lee *et al.*, 2009b; Dobre *et al.*, 2014).

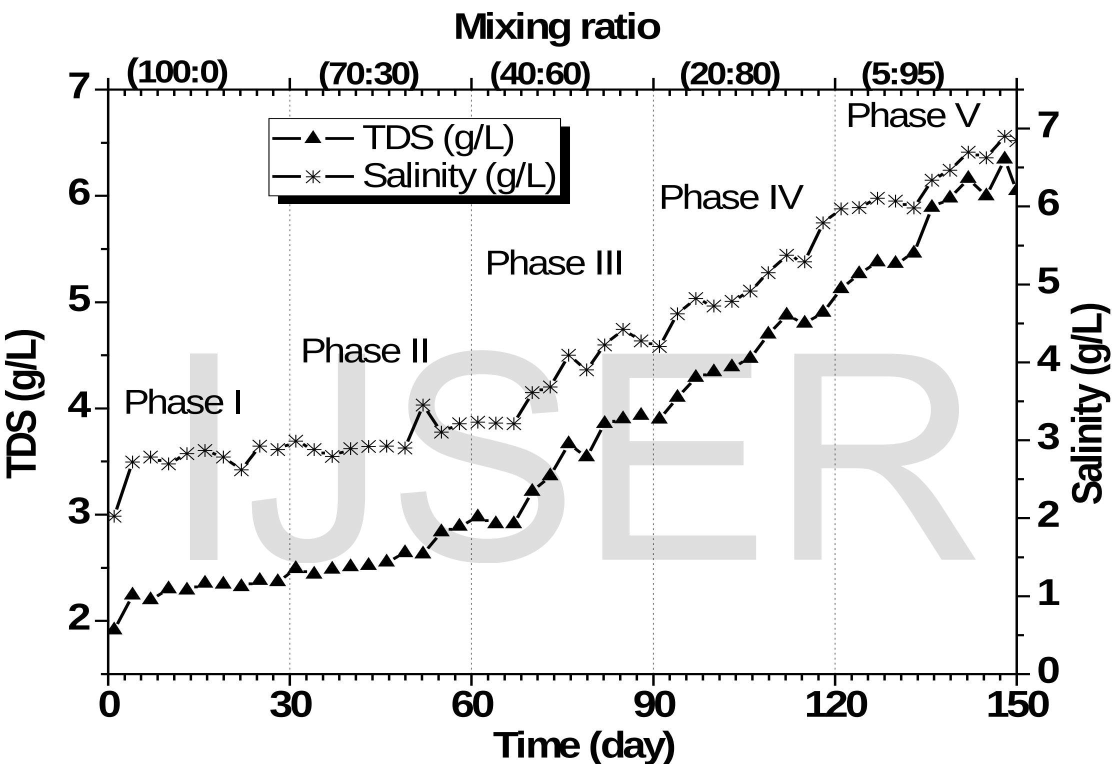

Figure 3: Variation of TDS and salinity level in the digester

The average TDS and salinity level in the digester were also ranged between 1.914 to 6.048g/L and 2.026 to 6.846g/L, respectively (Fig.3). TDS and salinity were lowest in first phase (when only cow dung was used) followed by the second phase, when the feedstock contains 30% tannery wastewater by volume percentage. The highest were observed in the last phase of operation. In other words, the level of TDS and salinity increased in the digester with increasing proportion of tannery wastewater. Tannery wastewater contains high value of TDS and salinity due to lime and other chemicals used in the tanning process (Lefebvre *et al.*, 2006). Studies indicated that high salinity (sodium concentration greater than 10 g/L) strongly inhibits anaerobic digestion process (Sunny and Mathai, 2013; Lefebvre *et al.*, 2006). Hence, the concentration of TDS and salinity in the digester were not in a level that can inhibit the activity of methanogenic bacteria.

