

## Effect of particle size on anaerobic digestion of different feedstocks

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### Abstract

The speed and stability of anaerobic digestion depends mainly on the particle size of the input material. In this paper, particle sizes of 500 $\mu\text{m}$ , 250 $\mu\text{m}$ , 100 $\mu\text{m}$  and 25 $\mu\text{m}$  were investigated to evaluate the effects of particle size on biogas production and identify the most suitable size that would improve the efficiency of anaerobic digestion. The study was conducted using anaerobic digestion batch scale tests and gas chromatography to investigate the quality of the gas produced and the optimum particle size to maximise methane production. Prior digestion, the distributions of particle sizes of all samples were determined using single and double pass distribution techniques. 59%, 53%, 33% and 39% of CD, CM, PM and SW were recovered, respectively. In both techniques, the weight of the feed recovered reduced as the sieve became smaller. Decreasing the particle size of the feedstock significantly increased the amount of biogas. At 25 $\mu\text{m}$  particle size, 583ml, 569ml, 538ml and 398ml of methane was produced on the 4th day of gas production for CD, PM, CM and SW, respectively. At optimal particle size (25 $\mu\text{m}$ ) methane was 3 – 30 % higher as compared to that of 100 $\mu\text{m}$ , 250 $\mu\text{m}$  and 500 $\mu\text{m}$  particle size in mesophilic batch digestion tests.

*Key words: Anaerobic digestion, Mesophilic batch digestion, Microbes*

### 1. Introduction

The process of anaerobic digestion is mainly regulated by both chemical and physical characteristics of the feedstock. As such, the performance of anaerobic digestion (AD) is influenced by the composition of digester contents. It is therefore crucial to maintain appropriate conditions for proper activity of process microorganisms. The growth and activity of anaerobic microorganisms is mainly influenced by process conditions such as exclusion of oxygen, stability of temperature, pH, nutrient supply, agitation as well as presence and amount of inhibitors such as ammonia (Anonymous, 1992). Any change in these can adversely affect the biogas production. Hence, these parameters should be maintained within a desirable range to operate the biogas plant efficiently.

pH also influences the fermentation process of AD. It has been reported that pH gives an indication of chemical factors in the digester therefore, biogas fermentation require an environment with natural pH (Anonymous, 1992). Further details on that report shows that when the pH value is below 6 and above 8 the process will be inhibited or even cease to produce gas due to the toxic effect on the methanogen population. Based on these observations, the author concluded that the optimum pH value in the digester for biogas production is must be between 7 and 8.

The volatile fatty acids (VFA) to alkalinity ratio adequately characterize the digestion process; thus, the lower the ratio, the higher methane yield is attained. In a study of volatile fatty acid formation in an anaerobic hybrid reactor, Buyukkamaci & Filibeli, (2004) stated that long chain fatty acids can also constitute inhibition to anaerobic digestion process because toxicity of long chain fatty acids results from adsorption onto the cell wall or membrane, causing disorientation of essential groups on the cell membrane and thus, transport and protection function problems (Angelidaki & Ahring, 1994).

Though particle size is not that much of an important parameter as temperature or pH of the digester contents, it still has some influence on gas production. A number of studies have suggested that the efficiency of anaerobic digestion could be improved by reducing the particle size to allow more rapid reaction rates through increased exposure of the surface area of the material to the microbes responsible for the process. Chynoweth & Pullammanappalli, (1996) investigated the effects of particle size reduction and solubilisation on methane yield from food waste (FW). Their report showed that the reduction of particle size has two effects: firstly, if the substrate has a high fibre content and low degradability, combination of the substrate increases gas production; secondly, it can lead to more rapid digestion. Palmowski & Muller, (1999) did similar study and demonstrated that smaller particles increase the surface area available to the microorganisms, resulting in increased food availability to bacteria; thus, anaerobic biodegradability increases. On the other hand, other studies have questioned the benefits of size reduction and drawn attention to other important factors such as pH, temperature, carbon nitrogen ratio, volatile fatty acids and pressure. For instance, Chynoweth, et al., (1993) reported no significant digestion benefit from extensive reduction, and noted that comminution of materials may be uneconomic due to the energy input required. In unmixed 'dry' digestion systems that operate in batch or plug flow mode, a small particle size may be disadvantageous as it can lead to 'slumping' of the waste within the reactor, making it more difficult to handle (Vandevivere, et al., 2003). Similarly, Brummeler, (1999) believe/s that in batch systems that rely on percolation of liquid through the waste, a smaller particle size may also cause channelling and short-circuiting through the waste mass.

