

Effect of Thermo-Chemical Pretreatment of Kenyan Market Waste on Mesophilic Biogas Production

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ABSTRACT

Effects of pretreatment on the anaerobic digestion of waste fruit and vegetable market waste were investigated in biogas production by batch experiments. The pretreatment was NaOH and HCl thermochemical, thermal and chemical methods. The wastes were chopped and blended before loading to the digester. Acid hydrolysis was done by adding 20ml 0.1M HCl with thorough mixing before purging with CO₂ and sealing. Alkaline pretreatment was done using 1M NaOH. In both cases, the setups were exposed to heat at 100°C for 12hours, after which they were allowed to cool for 3 hours. The pH was modified to 6.7 – 7.2 before loading the inoculum and studying biogas generation. The large-scale setups with 1.0l, 1.5l, 5l and 10l capacity were studied for biogas generation.

The results obtained show that thermochemical pretreatment results in more cumulative biogas production at 6200ml, followed by thermal at 4900ml and then chemical pretreatments at 3750ml for 500g mixed fruits and vegetable market wastes. Alkaline pretreatment is more efficient compared to acidic hydrolysis though highly influenced by proximate properties of the wastes and operation pH. The large-scale pretreatment resulted in 34500ml and 31400ml cumulative biogas from HCl and NaOH pretreatment.

In conclusion, thermochemical pretreatment of market waste results in increased biogas generation resulting from hemicellulose breakdown and disrupting lignin-hemicellulose ether bonds in acid hydrolysis. In contrast, alkaline pretreatment leads to swelling of lignocelluloses and partial lignin solubilization lignin breakdown. The overall biogas generation depends on proximate waste matter and digester operation pH.

Keywords : *Biogas, Market waste, Thermo-chemical, Pretreatment*

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I. INTRODUCTION

The main merit of the market waste substrate is its availability. However, anaerobic digestion of these

wastes faces competition from livestock feeding or composting. In biogas generation, the plant waste substrate tends to accumulate lignin and other indigestible materials that must be regularly removed

from digesters. This severely limits the use of plant wastes in continuous-feeding digesters (Anderson, 1972). Pretreatments have the potential to improve A.D. systems considerably. Their implementation must still be guided by the specific substrate's actual improvement potential and valued in their particular context for process design and framework conditions (Carlsson *et al.*, 2012). The time taken to degrade a substrate to biogas depend on the carbohydrate bonds (Noike *et al.*, 1985).

Like sugars like glucose, simple molecules are processed into biogas by microorganisms in anaerobic digestion (Günther and Lucy, 2013).

There are two types of chemical pretreatments; Alkali pretreatment and acid hydrolysis. The building blocks and content of lignocellulosic matter make them resistant to hydrolysis. Alkali addition causes swelling of lignocellulose (Kong *et al.*, 1992) and partial lignin solubilization. Lime and sodium NaOH is the most commonly employed alkalis in feedstock pretreatment. Several studies have been published that alkali treatment is beneficial for A.D. In batch tests using rice straw pretreated with 6 percent solid NaOH for three weeks at ambient temperature, He et al. (2008) showed a substantial increase in biogas yield. Liew et al. (2011) performed simultaneous solid-state pretreatment and methanization on fallen leaves using 3.5% NaOH and showed that the methane yield increased by 20 percent during batch tests. Substrates pretreatment with alkaline solutions lead to salt build-up and increased pH during continuous fermentation and pH balancing. The high salt concentration and the effect on the ammonium-ammonia balance inhibit methanization (Chen *et al.*, 2008). Beccari *et al.* (2001) demonstrated that pH rise favours low pH and high lipid content substrates. High costs of chemicals make the pretreatment technology economically unattractive (Chang *et al.*, 1997). Acid hydrolysis pretreatment does not interfere with lignin, but is believed to function by

breaking down hemicellulose and disrupting the ether bonds of lignin-hemicellulose (Knappert *et al.*, 1981). Typically, acidic hydrolysis is combined with heat.