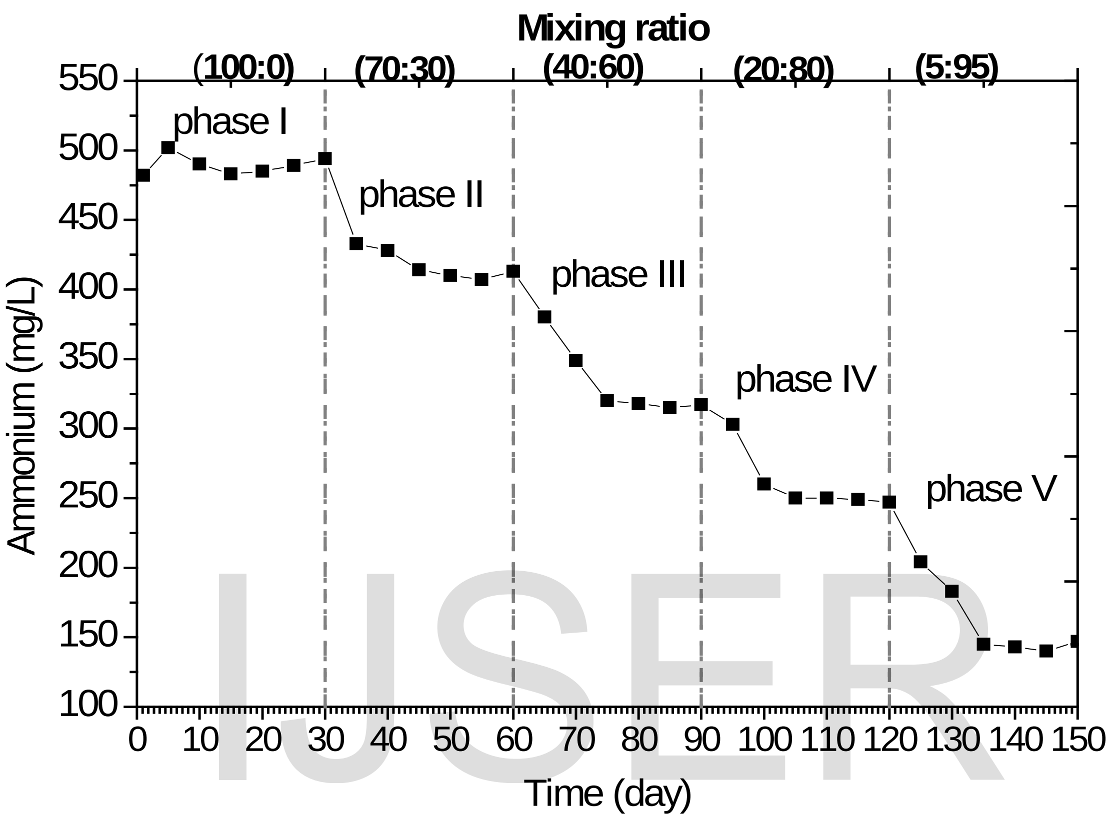

Figure 4: Variation of ammonia profile in the digester over the different phases

Moreover, the variation of ammonia concentration in the digester over the acclimatization period is shown in Figure 4. As it shown in the figure, the concentration of ammonia was varied in the range between 140 to 500 mg/l in all the experimental period. The highest ammonia concentration was observed when only cow dung was used as feeding substrate and the lowest was observed in the last phase of the acclimatization (5:95% of CW:TW). The average concentration of ammonia was 489±7.0 in phase I and 160±26mg/l in phase II. Ammonia concentrations less than 200 mg/l are important nitrogen sources (essential nutrient) for mico-organisms in anaerobic digestion (Chen *et al.*, 2008; Lise *et al*., 2008). However, high level of ammonia may cause reduction in microbial activity. This effect of ammonia is indicated by increasing volatile free fatty acid concentration and reduction in methane production rate (Rajagopal et al., 2013). Ammonium concentration in the range 560–568 mg NH_3-N/l at pH 7.6

might cause inhibition of methanogenesis process by 50% under thermophilic conditions (Sung and Liu, 2003: Lise *et al*., 2008). The concentration of ammonia in the digester was not in a level that can inhibit the activity of methanogenic bacteria.

3.3. Biogas production, gas composition and methane yield

The ASBR was run using various proportion of tannery wastewater and cow dung. Figure 5 illustrates the variation of biogas production rate per day and Figure 6 shows the variations of methane yield and biogas composition during the experimental period. During the initial phase (phase I), only cow dung mixed with tap water (1:3 CW to TW) was used as feeding substrate. The biogas production rate was lower in the first two weeks of the operation even if there was a continuous increment in the production rate in the digester. The methane content was also low. The low biogas yield with low methane content may partly attribute to the accumulation of VFAs due to slow acclimatization of methanogenic bacteria and partly the composition of cow dung. The composition is mainly fibrous material that is rich in lignocelluloses. The decomposition of these takes longer time due to the limited accessibility o lignocelluloses for enzymes (Chukwuma and Orakwe, 2014). Starting from the 17 days, the biogas production was reach as high as 47.5m^3 per day with an average gas production of 38.8m^3 per day.

