

## Effect of Various Amendments on the Solids Properties and Gas Production of Biosolids

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### Abstract

Four additives namely iron slag (IS), works debris (WD), fly ash (FA), and lime kiln dust (LKD) are added to biosolids and their effects are investigated on the selected properties of biosolids. The biosolids used are final products of the wastewater treatment process at a Wastewater Treatment Plant (WWTP), Auckland, New Zealand. The additives are mixed manually with biosolids at different percentages. Most of the mixtures, finally called amendments has selected amount of lime in them. The amendments are placed separately into respirometer reactors (air tight bottles) for two weeks, measuring gas continuously to find out the total gas production and to analyse methane (CH<sub>4</sub>) and carbondioxide (CO<sub>2</sub>) production to completely understand the biochemical activity. Water content (WC %), volatile solids (VS %), and pH are determined before putting the amendments into the reactors and after two weeks as well. Gases that are being produced from the respirometer reactors are analysed after 5, 10 and 15 days for CH<sub>4</sub> and CO<sub>2</sub> percentages. After comparing results of all the amendments and comparing results of solids parameters to that of gas analysis, it is concluded that FA 50% with lime 20% inhibited most of the biochemical activities and maintained pH of biosolids at elevated level of 12 or above and thus could be applied to biosolids for stabilization before landfilling. FA 50% with lime 20%, like all the other additives, is added to wet biosolids on the basis of dry weight. Solid content of biosolids is around 25% so the addition of even 70% additive to wet biosolids on the basis of dry weight is very less in amount.

**Keywords:** biosolids, fly ash, gas analysis, biochemical activity.

### 1. Introduction

Due to the increase in industrialization and urbanization, there is an increase in the volume of domestic and industrial waste water being produced around the globe. Biosolids, that is end product of wastewater treatment, has increased in large amounts [1] [2]; firstly due to more amount of sewage and secondly due to strict regulations of water disposal into marine environment after treatment. Large volumes of sludge called biosolids, after dewatering, needs to be disposed of or treated, which is a challenging task to the facility owners [3]. Biosolids, besides being produced in bulk, it is also very hazardous material and its contact with environment should be prevented efficiently as it causes various environmental hazards [4] [5]. Biosolids contains rich nutrients such as nitrogen and phosphorus besides organic matter and essential trace elements. It also contains various toxins in form of heavy metals which cause harm to the environment and pose a serious risk to human health if in contact [6] [7]. Upon disposal at the landfill, various biochemical reactions start within

biosolids. Gases are the final products of the biochemical reactions within the biosolids at the landfill. Gases are critical to analyze in order to completely understand these reactions. Methane and carbondioxide are the two major and harmful landfill gases and their analysis is essential to better analyze the biochemical activities within biosolids. In landfills, a proportion of the biodegradable organic compounds are hydrolysed, acidified and subsequently methanised into the landfill gases mainly methane and carbon dioxide along with trace components [8]. This hydrolysis of organic matter can be optimized by improving the methanisation process [9]. The understanding of the ongoing biodegradation requires measuring of the total volume of gas production. The biological activity results in the release of methane gas in high amount, cracking of the surface, and a lowering of the strength. Managing these mushy biosolids is very critical so the development of techniques that reduce biosolids volume by reducing biological activity is presently increasing [10] [11]. In the present research, four alkaline additives are mixed with biosolids along with some extra lime finally called amendments to test few solids parameters and to measure and analyze gas production. These additives are iron slag (IS), works debris (WD), fly ash (FA), and lime kiln dust (LKD).

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## 2. Materials and Equipment

### 2.1. Samples and Additives Collection

Biosolids and lime are collected from WWTP in air tight 10 liters plastic containers. Iron slag or smelter slag (IS) is picked up from NZ steel from big pile lying at the side. Slag is the by-product of smelting ore to purify metals. Works debris (WD) is collected from a stockpile area from Pacific Steel. It is generated as a by-product of the steel making process at Pacific Steel where scrap steel is refined. Fly ash (FA) is obtained from Huntly Power Station. FA is the residue of the combustion of coal. It is carried up out of the boiler with the exhaust gases flow collected using electrostatic precipitators (ESP) from stack gases [18]. Lime kiln dust (LKD) is obtained from MacDonald Lime, Otorohanga. A lime kiln is a kiln used to produce quicklime by the calcination of calcium carbonate (limestone). The by-product of this reaction is LKD.

