Agro-Waste from A Local South African Market: Characteristics and Co-Digestion Biogas Application

Nqobile Mkhize, Siphesihle Mangena Khumalo, Emmanuel Kweinor Tetteh and Sudesh Rathilal

Abstract**—**The demand for fruits and vegetables has increased recently, and the market is expanding because of population growth, particularly in South Africa. The increase in the production and supply of fruits and vegetables is adding to the threat to the environment by producing large amounts of agro-waste (AW) that is produced. Therefore, there is an urgent need for proper management of AW from fruits and vegetables. In addressing the challenge of AW management, the current study focuses on the production of biogas by anaerobic co-digestion of AW (i.e., banana, tomatoes, oranges, and spinach) with market wastewater (MW). AW samples were characterised for surface area, elemental analysis, functional groups, ash and moisture content, total solids, and volatile solids, and to find the potential of the substrate in the production of biogas. The characterisation resulted in a surface area of 0.92 to 1.7 m^2/g; functional groups of alkyl aryl ether, alcohols, aromatic chains, and cyclic alkene defined; ash content of 0.28 to 2%; and volatile solids of 75 to 85 %. The model AWs for the current study were found to be ideal feedstock with properties to produce biogas of 600 mL/day on average.

Keywords**—** Agro-waste, biogas, characterisation, co-digestion, fruit and vegetables, mechanical pre-treatment

I. Introduction

Agro-waste (AW) is produced in large amounts in South Africa, due to the large agricultural sector recording approximately 57% of the waste generated from human food waste [1, 2]. The management of AW in a sustainable manner is imperative to prevent the depletion of natural resources, reduce risks to human health, and minimise environmental impacts [3]. Various methodologies are presently employed for the treatment and administration of AW. In South Africa, waste management entities have applied landfilling and/or composting AW [1-3]. Landfilling of AW adds to the generation of greenhouse gases, in the release of strong odour gases and leachate during biodegradation on-site, thereby polluting local water bodies due to runoffs during rainy seasons [4].

Nqobile Mkhize, Siphesihle Mangena Khumalo, Emmanuel Kweinor Tetteh and Sudesh Rathilal, Green Engineering Research, Group; Department of Chemical Engineering, Faculty of Engineering and The Built Environment, Durban University of Technology, South Africa

The recycling and valorisation of AW materials have gained attention in the scientific community as a multidisciplinary area of study within the context of the circular economy. In circular bio-economy, AW provides a valuable source of raw material that has the potential to generate significant social and economic benefits [5]. Several proven applications of these waste materials include their utilisation as soil fertilisers or animal feed [6, 7]. Additionally, Aws are currently being employed as biomass for the production of energy or fuels [5, 7].

Anaerobic digestion (AD) can utilise the substrates from the agricultural ,industrial and domestic for their degradation for the production of biogas [8]. AD serves as a crucial technology, facilitating the simultaneous production of a renewable energy source known as biogas and a digestate, which can be utilised as a biofertilizer [9]. Co-digestion offers numerous advantages in the AD of AWs, since it provides the synergistic impact of microorganisms by reducing hazardous substances, increasing the amount of biodegradable organic matter, , and thus a higher biogas yield [3]. Another way of enhancing the AD process is the pretreatment of the substrates using chemical, biological, and mechanical technologies [10-13].

In the context of anaerobic co-digestion (Co-AD), the pre-treatment is applied to a single co-substrate, specifically the one with lower biodegradability, rather than treating the entire mixture as a whole [14]. The mechanical pre-treatment process involves the blending and/or grinding of solid particles in the AW substrates [15]. The mechanical pretreatment approach is employed for the particle size reduction and to enhance the surface area available for substrate adsorption, which leads to an improvement in the subsequent biodegradation process, ultimately resulting in enhanced biogas production [8, 16, 17]. The increase in the surface area facilitates digestion by improving the interaction between the substrate and anaerobic microorganisms, thus enhancing the anaerobic digestion process [18]. However, other researchers [19] have reported findings suggesting that no discernible impact on the kinetics of methane production.

Previous studies [20-24] have been conducted to examine

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the surface, physical, and chemical features of AW, to propose a detailed path for its valorisation as biosorbents and as raw material for biofuels and biochemicals. Reported studies in literature suggest that AW have a surface area ranging between 0.6 m^2/g and 1.7 m^2/g [20]; and particle size of greater than 0.7 mm and not more than 10 mm [19, 25].