As collected, organic solid waste spans a wide range of particle sizes, and it is usually necessary to provide mechanical pre-treatment before anaerobic digestion (Igoni, et al., 2008). This gives a reduced size range compared to that in the original waste. However, the mean particle size and the difference between the ranges of particles produced will depend on the degree and type of the treatment. Most studies on particle size reduction have focussed on improving mechanical processes for the separation of waste fractions (Biala & Muller, 2001) while relatively few have considered the effect of size reduction method on the biological process (Muller, et al., 2001). This is an important consideration, however, and it is likely that the preferred particle size distribution may be a compromise between promoting the maximum biological activity and maintaining physical and biochemical stability.

The primary objective of this study was to investigate the effect of particle size on methane chicken manure (CM), pig manure (PM) and sewage waste (SW) as a feedstock for anaerobic digestion were investigated to assess their suitability and potential as biogas feed stocks.

## 2. Materials and method

### 2.1 Sample characterization

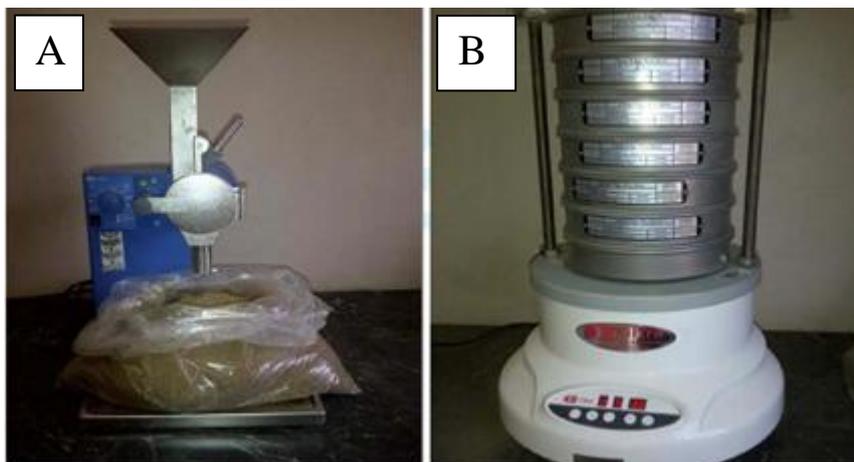
Representative samples of chicken manure, pig manure, cow dung and sewage waste obtained from white poultry at Lenasia, Elandsfontein farm at Walkersville and Moletjie farm respectively, were used. "With the exception of the sewage sample, raw materials of these substrates were obtained with high moisture content. For ease of storage and to minimize deterioration of the samples, due to fermentation, at high moisture level, the wet samples were dried at temperatures of 65 – 70 °C (Table 1) and stored at 4 °C for further experiments."

Table 1: Drying conditions for CD, PM and CM

Raw Materials	Drying Temperature [°C]	Drying Period [days]
Cow Dung	65	5
Pig Manure	70	7
Chicken Manure	65	5

Dried sewage waste was supplied by east rand water care company (ERWAT). After drying, raw materials were allowed to cool down over night and were passed through a grinding impact mill as shown in Figure 1A for size reduction. Particle distribution test were performed on each sample using a digital electromagnetic sieve shaker as shown

in Figure 1B which simply shakes the samples through sieves until the amount retained becomes more or less constant. Four different sieve sizes (500  $\mu\text{m}$ , 250  $\mu\text{m}$ , 100  $\mu\text{m}$ , and 25  $\mu\text{m}$ ) were used to group the particles based on size. These samples were later used to determine the effect of particle size on methane yield.



**Figure 1:** Grinding impact mill (A) and digital electromagnetic sieve shaker (B), respectively

## 2.2 Analytical methods

The moisture content (MC), ash content (AC) and volatile solids (VS) of CD, CM, PM and SW were analyzed in accordance with standard methods ISO589:2008, ISO1171 and ISO562 respectively. Percentage VS are given by eq. 1.

$$\%VS = \frac{m_i - m_a}{m_i} \times 100 \quad (1)$$

where  $m_i$  is the initial dry mass and  $m_a$  represent the mass of the ash after ignition.