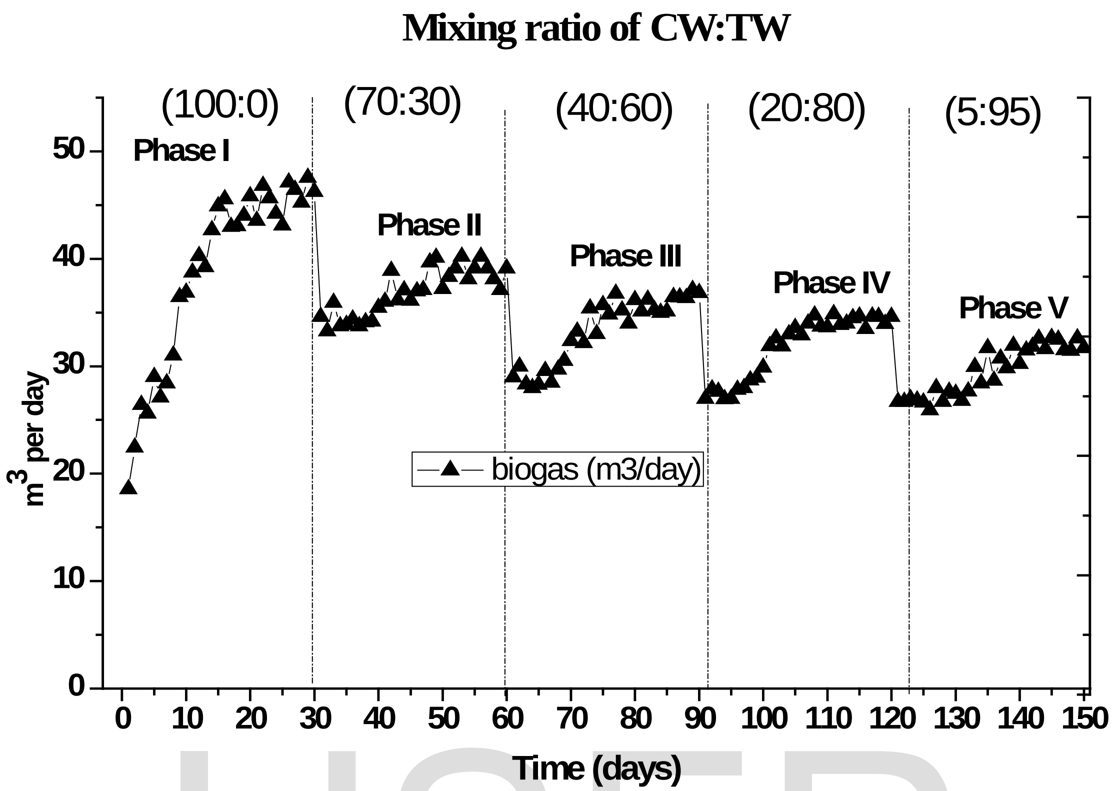

Figure 5. The variation biogas production rate at the different phases

Similarly, there was slight reduction in biogas production per day at the beginning phase II (at mixture of 70% cow dung and 30% tannery waste water). However, after the first ten days of the operation the digester showed steady state performance in biogas production with an average production rate of 36.9m^3 per day with 56.7% average methane content. The biogas yield was 0.24m^3/kg COD removed. When the digester fed with a mixture consists of 40% cow dung and 60% tannery wastewater, the average biogas production rate was 33.3m^3 per day with the methane content and methane yield of 59.6% and 0.25m3/kg COD removed, respectively. When proportion of tannery waste water in the mixture reaches 80% by volume, the methane content and methane yield was increased as high as 72% and 0.29m^3/kg COD removed with 31.8m^3 gas production rate per day.