Therefore, the present study focuses on the characterisation of the most detected AW in South African fruits and vegetables markets i.e., banana, tomatoes, oranges, and spinach to find their potential as feedstock to produce biogas via anaerobic digestion. AW characterisation was conducted using different techniques such as scanning electron microscopy (SEM), Brunauer-Emmett-Teller (BET), Fourier-transform infrared (FTIR), and calorific values. Moreover, the study focuses on the quantification of biogas generation from the co-digestion of the AW with market wastewater (MW) emanating from a local South African fruits and vegetables market using a laboratory-scale biochemical methane potential (BMP) test. The present study involved the performance of a biochemical methane potential (BMP) test under controlled mesophilic temperature conditions. The experimental setup included a uniform organic loading rate (OLR) and pH conditions. The primary goal of the study was to assess the daily biogas production (mL/Day) from various AWs.

II. Materials and Methods

A. Materials

Agro-waste sample collection

AW samples were collected from the eThekwini Municipality fresh produce market, which is the largest fruits and vegetables market in Durban, South Africa. The fresh produce market consists of two sales halls i.e., sales hall I which deals with vegetables and fruits that require cold rooms to be kept fresh, and sales hall II which deals with vegetables that do not require a cold room. Apart from the sales halls, the market has 22 cold rooms as well as 30 ripening rooms. All the waste generated from the sales halls, ripening rooms and cold rooms is discarded in a dumpsite (Fig. 1 (a)) inside the market where the waste is separated into non-organic waste (papers, plastics, etc.) which is sent for recycling and all the organic waste is sent into waste compactors as depicted in Fig. 1 (b). In the compactors, fruits and vegetable waste are compacted into bundles of waste for easy transportation.

For the current study, AW samples were collected before being sent to the compactors using a 30 L container. The targeted AW i.e., bananas, tomatoes, oranges, and spinach were manually segregated collect to 3 kg of each AW.

(a) (b)

Fig. 1: (a) Compaction area and (b) Waste dumpsite

B. Market wastewater and inoculum sample collection

Market Wastewater (MW) samples were collected from the market sump before being pumped into a local wastewater treatment facility. Activated sludge (AS) was used as inoculum for the co-digestion process and was collected from a local wastewater treatment facility in Durban.

C. Sample preparation

AW samples underwent a thorough washing process using deionized water from the water deionizer (PURELAB ELGA Option-Q, United Kingdom). This was done to effectively eliminate any dirt and contaminants that may have been present. Thereafter, the washed samples were fragmented into little segments and pulverised into fine particles using a blender (LOGIK, China). before putting the solid waste into anaerobic digestion, all solid materials need to undergo a pulverisation process which is necessary to implement a pre-treatment phase to enhance the rate of hydrolysis and degradation by microbes [26, 27]. Most of the physiochemical analyses required that the waste be subjected to oven drying (Tri lab oven, South Africa) at the temperature of 105 ℃ for 24 hours. This was done to eliminate moisture from the samples.

D. Characterisation

Fourier Transform Infrared analysis (FTIR)

The functional groups and molecular breakdown of the model bio-coagulants were found by employing a Fourier Transform Infrared (FTIR) spectrometer, (Microlam, Agilent Technologies, United States of America). The measurements were conducted at room temperature, covering a wavenumber range of 400 – 4000 cm^{-1}.

Brunauer-Emmett-Teller (BET)

The Brunauer-Emmett-Teller (BET) method (Micrometrics TriStar 11 PLUS, United States of America) was used to establish the BET surface area, pore size and pore volume of the AW samples. This characterization approach is based on the physical adsorption of inert gases, such as nitrogen, onto the solid surface of the sample.

Scanning electron microscopy (SEM)

The morphology of the AW was analysed using scanning electron microscopy techniques (JOEL JEM-2100, Japan). This analysis was conducted at the Council for Scientific and Industrial Research (CSIR), South Africa. A voltage of 200 kilovolts (kV) was employed for acceleration. The specimen was dissolved in Ethanol and subjected to sonication for approximately 20 minutes. Subsequently, they were scattered

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onto a copper grid that had been coated with a layer of carbon, in preparation for analysis.

Calorific values

An oxygen bomb calorimeter (GLOMRO BXT-ZDHW9B, China) was used for the calorific values of the waste. About 0.5g of each part was used for the analysis.