Fixed carbon (FC) was further obtained by subtracting %MC, %VM and %AC from 100%. The heating value of the samples was also measured in accordance with standard method ASTM D3286. A representative sample of 1g was used for each test. For inherent moisture test, the samples were kept in the oven for 90 minutes at 100°C, ash content for 2 hours at 900°C while for volatile matter they were kept for 7 minutes at 600°C. The values reported are the averages of duplicate samples with relative errors <5%. Again carbon (C), nitrogen ( $\text{N}_2$ ), oxygen ( $\text{O}_2$ ), hydrogen ( $\text{H}_2$ ), sulphur (S), potassium (K), and pH of the samples were also analyzed. S,  $\text{H}_2$ ,  $\text{O}_2$ , C and  $\text{N}_2$  contents of the samples were determined using an elemental analyzer were C and  $\text{N}_2$  obtained were used to calculate the C/N ratio. FTIR tests were performed using infrared spectroscopy to identify functional groups present in the feedstock. The data collected

from this experiment was analysed using GenStat<sup>®</sup> Statistical Analysis Software12 (Genstat, 2009).

### 2.3 Bio methane potential Tests (BMP)

Furthermore, raw materials of known particle sizes of 25 $\mu$ m, 100 $\mu$ m, 250 $\mu$ m and 500 $\mu$ m were fed into 1L digester. Then, 100g of each sample was added to 800ml of water and digested at 35 °C. Biogas was collected from the bottles daily for a period of six days using a gas syringe at atmospheric pressure and the volume was measured. The volume of biogas produced was corrected to the standard temperature and pressure (STP) conditions. Methane content in biogas was analyzed using a gas chromatograph (GC) which uses helium as a carrier gas, while the temperatures of the oven, injector port and detector were maintained at 51, 80 and 300 °C, respectively. The experiment was conducted twice with two replications for each treatment arranged in a randomized block design. The data collected from both experiments and a two way ANOVA was performed using GenStat<sup>®</sup> Statistical Analysis Software12 (Genstat, 2009).

## 3. Results and Discussion

### 3.1 Proximate and ultimate analysis

The average MC, VS, FC, and ash content and calorific values of CD, CM, PM and SW samples are shown in Table 2. All the values for MC and VM are reported on dry weight basis. According to Keener, (2006), the moisture content of bio waste is an important factor for anaerobic digestion to proceed and for the final efficiency of digestion. The author further indicated that the average MC for dry feedstock is about 10 – 35%, while for a wet feedstock it is in the range of 68 – 80%. For this study, the average MC for the dry and wet samples ranged from 7 – 34% and 70 – 81%, respectively. These ranges show that the raw materials contain sufficient moisture for anaerobic digestion. Moisture content of the substrates used in this study varied significantly ( $P < 0.001$ ) as shown in Table 3. In general, CD had the highest moisture content followed by CM, while both PM and SW had approximately 75% less moisture than these CD and CM. These results show that CD has a higher potential to for better methane yields followed by PM. Although moisture content is often not considered as an important parameter for AD processes, Rockland & Beuchal, (1987) reported that no process water is added to dry anaerobic digestion (DAD) during reactor feeding, but in some systems process water is mixed into the digesting material to improve biogas production and digestion efficiency. They further reported that the core of microorganism involved in AD processes for methane production from organic matter require an aqueous environment with as water activity of  $>0.91$  for high rate hydrolysis of polymers, acidogenesis of monomers, acetogenesis of fatty acids and methanogenesis of acetate. Simulation with the anaerobic digestion models revealed mass transfer limitations due to limited hydrolysis rate for AD processes (Abbassi-Guendous, et al.,

2012). This confirms that the possibility for less methane yield in SW are higher due to limited moisture in the feedstock which is related to high dry matter contents of the substrates. Abbassi-Guendous, et al., (2012) observed slight decrease in methane production in response to an increase in dry matter content from 10% – 25%. Based on these observations it was concluded that some of the substrates need addition of some water to raise their moisture content in order to meet the requirements for successful anaerobic digestion. However, freshly collected pig manure may have higher moisture content than the one that we used, requiring minimal or no addition for further process.

Although statistically not significant, the trend showed that the substrates differ in their ash content (Table 3). While CD and PM had similar values of 10%, this parameter was doubled in CM and was more than four times in SW. A study by (Cantrel & Mahajan, 2007) indicated that wood has typical ash contents ranging from 0.5 – 2% compared to animal manures. That high ash levels in animal manures may results in less oil and gas per unit (volume or mass) of dry solids being processed. In addition, animal waste may also contain soil, which increases the ash content of the feedstock. The pig manure resulted with a high level of ash content due to their diet pigs. Compared to chicken and cows, pigs are known for their high consumptions of food (including left overs) and grass especially green ones which have high fibre content resulting in increased ash. In addition, the soil present around the pig's diet has an influence in the increased ash content compared to the CM, SW and CD. It was further observed that the average VM varied between 44 – 58%. The VS differences are believed to be related to the differences in MC, which may be attributed to the drying condition during sample collection and preparations. The calorific values in MJ/kg from bomb calorimeter were 11.892, 12.405, 17.031 and 11.555 for CD, CM, PM, and SW, respectively.