In thermochemical pretreatment, the influence of heat and chemical is combined. Various acids and bases have been employed though ammonia, and other solvents usage has been used. The employed temperatures range from 60 to 220°C. Methane generation decreased at temperatures ranging between 160–200°C, subject to the substrate composition (DiStefano and Ambulkar 2006; Penaud *et al.*, 1999; Delge 'nes *et al.*, 2000). Heating of substrate during alkaline pretreatment of sludge results in an increase in C.O.D. solubilization (100%) and a higher gas yield (20%) in comparison to alkali pretreatment (Kim *et al.*, 2003). In a study by Zhang et al. (2011), the influence of acidic (1.32–4.68% (w/w)) pretreatment and heat (150–170°C) on cassava substrate was investigated. They recorded a 57% gas yield increase compared to untreated cassava. Maximum biogas production was recorded at 160°C, 3% H₂SO₄ and 20 minutes' hydraulic retention time(H.R.T.) (Zhang *et al.*, 2011). In this study, the influence of thermochemical, thermal, and chemical pretreatment of market wastes using alkaline and acidic media was investigated and its influence on biogas production at mesophilic conditions.

II. Methodology

Substrate and inoculum

Fresh solid vegetable and fruits market wastes(F.V.M.W.); Cabbage (*Brassica oleracea capitata*), Coriander (*Coriandrum sativum.*),Papaya (*Carica papaya*), Spinach (*Spinacia oleracea*), Kales (*Brassica oleracea acephala*), Pumpkin Leaves (*Cucurbita maxima*), *Kahurura* (*Cucumis ficifolia*), Pig Weed (*Amaranthus spp.*), African Nightshade (*Solanum nigrum*),Mango (*Mangifera indica*), *Togotia* (*Erucastrum arabicum*), comfrey (*Symphytum*

officinale), Banana (*Musa spp*), Sweet Potato (*Ipomoea batatas*), Cucumber (*Cucumis sativus*), Watermelon (*Citrullus lanatus*), Tomato (*Lycopersicon lycopersicum*), Potato (*Solanum tuberosum*), Avocado (*Persea americana*), and Courgette (*Cucurbita pepo*) were obtained from Kangemi/Wakulima market in Nairobi County. The F.V.M.W. were subjected to size reduction using a knife before being homogenized using a blender. The inoculum used was obtained from a running digester composing of cow dung.

Waste analysis

The wastes were analyzed for proximate matter and the physicochemical properties as described in Kamau *et al.*, 2020.

Thermo-Chemical Pretreatment

The waste was blended to increase pretreatment surface area. After loading into a 250ml glass bottle, it was subjected to thermal, chemical and thermochemical pretreatment before biogas production at psychrophilic conditions. Further studies were carried out at thermo-chemical pretreated wastes.

Alkali pretreatment

Each waste was cut into small pieces before blending using a kitchen blender. The waste (200g) was then placed in a glass bottle, and 20ml 1M NaOH was added. The mixture was thoroughly shaken before purging and sealing. The set up was then placed in a water bath and maintained at 55°C for 24 hours, after which it was removed and allowed to stabilize for 6 hours. The inoculum was added, and the biogas generation was studied at 25°C for ten days. The same was done with the waste mixture (F.V.M.W.) for thermal and chemical pretreatment.

Acid hydrolysis

The market waste was then mixed with 0.1M H.C.L. (pH 1) and pre-hydrolysis allowed for 24-48 hours at

37-40 degrees with stirring. After the pretreatment step, the waste was loaded to the digester and pH adjusted to 6.8 – 7.2 using NaOH. The inoculum was added, and the mixture was purged with CO₂ to create an anaerobic environment before sealing. Cumulative biogas produced at mesophilic conditions was monitored for ten days. The setup is shown in figure 1.



Figure 1: A setup of F.V.M.W. pretreatment process

Large-scale waste pretreatment

The above procedures were repeated using 1.0, 1.5, 5 and 10 litres' digesters loaded with mixed market wastes. The setup was removed from the water bath and allowed to stabilize for 6 hours before adjusting the pH to 6.8-7.2. The inoculum was then added and mixed thoroughly. Cumulative biogas generation was studied for 17 days' retention time. The setup is shown in figure 2.