Biochemical Methane Potential Test (BMP)

A laboratory-scale batch Co-AD system was used for biogas generation. The inoculum in use was activated sludge (AS). The system consisted of 1 L bluecap Schott bottles; it is worth noting that a working volume of 800 mL was used. The operating temperature was kept constant at 40℃ for mesophilic conditions while keeping the pH at 7. The organic loading rate was kept constant at 2.5 kgs.'/m^3.day for a hydraulic retention time of 21 days. The operating conditions were predetermined after a series of system test runs and they are consistent with what has been reported in the literature [28, 29]. Table I and Table II represent the data of the characterization of the co-substrates (agro-waste and market wastewater) and inoculum using the Standard Methods for the Examination of Water and Wastewater [30]

TABLE I
SUMMARY OF CHARACTERISATION OF THE AGRO-WASTE BEFORE CO-DIGESTION

Substrate	Moisture content (%)	Ash Content (%)	Volatile Solids (%)	Total Solids (%)
Bananas	73	2	85	18
Tomatoes	90	0.28	75	8
Spinach	85	1.35	80	13
Orange	86	0.75	80	10

TABLE II
SUMMARY OF CHARACTERISATION OF THE MARKET WASTEWATER AND INOCULUM BEFORE CO-DIGESTION

Substrate	Volatile Solids (%)	Total Solids (%)	COD (mg/L)	pH
MW	12.2	16.3	2685	6.0
AS	16	20	7652	6.2

III. RESULTS AND DISCUSSION

A. Total Biogas Production

Table III presents the findings of the present study on the Co-AD of the individual model AW with MW and the inoculum (AS) over 21 days. The highest total biogas production as shown in Table III, was recorded in the order of bananas and MW (610 mL/day) > tomatoes and MW (490 mL/day) > spinach and MW (400 mL/day) > oranges and MW (380 mL/day) > MW only (350 mL/day). The addition of the AW to the MW produced higher biogas productions when compared to MW only. Th performance of bananas can be attributed to a lot of properties that were determined from this study, bananas had the highest volatile solids of 85% and a low moisture content at 73%. Bananas has the smallest surface area of 0.92 m^2/g but had biggest pores of 0.185 nm compared to the other AW. The larger pores result in an increase in porosity which enhances the biogas production process of the AW [12].

TABLE III
TOTAL BIOGAS PRODUCTION FOR THE CO-DIGESTION

Co-substrates	Total biogas production (mL/day)
Bananas and MW	610
Tomatoes and MW	490
Spinach and MW	400
Oranges and MW	380
MW only	350

B. Calorific values

The calorimetric values of the analysed substances were analysed by a bomb calorimeter., and are presented in Table IV, below. The oranges had the highest calorific value of 14.60 MJ/kg when compared to bananas and spinach with calorific values of 14.54 MJ/kg each. Tomatoes showed the lowest calorific value of approximately 14.10 MJ/kg. The significant calorific values seen in the AW samples show that these readily accessible renewable resources, which have the potential to be transformed into bio-energy products by employing different technologies. For this experiment, co-digestion of AW with MW was conducted, and the results showed a high energy content in form of the biogas production as tabulated in Table III.

TABLE IV
CALORIFIC VALUES OF THE AGRO-WASTE

Agro-Waste	Calorific Value (MJ/kg)
Bananas	14.54
Spinach	14.54
Tomatoes	14.10
Oranges	14.60

C. BET

The BET surface area, pore size and pore volume are shown in Table V, below. The BET surface area values for bananas, tomatoes, spinach, and oranges are 0.92, 1.10, 1.70, and 1.00 m^2/g, respectively. The surface area of the bananas at

0.92 m^2/g is higher than that exhibited by the analysis done by [20] of 0.65 m^2/g. All the surface areas of these AWs were low. [22-24, 31] reported that the surface areas for bananas ranged between 1.2-13 m^2/g. Oranges had a range between 2.14 m^2/g and 47.03 m^2/g [24, 32, 33]. The surface areas from the current study were observed to be low, carbonaceous materials are known to have low surface [34].

TABLE V
BET SURFACE AREA, PORE SIZE AND PORE VOLUME FOR AW

Substrate	BET surface area (m^2/g)	Pore volume (cm^3/g)	Pore size (nm)
Bananas	0.92	0.000079	0.185
Tomatoes	1.10	0.000035	0.034
Spinach	1.70	0.000041	0.065
Orange	1.00	0.000007	0.096

D.FTIR

FT-IR spectra graphs were obtained for the determination of the different functional groups existing in the different AW samples and are shown in Fig. 2. The AW had been crushed and blended mechanically into smaller pieces as a form of pretreatment before digestion. Table VI confirms the presence of alcohols, amine, phenols, esters, ethers, alkanes (CH_3 and CH_2) amino acids, alkene, aromatics and carboxylic acids. These functional groups can either enhance or limit the digestion process, depending on their quantities and this is also concluded by the findings reported in the literature [35, 36]. From the literature, it was found that there was a relationship between aromatic structures and their inhibitory impacts on methanogenic bacteria [35].