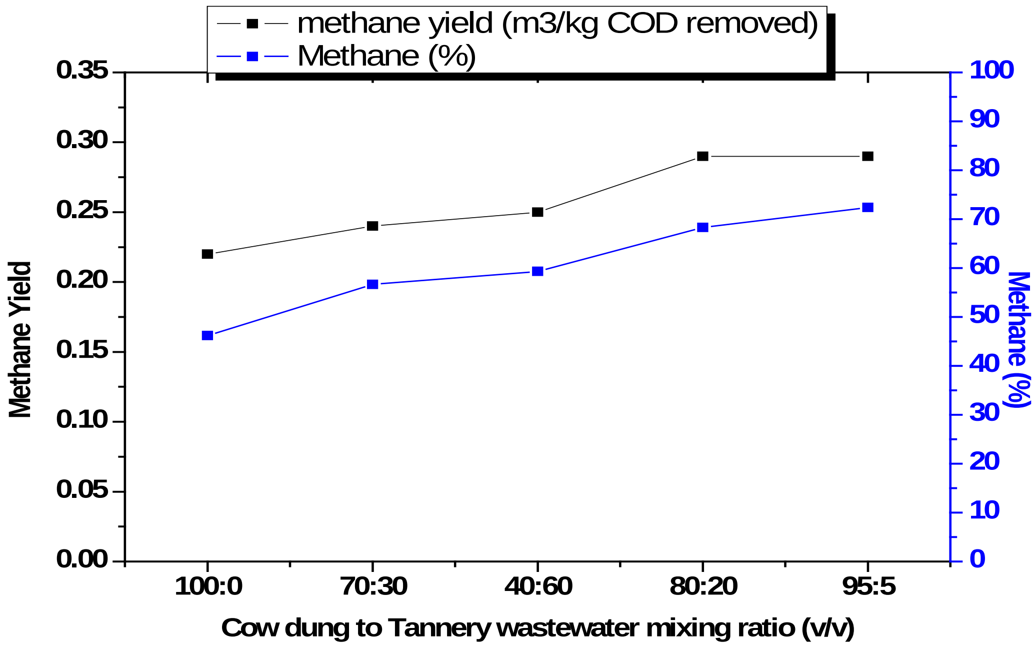

Figure 6**:** Variation of methane content and yield with mixing ratio

The analysis of variance showed that there is significant variation in both methane content and methane yield with increasing proportion of tannery wastewater ($p<0.05$). In the co-digestion of 50% cattle slurry and cheese whey wastewater, Comino *et al.* (2012) was achieved high methane yield (0.343-0.43 CH_4/kg-VS) at OLR of 2.65kgCODm^{-3}d^{-1} and HRT of 42day.

Martinez *et al.* (2009), Boubaker and Ridha (2007) and Marques *et al.* (1998) investigated the co-digestion of treatability olive oil wastewater with others substrates. Martinez *et al.* (2009) found 522l/d biogas production with high methane content from the co-digestion of olive mill wastewater mixed with piggery waste using laboratory scale anaerobic fixed bed reactor at OLR of 1.25-5 kgCODm^{-3}d^{-1} and HRT of 11-45day using laboratory scale anaerobic fixed bed reactor. Similarly, Boubaker and Ridha (2007) also achieved 31.1l per day biogas production with 67% methane and 0.29m3kgCOD$^{-1}_{removed}$ methane yield in the co-digestion of olive mill wastewater and olive mill solid waste at OLR of 0.67-6.67kgCODm^{-3}d^{-1} and HRT of 24day. In the digestion of olive mill wastewater and piggery wastewater, Marques *et al.*(1998) obtained 4.3-5.3 l per day gas production with high methane quality (67%) at 5.0 - 5.7 kgCODm^{-3}d^{-1} and HRT of 6 - 7 day.

Hublin *et al.* (2012) has also found 21.8dm3/dm3 per day biogas production in the co-digestion of 10% whey waste water and 90% cow manure. Zhang *et al.* (2011) also evaluated co-digestion of food waste and piggery wastewater. They obtained high methane yield (0.396m3/kg VS $_{added}$) and VS reduction (75.6%).

3.4. Removal efficiency of COD, TS and VS

The performance the ASBR in the removal of total solid (TS), volatile solid (VS) and COD are shown in Figure 7. The removal efficiency of TS and VS found in the various mixing ratio were in the range of 70-80% and 81-89%, respectively. The highest TS reduction (80%) was observed when only cow dung was used as feedstock while the lowest TS and VS removal efficiency (70 and 89%) was observed at substrate mixture containing 70% cow dung and 30% tannery wastewater. On the other hand, the highest VS removal efficiency was observed at a mixture consisting 20% cow dung and 80% tannery wastewater. The results of the study showed that the removal efficiency of VS was higher than TS. This indicated that the methanogenic bacteria had higher uptake rate of organic fraction of total solids (Minale and Worku, 2014).