Figure 2 : Large-scale biogas production from pretreated market wastes

III. RESULTS AND DISCUSSION

The proximate analysis involves analyzing crude proteins, fibre, fat, carbohydrates, moisture, ash, nitrogen-free extract and Energy. Table 1 shows the

proximate properties of various fruit waste from Nairobi County. Table 2 shows the physical properties of the market wastes on a dry and fresh weights basis. These properties influence the pretreatment process. For example, Peces *et al.* (2015) demonstrated that substrate moisture (total solid concentration) is a significant parameter for pretreatment performance. However, it has been rarely considered in pretreatment optimization. Specifically, moisture optimization increased the methane yield of brewer's spent grain by 6% for low-temperature pretreatment (60°C) and 14% for ultrasound pretreatment (1000kJkgTS⁻¹). However, a study by Chen, Ke and Liang (2019) reported no significant difference in methane production for the three moisture contents studied during pretreatment (54%, 70%, and 77%) of the rose stalk.

Different waste pretreatment results in different biogas generation levels for similar wastes. In thermal pretreatment setups, the highest cumulative biogas obtained was 2384ml, 4126ml and 5207ml for 500ml, 1liter and 1.5liters digesters, respectively, compared to 2297ml 3139ml and 4127ml in chemical pretreatment for similar digesters. The highest cumulative biogas was reported in the thermochemical methods at 3579ml, 4888ml and 6160ml for 500ml, 1liter and 1.5liters digesters, respectively, as shown in figure 3.

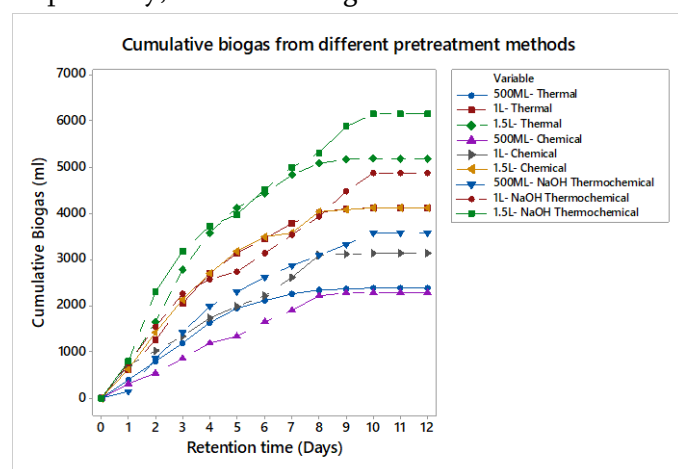


Figure 3 : Cumulative biogas produced from F.V.M.W. with varying pretreatment methods

In thermal treatment, the substrate building blocks are disintegrated by heat, thereby increasing the substrate surface area. In figure 4, acidic hydrolysis and alkaline pretreatment thermochemical methods were compared. Higher cumulative biogas production was evident in NaOH digesters compared to HCl hydrolysis at 2909ml, 422ml and 5137ml in 500ml, 1liter, and 1.5liter HCl pretreated digesters, respectively compared to 3579ml, 4888ml and 6160ml NaOH waste pretreated, respectively. The acetate groups are separated from hemicellulose in alkali pretreatment, rendering the hemicellulose more available to hydrolytic enzymes. It strengthens digestibility. The addition of alkali also induces lignocellulose swelling, which is a secondary influence (Kong *et al.*, 1992). This causes swelling, leading to an increase in internal surface area, decrease in the degree of polymerization, decrease in crystallinity, separation of structural linkages between lignin and carbohydrates, thus increasing the cellulose hydrolysis (Kleinert, 2008). Alkali pretreatment appears to be a more efficient choice for pretreatment purposes (Damisa, Ameh & Umoh, 2008). Mancini *et al.* 2018 employed different chemicals in the pretreatment of wheat straws, the organic solvent N-methylmorpholine N-oxide (N.M.M.O.) at 120C for 3 h, ii) the organosol method, employing ethanol as the organic solvent at 180C for one hour and iii) using an alkaline pretreatment with NaOH at 30C for 24 h. The study observed that the cumulative bio-methane production yield of 274 mL CH₄/g VS obtained with the untreated feedstock was enhanced by 11% by the N.M.M.O. pretreatment 15% by both the organo-solvent and alkaline pretreatment depending on the different impact on the chemical composition of the straw.