Table 2 also shows the average composition of CD, CM, PM and SW. As shown in the table, the percentage carbon obtained was 32.49, 34.70, 40.57 and 25.79 with the corresponding percentage nitrogen of 1.24, 4.27, 2.3 and 3.01 for CD, CM, PM and SW, respectively. It is necessary to maintain proper composition of the feedstock for efficient plant operation so that the C:N ratio in the feed remains within desired range. Yilmaz & Demiree, (2008) reported that during anaerobic digestion, microorganisms utilize carbon 25–30 times faster than nitrogen. Thus to meet this requirement, microbes need a 20–30:1 ratio of C to N with the largest percentage of the carbon being readily degradable. In contrast, excessive availability of N can lead to ammonia inhibition of the digestion (Richards, et al., 1991). The C/N ratios in the substrates was significantly different ( $P < 0.001$ ). The C/N for CD, CM, PM and SW were found to be 26.20, 8.13, 17.64 and 8.57, respectively. Based on the previous recommendations of optimal C/N (Yilmaz & Demiree, 2008, Deublein & Steinhauser, 2008), CD followed by PM are expected to provide higher biogas yields than CM and SW, who fall short of the minimal requirements.

Several studies have shown that pH has a significant effect on AD processes. For instance, Mashad, et al., (2008) reported that the rate of methane production declines when the pH value falls below 6.3 or becomes greater than 7.8. Other researchers documented that the favourable pH range for maximum methane yield in anaerobic digestion is 6.8 – 7.2 (Anonymous, 1992). In addition, Angelidaki & Ahring, (1994) showed that methanogenic bacteria are extremely sensitive to pH fluctuations and the preferred pH for this type of bacteria must be around 7.0 as their growth rate would be greatly reduced with pH below 6.6. The pH value of 7.3, 7.42, 7.6 and 6.6 were obtained for CD, CM, PM and SW, respectively, shows these substrates are within the optimal range of pH requirement for successful AD process.

Any instability in the AD process may lead to accumulation of volatile fatty acids (VFAs) inside the digester, which can lead to a drop of pH-value which inhibits the microorganisms (Buyukkamaci & Filibeli, 2004). However, the accumulation of VFA will not always be expressed by a drop of pH value, due to the buffer capacity of the digester, through the biomass types contained in it (Buyukkamaci & Filibeli, 2004). An average of 27.76, 53.31, 62.49 and 46 g/kg VFA were obtained from CD, CM, PM and SW. Given the need for VFAs for AD processes, it must be noted that the lower the VFA, the less methane is produced. However, extremely high VFA value in animal manure could be an inhibiting factor to microbes. This is supported by Keener, (2006) who reported that animal manure has a surplus of alkalinity, which means that the VFA accumulation should exceed a certain level before it can be detected due to significant decrease of pH due to high concentrations of VFA in the digester that would severely inhibit the AD process. Other elements such as potassium are also identified in feedstocks.