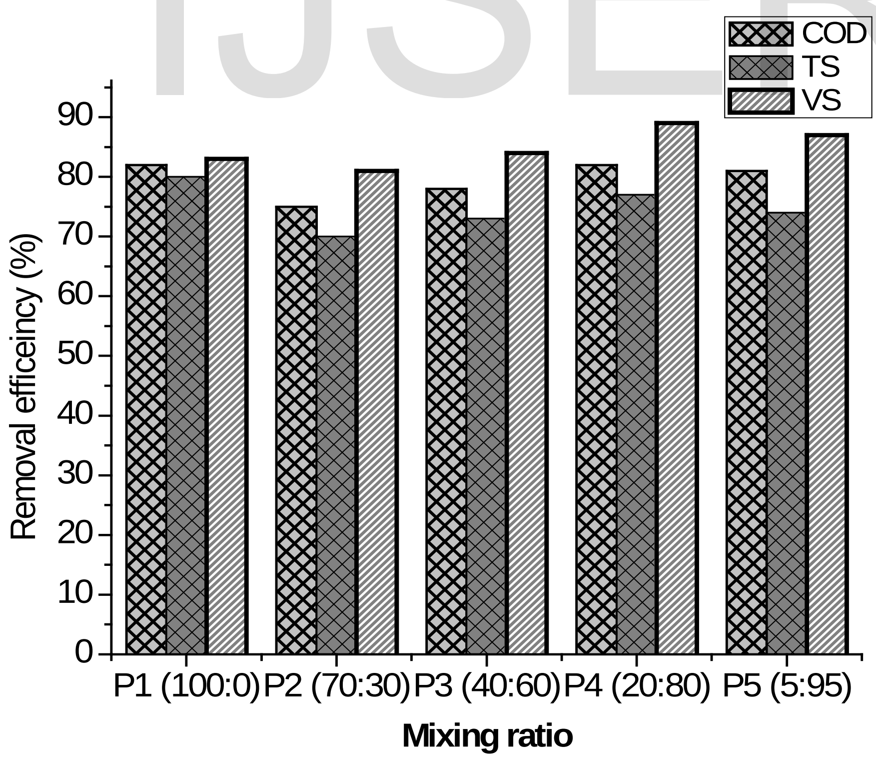

Figure 7: Variation of COD removal efficiency with mixing ratio

As it shown in the figure, the average COD removal was 80% when the digester was feed only with cow dung. On other hand, the average COD removal efficiency was reduced to 75% when the cow dung was digested with tannery wastewater in 70:30 ratios by volume. This removal efficiency was significantly lower than the removal efficiency obtained from the cow dung digestion alone ($p<0.05$). The reduction in COD removal efficiency might be due to slow acclimatization of the anaerobic bacteria for the change in composition of the substrate resulted from the addition of tannery wastewater. The COD removal efficiency was shown slight increment (78%) when the proportion of tannery wastewater was raised 60 percent by volume. The COD removal efficiency was increased to 82% when the proportion of tannery wastewater was raised to 80% by volume in the mixture substrate. It remained almost the same (81% compared to 82%) when the proportion of tannery wastewater was increased from 80 to 95% in the mixture substrate. The lowest COD removal efficiency was observed at a mixture consisting of 70% cow dung and 30% tannery wastewater by volume.

Comparable COD removal efficiency (82%) with this study was obtained in the co-digestion of 50% cattle slurry and cheese whey wastewater (Comino *et al.*, 2012). On the other hand, Hublin *et al.* (2012) found lowest COD (56.3%) and TS (32.3%) from the co-digestion of whey and cow manure. Martinez *et al.* (2009) achieved high COD removal efficiency (78-91%) in the co-digestion of olive mill wastewater mixed with piggery waste. Similarly, Boubaker and Ridha (2007) also reported high COD removal efficiency (89.7%) in the co-digestion olive mill wastewater and olive mill solid waste. Azaizeh and Jadoun (2010) have also studied the co-digestion of olive mill wastewater with swine manure using UASB reactor. They obtained high COD removal efficiency of 85-95% at a mixture containing 33% olive mill wastewater and 67% swine manure. However, Marques *et al.* (1998) achieved low the COD removal efficiency (73 - 75%) from the co-digestion of olive mill wastewater and digested piggery.

4. Conclusion

This study investigated co-digestion of tannery wastewater with cow dung and evaluated the biogas production, methane yield, COD, total solid and volatile solid removal efficiency. The results of this study showed that the removal efficiencies of COD, TS and VS increased as the proportion of tannery wastewater increased from 30% to 80%. Similarly, methane yield and methane content have also shown increasing trend until the proportion of tannery wastewater increased to 80%. The methane yield and methane content decreased from 0.3 to 0.26 m3/kg COD removed and 72 to 68 %, respectively when the proportion of tannery wastewater increased 80 to 95%. Likewise, the removal efficiencies of COD, TS and VS showed slight reduction. The co-digestion of tannery wastewater and cattle dung in mixing ratio of 80:20 enhances the quantity of methane yield and the quality of biogas. Tannery wastewater can be co-digested with cattle dung and produced biogas while the organic matter removed from the tannery wastewater.

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