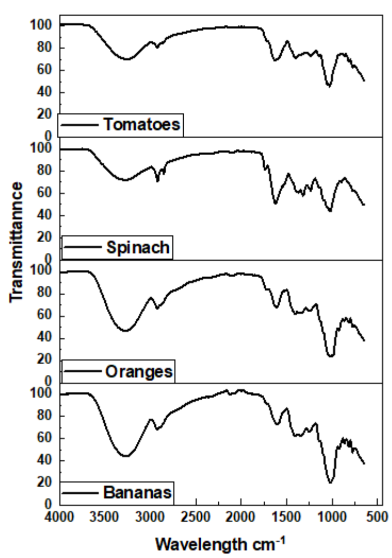

Fig. 2 Fourier Transform Infrared graphs of the agro-waste.

TABLE VI
SUMMARY OF PEAKS OF THE FT-IR GRAPHS

Observed Peaks (cm^{-1})				
Bananas	Spinach	Tomatoes	Oranges	Analysis
1021.29	1021.29	1028.75	1021.29	Primary alcohol (C-O stretch, primary amine, C-N stretch)
	1237.48	1237.48		Primary or secondary, O-H in-plane bend, phenol or tertiary alcohol (C-O stretching)
		1319.48		C-H alkane, C-H_2 bending
1341.84	1371.66		1394.02	(C-O (alcohols, esters, ethers)
1401.48		1408.93		C-H alkanes. bending of CH_3
1602.76	1625.12	1632.57	1625.12	C=C (amino acids, alkene, aromatics)
2899.87	2922.23	2922.23	2914.78	Carboxylic acids (O-H stretching)
3265.15		3242.78	3265.15	Alcohols (O-H stretching)

E. SEM

SEM micrographs provide information about the particle geometry and the structures of the mechanically pre-treated AW. Fig. 3 (a) to (d) show the images taken for the SEM analysis. For bananas as seen in Fig. 3 (a), it can be observed that the particles have pores and are rough, suggesting that particles are clumped together into larger separate clumps. Fig. 3 (b) shows an image of tomatoes with a lot of pores and an irregular shape. Spinach has a very large flat structure as can be observed in Fig. 3(c). Oranges as seen in Fig. 3(d) have particles of the same shape and size, small. All these AW have surface areas that offer a good area for substrate adsorption.

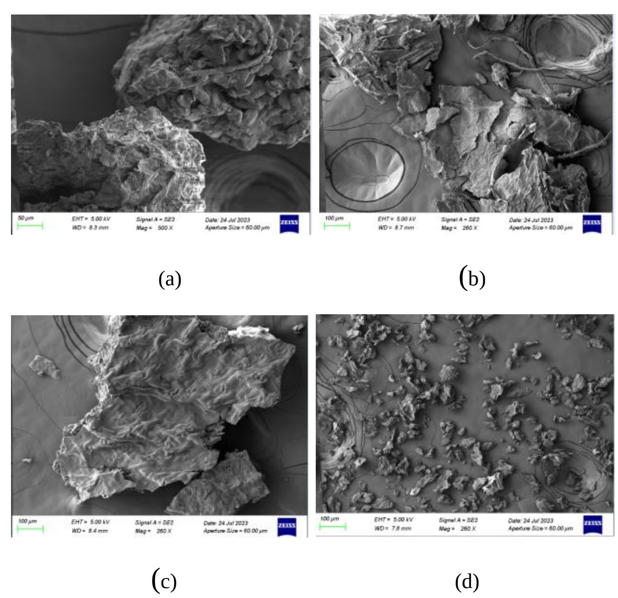

Fig. 3 SEM micrographs for (a) bananas (b) tomatoes (c) spinach (d) oranges

IV. Conclusion

An experiment to analyse the physical and chemical parameters of bananas, tomatoes, spinach and oranges from a local South African market for their suitability as feedstock in an anaerobic co-digestion process to produce biogas. The co-digestion process of the Bananas and MW produced the highest biogas production of 610 mL/day. The performance of the bananas was attributed to the high volatile solids content of 85% and having the biggest pore sizes of 0.185 nm even though they had the lowest of 0.92 m^2/g. The spinach had the highest surface area of 1.70 m^2/g. All the surface areas were found to be rather small in comparison with other studies, but the areas were still enough to provide for the substrate adsorption process during the co-digestion process. Calorific values were high for all the AW and was ranging between 14 to 15 MJ/kg. All the AWs contained aromatics as functional groups which has been reported to limit the production of biogas.

The findings of this study have the potential to contribute to the data that can be used by small-scale and industrial-scale applications of using the AW as substrates and/or co-substrates in producing biogas in the market and around South Africa. This database would be valuable for informing decisions related to the choice of energy conversion technologies and the establishment of best operating conditions.

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