**Table 2:** Average composition of feedstock

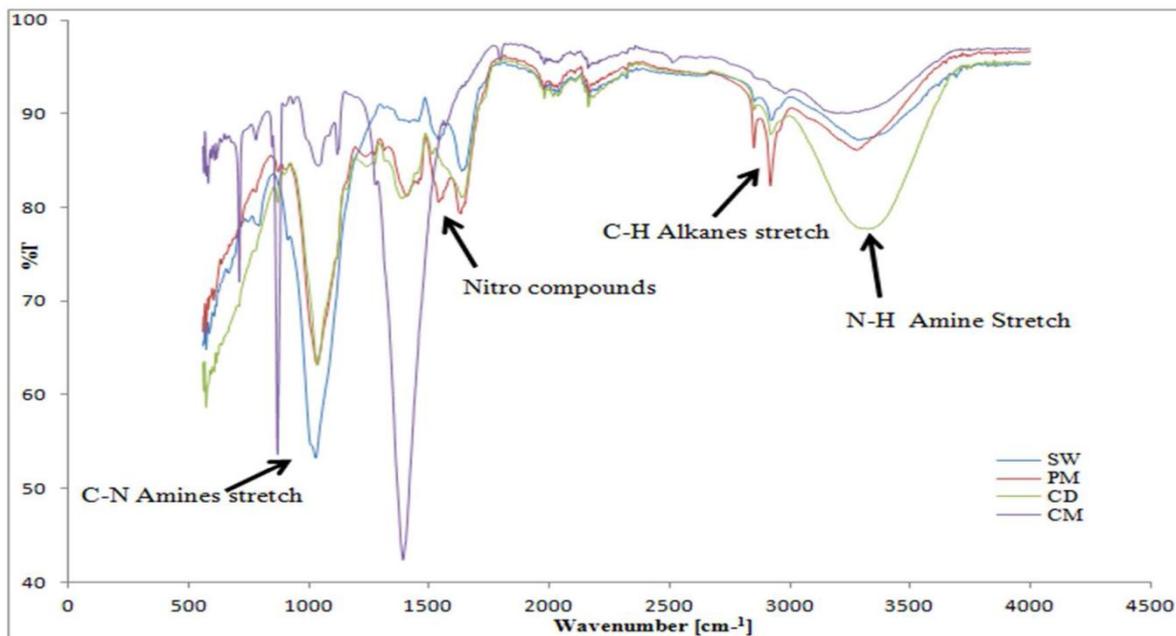
Characteristics	Unit	CD	PM	CM	SW
Moisture Content [MC]	%	33.31	7.072	28.4273	7.01
Volatile Solids [VS]	%	44.9914	58.316	51.464	44.435
Ash Content	%	10.972	20.994	9.671	43.951
Fixed Carbon [FC]	%	10.727	13.619	10.438	4.605
Calorific Value	MJ/kg	11.892	17.031	12.405	11.555
Carbon [C]	%	32.49	40.57	34.7	25.79
Hydrogen [H]	%	4.68	5.56	4.82	4.12
Sulphur [S]	%	1.03	1.44	1.38	1.72
Nitrogen [N]	%	1.24	2.3	4.27	3.01
C/N	-	26.2	17.64	8.13	8.57
pH	-	7.3	7.6	7.42	6.6
Volatile Fatty Acids [VFAs]	g/kg	27.76	62.49	53.31	46
Total potassium [K]	mg/l	38.3	106.8	105.65	23.808

**Table 3:** Statistical summary of the chemical and physical characterization of different feedstocks

Feedstock	Variables													
	MC	VS	AC	FC	CV	C	H	S	C/N	N	pH	TS	VFA	K
Chicken dung	33.31 <sup>a</sup>	78.17 <sup>a</sup>	11	10.73 <sup>b</sup>	11.89 <sup>bc</sup>	32.48 <sup>c</sup>	4.68 <sup>b</sup>	1.02 <sup>c</sup>	1.23 <sup>c</sup>	26.30 <sup>a</sup>	7.30 <sup>c</sup>	20.09 <sup>c</sup>	27.72 <sup>d</sup>	38.30 <sup>b</sup>
Cow manure	28.43 <sup>b</sup>	79.89 <sup>a</sup>	9.7	10.44 <sup>c</sup>	12.27 <sup>b</sup>	34.70 <sup>b</sup>	4.82 <sup>b</sup>	1.38 <sup>b</sup>	4.27 <sup>a</sup>	8.14 <sup>c</sup>	8.20 <sup>a</sup>	25.14 <sup>b</sup>	53.30 <sup>b</sup>	106.80 <sup>a</sup>
Pig manure	7.07 <sup>c</sup>	65.39 <sup>b</sup>	21	13.62 <sup>a</sup>	16.87 <sup>a</sup>	40.56 <sup>a</sup>	5.55 <sup>a</sup>	1.44 <sup>b</sup>	3.05 <sup>b</sup>	13.31 <sup>b</sup>	7.55 <sup>b</sup>	27.24 <sup>a</sup>	62.48 <sup>a</sup>	105.65 <sup>a</sup>
Sewage waste	7.01 <sup>c</sup>	43.57 <sup>c</sup>	22	13.62 <sup>a</sup>	11.33 <sup>c</sup>	26.79 <sup>d</sup>	4.12 <sup>c</sup>	1.72 <sup>a</sup>	3.01 <sup>b</sup>	8.91 <sup>c</sup>	6.60 <sup>d</sup>	6.21 <sup>d</sup>	45.49 <sup>c</sup>	23.80 <sup>c</sup>
F- values	<0.001	<0.001	0.479	<0.001	<0.001	<0.001	<0.001	0.002	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001