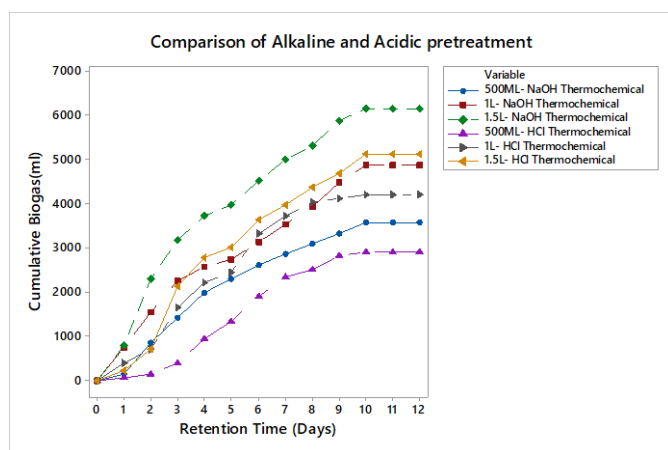


Figure 4 : Cumulative biogas generated from alkaline and acidic pretreated F.V.M.W.

On the other hand, acid pretreatment, mostly diluted acid pretreatments, increases cellulose accessibility by solubilizing hemicellulose. In figure 5, the cumulative biogas generated from the market wastes pretreated with NaOH is shown. Low cumulative biogas is recorded in spinach waste at 1069ml, while the highest was recorded in avocado fruit wastes at 4705ml. The high-fat content explains this in avocado (9.03 ± 1.36) compared to spinach (0.17 ± 0.10). In general, wastes with high fat, carbohydrates and protein content recorded higher biogas production.

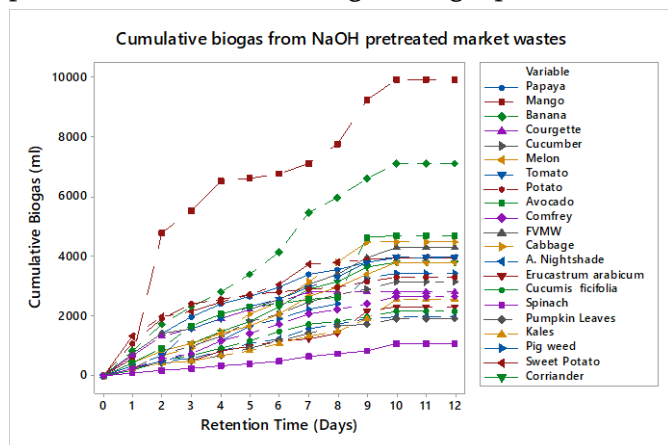


Figure 5 : Biogas generated from NaOH pretreated market wastes

The influence of the alkali pretreatment in mesophilic biogas production depends on the level of decay of the waste. A 10 -20% increase in biogas production was observed for all the wastes except for avocado,

banana, and mango, which recorded more than 40-50% biogas increment. Owing to their structure and composition, the lignocellulosic materials are hydrolysis resistant. Lignin is also partially solubilized by pretreatment with alkali, enabling cellulose and hemicellulose to be more available. Lime, K.O.H. and NaOH are the most common alkali employed in pretreatment (Monlau *et al.*, 2013; Bochmann and Montgomery, 2013). The effectiveness of alkaline pretreatment depends on the composition of the biomass as well as the pretreatment conditions. Alkali pretreatment contributes to salt build-up and increased pH during continuous fermentation. The high concentration of salt and the effects on ammonium-ammonia balance prevent methanisation (Chen *et al.*, 2008). The condition of alkali pretreatment varies depending on the type and composition of biomass used for pretreatment. The most critical parameters affecting the pretreatment of lignocellulosic biomass are the type of alkali, the concentration of alkali, biomass loading, pretreatment temperature, and pretreatment time (Raveendran, Ashok and Parameswaran, 2015).

Acid hydrolysis resulted in almost similar biogas generation levels as alkaline pretreatment. Higher production levels were witnessed in courgette and *Erucastrum arabicum* at 5490ml and 5210ml, respectively, as shown in figure 6.