### 3.2 FTIR

Analysis of CD, CM, PM and SW were conducted using Fourier Transform Infrared Spectroscopy (FTIR) to identify functional groups present in each. Figure 2 shows specific functional groups present in CD, CM, PW and SW. The SW analysis showed noticeable fraction of NO<sub>x</sub> group which are said to be toxic compound, resulting in lower methane yields. In addition, the methanogenic bacteria cannot process the fats, proteins, and carbohydrates in pure form (Tiehm, et al., 2001). For processing they need nitro and microbic compounds which flourish in the manure and animal slurry (Gene, 1986, Cook, 1986). Therefore, high fractions of nitro compounds which are known to promote methane production are found in CD, CM and PM. Other functional groups including NH amines stretch and CN stretch are also identified in SW and PW. CH alkenes stretch is also identified in CD, CM, PM and SW which is also known as anaerobic reaction promoter.



**Figure 2:** Common functional groups identified in cow dung (CD), chicken manure (CM), pig manure (PM) and sewage waste (SW)

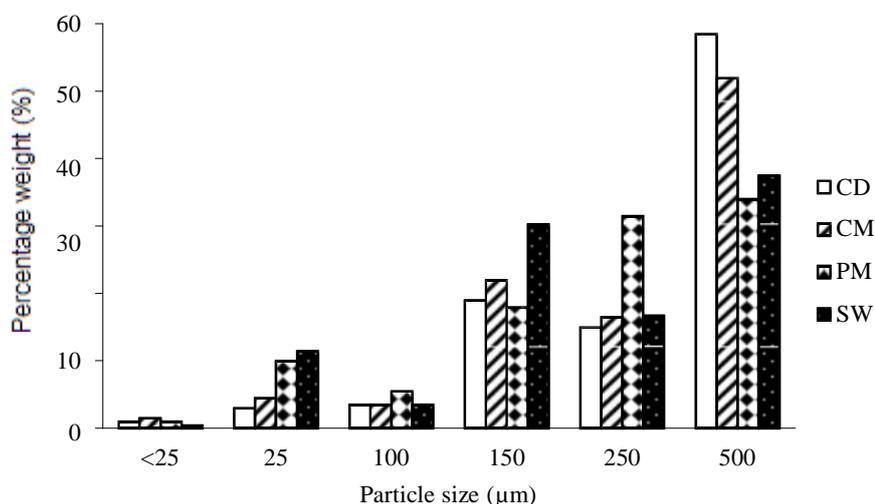
### 3.3 Particle distribution

Particle size may significantly affect the efficiency and stability of anaerobic digestion processes. It is, therefore, important to consider size of the feeding material for these processes for optimum production of gas. In this study, the distribution of particle size in different feed stocks after passing in either single or double sieving was significantly different ( $P < 0.001$ ). Figure 3 represents the weight percentages of CD, CM, PM and SW feeding materials recovered from sieve 500  $\mu\text{m}$ , 250  $\mu\text{m}$ , 150  $\mu\text{m}$ , 100  $\mu\text{m}$ , and 25  $\mu\text{m}$  sieve size through a single pass. It was observed that 59%, 53%, 33%, 39% of CD, CM, PM and SW were recovered, respectively. The weight percentages of CD, CM, PM and SW were reduced as the sieve size became smaller.

The distribution of each particle size in different feed stocks was also significantly different Table 4. Although the grinding process was uniformly applied, such differences might have been due to the physiochemical properties of each feed stock.

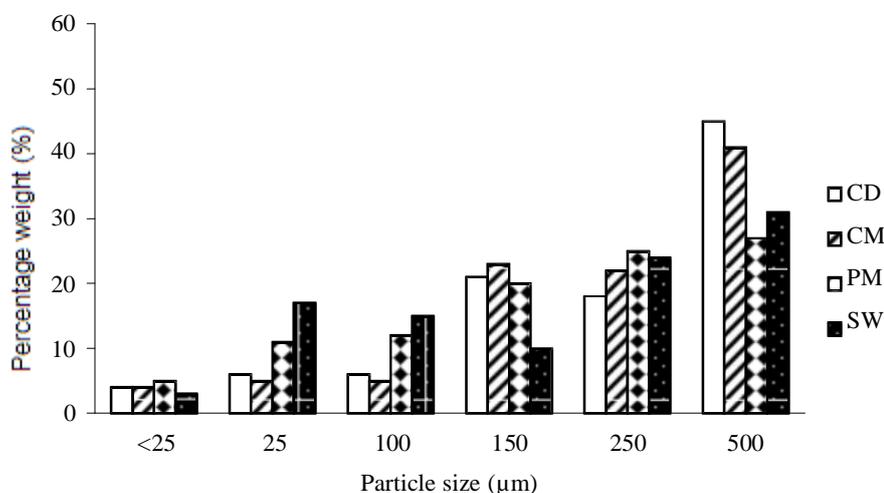
**Table 4:** Statistical summary of the distribution of particle size on different feedstock after single and double pass