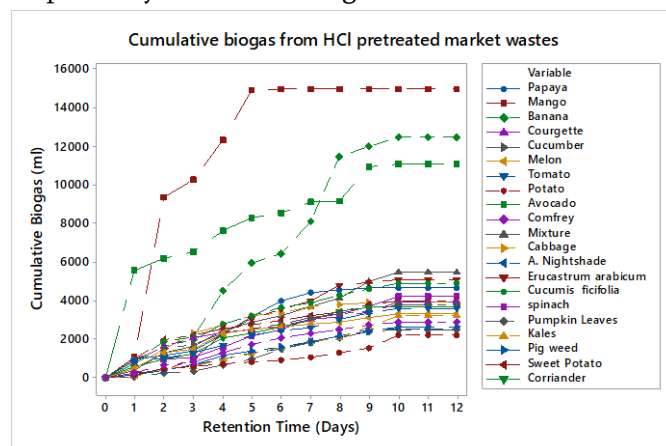


Figure 6 : Biogas generated from HCl pretreated market wastes

Sludge disintegration and cell lysis are caused by acid pretreatment, which releases intracellular organics that become more bioavailable and thus improves the rate and efficiency of the digestion method (Eskicioglu *et al.*, 2007). The H-bond, Van der Waals forces and covalent bonds in lignocellulosic matter are disrupted during pretreatment resulting in the breakdown of hemicellulose and cellulose reduction of cellulose (Li *et al.*, 2010). In a study by Devlin *et al.* (2011), WAS was digested using HCl at pH 2, 35°C and 12-day H.R.T. resulting in a 14.3% increment in CH₄ production in comparison to untreated WAS. Dilute H₂SO₄ pretreatment was used by Taherdanak *et al.* (2018) to enhance bio-methane production from the wheat plant under mesophilic anaerobic digestion. A maximum methane yield of 15.5 percent higher than that of the untreated wheat plant was obtained at 121 C after pretreatment for 120 minutes.

The influence of alkaline and acidic pretreatment of market wastes on cumulative biogas generation is comparable. Proximate properties, pH and temperature, are the significant factors that influence biogas production. This is because the waste collected is at the decomposing stage, and therefore, lignin is already disintegrating. However, depending on the nature of the waste and the decay level, pretreatment influence biogas production levels. For example, the cumulative biogas from untreated avocado, mango and banana wastes at mesophilic anaerobic digestion is 300ml, 900ml and 1500ml, respectively. Figure 7 shows that pretreating these wastes with HCl results in 11088ml, 14798ml and 12476ml in avocado, mango and banana wastes while pretreating with NaOH give 4705ml, 9922ml and 7113ml, respectively, as shown in figure 7.

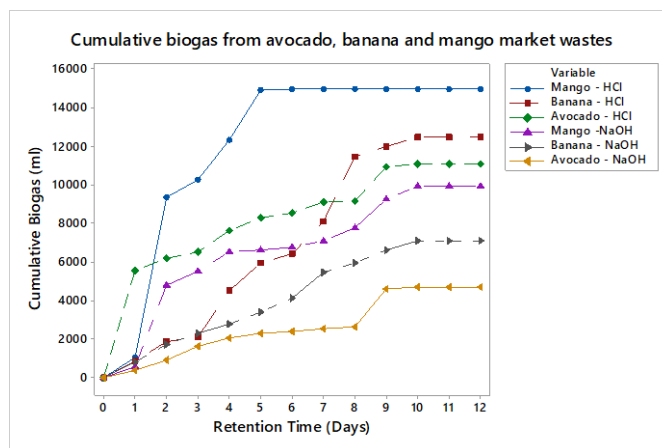


Figure 7 : Biogas generated from NaOH and HCl pretreated avocado, mango and banana wastes

The influence of acidic thermochemical pretreatment results in over 30-fold increment in biogas generation in avocado, 16-fold increment in mango and 8-fold increment in banana. The same is observed with alkaline thermochemical pretreatment with 15-fold, 11-fold and a 5-fold increase in avocado, mango, and banana.

In the large-scale studies, the influence of the amount of substrate, pretreatment chemical and retention time on cumulative biogas generation is shown in figure 8. The highest levels of biogas were generated from wastes treated with HCl at 34400ml.

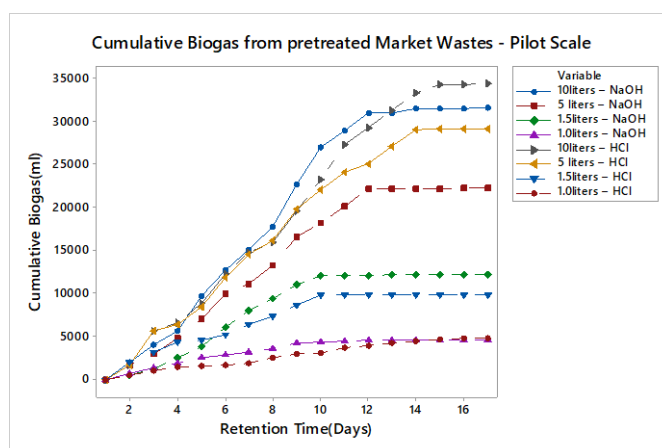


Figure 8 : Cumulative biogas produced from pretreated F.V.M.W. at large scale

IV. CONCLUSION

The thermochemical pretreatment gave the best result among three methods, followed by thermal and then chemical pretreatments with cumulative biogas generation increased by 20-57%, 17-36% and 10-33%, respectively. The effectiveness of the thermochemical pretreatment relies heavily on the proximate properties of the wastes. Alkaline pretreated litters produced more biogas on the large-scale compared to acidic pretreated wastes. Therefore, this study recommends thermochemical pretreatment of market wastes before loading into the digester for biogas production.

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Table 1: Proximate analysis on wet weight fruit and vegetable wastes

Sample	% Moisture	% Protein	% Fat	% Ash	% Fiber	% Carb.	% N.F.E.	Energy (Kcal/100g)
Kales	89.85±3.63	2.27±0.12	0.34±0.17	1.94±0.05	1.57±0.12	4.03±1.00	4.03±1.11	28.27±3.97
Cabbage	94.87±2.56	0.83±0.07	0.05±0.01	0.49±0.02	0.54±0.06	3.22±0.92	3.22±0.89	16.64±4.01
Pumkin Leaves	90.78±1.55	2.27±0.36	0.18±0.08	2.06±0.12	0.94±0.13	3.77±0.87	3.77±0.99	25.78±2.88
Cucumis ficifolia	86.62±2.98	3.49±0.72	0.33±0.11	2.34±0.05	1.48±0.52	5.74±1.02	5.74±1.04	39.89±2.37
Pigweed	88.64±2.00	2.61±0.55	0.21±0.7	2.86±0.01	2.06±0.78	3.62±0.85	3.62±0.88	26.81±7.00
Erucastrum arabicum	89.37±2.11	2.82±0.89	0.19±0.02	1.99±0.07	1.68±0.23	3.95±0.47	3.95±0.03	28.79±1.99
Coriander	92.12±4.47	2.6±0.23	0.09±0.03	1.91±0.05	1.12±0.09	2.16±0.36	2.16±0.08	19.85±1.97
A.Nightshade	88.15±1.99	2.68±0.36	0.26±0.10	1.97±0.03	2.73±0.11	4.12±0.56	4.21±1.10	29.91±1.13
Spinach	93.27±2.33	1.53±0.09	0.17±0.10	1.73±0.03	0.92±0.12	2.38±0.54	2.38±0.19	17.17±2.00
Comfrey	85.04±3.56	3.24±0.78	0.29±0.12	3.46±0.14	2.07±0.23	5.9±1.11	5.90±1.88	39.17±2.22
Tomato	95.16±4.00	0.57±0.01	0.12±0.01	0.46±0.01	0.76±0.01	2.93±0.09	15.08±1.11	2.93±0.05
Potato	83.78±4.23	1.41±0.87	0.54±0.21	0.81±0.02	1.74±0.14	11.72±1.00	57.38±6.88	11.72±0.99
Sweet Potato	62.05±2.99	1.67±0.09	1.54±0.14	1.06±0.05	1.51±0.23	32.17±2.31	149.22±20.01	32.17±2.44
Pawpaw	89.22±2.12	0.68±0.03	0.34±0.07	0.5±0.04	1.31±0.45	7.95±0.98	37.58±5.83	7.95±1.77
Banana	74.3±2.10	3.05±0.12	0.5±0.07	1.67±0.05	1.24±0.14	19.24±1.00	93.66±19.34	19.24±2.00
Avocado	82.83±3.00	1.32±0.14	9.03±1.36	0.84±0.02	2.61±0.98	3.37±0.55	100.03±12.90	3.37±1.11
Courgette	95.34±2.00	1.06±0.54	0.25±0.08	0.72±0.03	0.69±0.10	1.99±0.12	14.46±1.69	1.94±0.11
Cucumber	95.86±2.04	0.52±0.08	0.21±0.03	0.46±0.04	0.78±0.11	2.17±0.34	12.65±2.17	2.17±0.33
Mango	86.82±3.89	0.87±0.07	0.68±0.08	0.44±0.02	1.28±0.21	9.91±1.00	49.24±2.88	9.91±1.00
Water Melon	92.85±4.55	0.90±0.09	0.33±0.04	0.74±0.04	0.76±0.09	4.42±0.88	24.18±2.45	4.42±0.78