Source of variation	F-probability	
	Single pass	Double pass
Particle size	<0.001	<0.001
Feed stock	1	1
Particle size * Feed stock	<0.001	<0.001



**Figure 3:** Single pass of CD, CM, PM and SW through sieve shaker

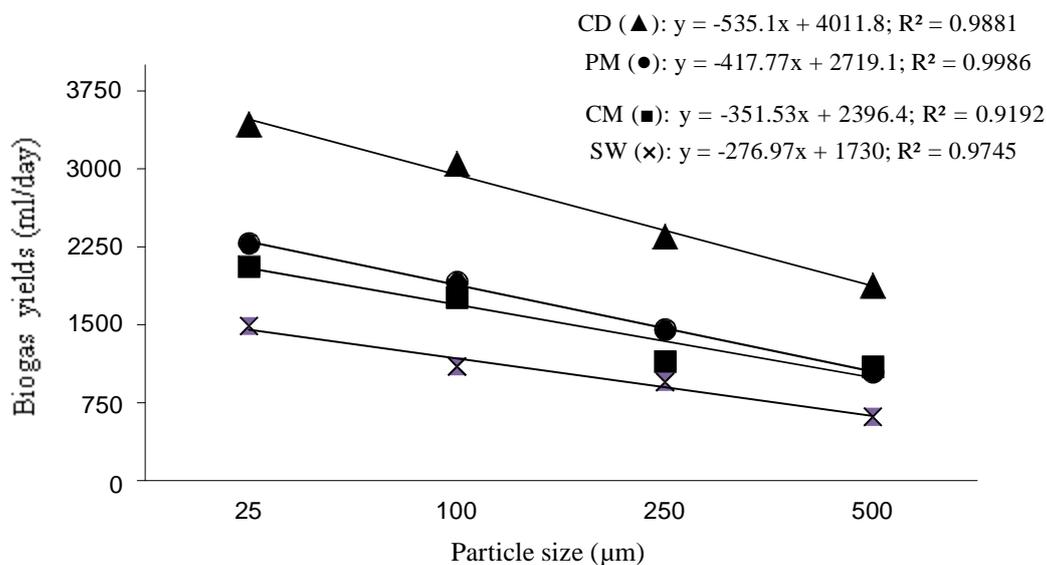
The same raw materials were further passed through a digital electromagnetic sieve shaker to determine if the duration of the raw material in the shaker will increase the distribution. A significant change in distribution was observed as shown in Figure 4. For 500 $\mu\text{m}$  sieve, the weight percentage decreased by 14%, 12%, 6% and 8% for CD, CM, PM and SW, respectively. While on the other hand, the slight increase in weight percentage recovery for 250 $\mu\text{m}$ , 150 $\mu\text{m}$ , 100 $\mu\text{m}$ , 25 $\mu\text{m}$  was observed. The difference in change in distribution for double pass process was due to the increase in time spent in the shaker.



**Figure 4:** Double pass distribution of CD, CM, PM and SW through sieve shaker

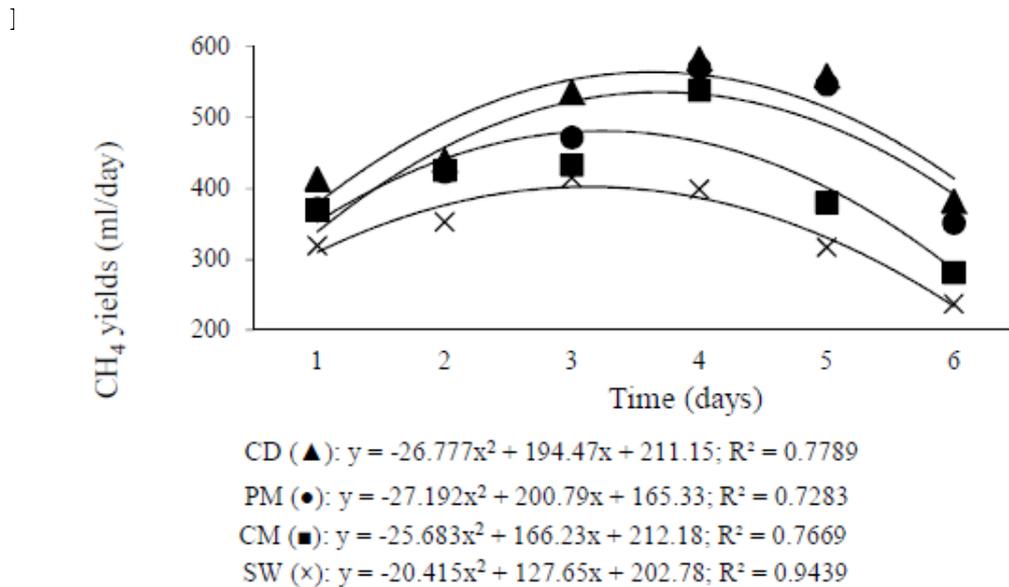
### 3.4 Effect of particle size on methane production