Table 2: Physical analysis properties of various market wastes

SAMPLE	% Moisture		Total Solids		% A.S.H.		%Mineral Matter		%Volatile Matter		% Fixed Solids	
	WET	DRY	WET	DRY	WET	DRY	WET	DRY	WET	DRY	WET	DRY
Kales	89.85	10.53	10.15	89.47	1.94	18.45	2.134	20.295	8.21	71.02	6.27	52.57
Cabbage	94.87	5.13	5.13	94.87	0.49	9.7	0.539	10.67	4.64	85.17	4.15	75.47
Pumkin Leaves	90.78	8.77	9.22	91.23	2.06	23.86	2.266	26.246	7.16	67.37	5.1	43.51
Cucumis ficifolia	86.62	13.38	13.38	86.62	2.34	17.52	2.574	19.272	11.04	69.1	8.7	51.58
Pigweed	88.64	11.36	11.36	88.64	2.86	25.26	3.146	27.786	8.5	63.38	5.64	38.12
Erucastrum arabicum	89.37	10.63	10.63	89.37	1.99	18.76	2.189	20.636	8.64	70.61	6.65	51.85
Coriander	92.12	7.88	7.88	92.12	1.91	24.3	2.101	26.73	5.97	67.82	4.06	43.52
A. Nightshade	88.15	11.85	11.85	88.15	1.97	16.67	2.167	18.337	9.88	71.48	7.91	54.81
Spinach	93.27	6.73	6.73	93.27	1.73	25.67	1.903	28.237	5.00	67.6	3.27	41.93
Comfrey	85.04	14.96	14.96	85.04	3.46	23.13	3.806	25.443	11.5	61.91	8.04	38.78
Tomato	95.16	4.84	4.84	95.16	0.46	9.53	0.506	10.483	4.38	85.63	3.92	76.1
Potato	83.78	16.21	16.22	83.79	0.81	5.02	0.891	5.522	15.41	78.77	14.6	73.75
Sweet Potato	62.05	37.94	37.95	62.06	1.06	2.81	1.166	3.091	36.89	59.25	35.83	56.44
Pawpaw	89.22	10.78	10.78	89.22	0.50	4.65	0.55	5.115	10.28	84.57	9.78	79.92
Banana	74.3	25.70	25.70	74.30	1.67	6.53	1.837	7.183	24.03	67.77	22.36	61.24
Avocado	82.83	17.17	17.17	82.83	0.84	4.92	0.924	5.412	16.33	77.91	15.49	72.99
Courgette	95.34	4.65	4.66	95.35	0.72	15.58	0.792	17.138	3.94	79.77	3.22	64.19
Cucumber	95.86	4.14	4.14	95.86	0.46	11.14	0.506	12.254	3.68	84.72	3.22	73.58
Mango	86.82	13.18	13.18	86.82	0.44	3.33	0.484	3.663	12.74	83.49	12.3	80.16
Water Melon	92.85	7.14	7.15	92.86	0.74	10.49	0.814	11.539	6.41	82.37	5.67	71.88