The amount of gas produced from CD, CM, PM and SW under the same operating condition for different particle sizes is presented in Fig 4. The average gas produced per day for 25μm particle size for the four digesters were 3425ml, 2062ml, 2285ml and 1488ml, respectively. For the 100μm particle size, the average gas produced was found to be 3047ml, 1764ml, 1912ml and 1097ml. Similarly, for 250 μm, the gas collected was found to be 1456ml, 1147ml, 2348ml, and 950ml for CD, CM, PM and SW respectively. The highest size used (i.e., 500μm) also showed 1875ml, 1096ml, 1045ml, and 614ml gas production for all representative samples. In each feedstock tested, the amount of gas produced was observed to decrease as the particle size was increased. The difference was believed to be due to the difference in the surface area of the substrate exposed to microbial. Supportive reports by several researchers (Chynoweth & Pullammanappalli, 1996) (Kayhanian & Hardy, 1994) (Sharma, et al., 1988) showed that the bigger the particle size, the less surface area is exposed to the microbes for gas production. This was confirmed by the digester with fine raw materials of 25μm showing an increase on the overall gas production by 29% to 33% compared to those with 500μm, 250μm, 150μm and 100μm.



**Figure 5:** Rate of biogas production from different feedstocks with differing sizes

The effects of particle size on methane production are attributed to the larger specific surface area provided by smaller particles for enhanced hydrolysis (Izumi, et al., 2010). The optimum particle size for anaerobic monodigestion of CD, CM, PM and SW have been examined in different ranges. In this study, the optimum particle size was concluded to be 25µm. At this size 574ml, 483ml, 552ml and 416ml methane was recovered for CD, CM, PM and SW, respectively. The amount of biogas recovered for 25µm was almost double as compared to that of 100µm, 250µm and 500µm particle size in mesophilic batch digestion tests as shown in Fig. 6. The findings of the study are comparable to those obtained by (Izumi, et al., 2010), who investigated the effects of particle size in mesophilic batch digestion tests. According to their report, a particle size of 0.718 mm yielded 28% more biogas than that with the bigger particle size of 0.888 mm. (Sharma, et al., 1988) did a similar study using agricultural and forest residues with varying particle sizes of 0.40mm, 1.0mm and 6.0mm. They observed a difference of 7% to 10% in methane production. Further study by (Kim, et al., 2000) on the effects of particle size on anaerobic thermophilic digestion in FW treatment showed that the maximum substrate utilization rate coefficient doubled with a decrease in the average particle size from 2.14 to 1.02 mm, indicating that particle size is one of the most important factors in anaerobic FW digestion.



**Figure 6:** Relationship between feedstocks and rate of methane yields

#### 4. Conclusion

The study showed that 59%, 53%, 33%, 39% of CD, CM, PM and SW were recovered respectively and the weight percentage reduced as the sieve size became smaller for single pass distribution technique. While on the other hand the slight increase in weight percentage recovery for 250 $\mu$ m, 150 $\mu$ m, 100 $\mu$ m, 25 $\mu$ m was observed for double pass technique. The difference in change in distribution for double pass process was due to the increased in the time spent in the shaker. Optimum biogas production was achieved when the particle size was reduced to 25 $\mu$ m. The effect of particle size on biogas production was directly linked to the differences in the total surface area of the feedstock exposed to microbes. The higher the total surface area, the higher the accessibility to digestion by the anaerobic microbes and hence, biogas production. Based on the results of this experiment as well as previous report, it was concluded that the efficiency of biogas production in anaerobic digestion can be significantly improved by using the smallest particle sizes of the target substrate.

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