### 2.2. Supplies and Experimental Setup

SRI 8610C Gas Chromatograph (GC) is used for the gas analysis (for CH<sub>4</sub> and CO<sub>2</sub>) with the column 8ft x 1/8" OD SS, hayesep Q packing 80/100 at temperature 90 °C with Helium flowrate of 30mL/min. Thermal conductivity detector TCD was at 120 °C with the initial internal temperature inside GC is 40 °C and then the final temperature was 110 °C which was controlled by PolyScience digital temperature controller. Gas sampling syringe 1ml (1000uL) with pressure lock is from Hamilton Co., Nevada. ANR-100 Anaerobic Respirometer System shown in Figure 1 and Figure 2 consists of reaction vessels, a gas monitoring cell base unit, an interface module, temperature maintaining unit, water bath and a computer. Reaction vessels (respirometer reactors) are four in number and are 1 liter clear glass bottles KIMAX-35 500ml with airtight cap. The caps have rubber septum so that gas sampling syringe needles can pass through the caps for gas sampling and gas volume monitoring without letting gas to escape. The gas monitoring cell base unit contains four flow cells and associated interconnecting circuitry needed to pass detection signals to interface module. The cell base is located in a place at the table that it is free from movement or excessive vibrations during operation. Each notch has a photo cell and detector for bubble detection. The gas collection manifold lies between the two rows of cells. It receives exhaust gas from each cell and channels it to a common point for collection or disposal. Interface module contains the circuitry to receive signals from the cell base and it processes this information for storage by computer. Temperature maintaining unit consists of a temperature controller & monitor and a propeller constantly running to keep water flowing to maintain a constant temperature in steel water bath. Steel water bath has a closing lid to place the reaction vessels in it during experiment. Computer and monitor system process and stored data from the flow measuring cells using especially dedicated software. The software uses the Windows operating environment and MS EXCEL spread sheet program. It operates in background mode by instantaneously adding counts received from the interface module to counter registers for each flow measuring cell. This software includes set up instructions, data processing and storage instructions, user supplied information about the test units. ANR-100 data is stored in an ASCII format for subsequent processing. The computer monitor displays the test data in a convenient format including: test heading, cell number and sample name, accumulated counts, accumulated gas production volumes in milliliters, and rate of gas production in mL/min. When the respirometer is in use for gas measurement, the keyboard is used only to change the computer operation using specific keys.



Fig. 1. ANR-100 anaerobic respirometer system.

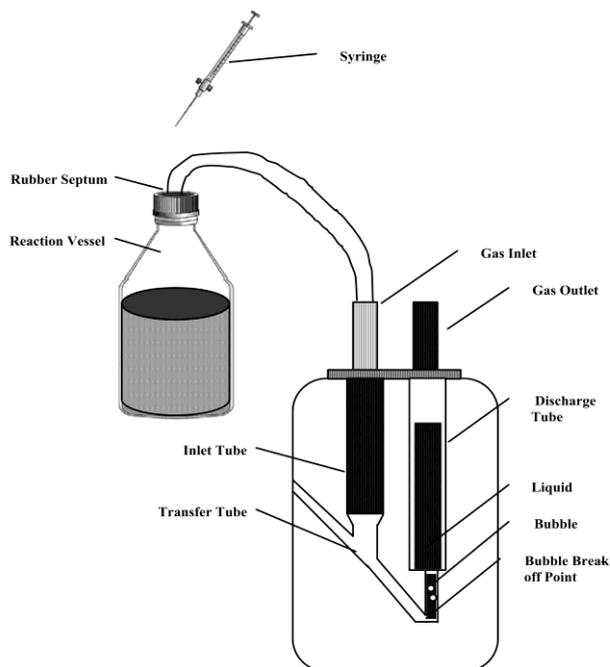


Fig. 2. Schematic of ANR-100 anaerobic respirometer system.

## 3. Methods

### 3.1. Measurement of total gas production via respirometer reactors

Respirometer reactors are glass bottles with airtight caps having rubber septum for the insertion of measuring needle. The reaction vessels are filled upto 500ml with biosolids as per plan shown in table 1 respectively. Percentages of all the amendments shown in table are added to biosolids on the basis of dry weight. There are four sets of experiments and each set lasted for 2 weeks. In first set, LKD with biosolids in the respirometer reactors is tested for two weeks. Raw biosolids, LKD 30% Lime 0%, LKD 30% Lime 20% and LKD 50% Lime 20% are in reactors 1, 2, 3 and 4 respectively. After two weeks, all the amendments are removed from reactors and are tested for their solids properties. Then reactors are washed, cleaned and dried before starting of the next test. In the second test FA with biosolids in the reactors is tested such that FA 50% Lime 0%, FA 50% Lime 20%, FA 30% Lime 0%, and FA 30% Lime 20% are in reactors 1, 2, 3 and 4 respectively. Similarly, IS and WD are tested in third and fourth sets respectively. The biosolids without amendments are weighed equally prior to addition of additives and lime in them such that equal amounts of basic biosolids are in all the reaction vessels in all four sets of experiments with additives on top of them.

**Table 1. The respirometer plan for the analysis of CH<sub>4</sub> and CO<sub>2</sub> and solids properties.**

Additives	Reactor 1	Reactor 2	Reactor 3	Reactor 4
LKD	Raw	LKD 30%	LKD 30%	LKD 50%
	Biosolids	Lime 0%	Lime 20%	Lime 20%
FA	FA 50%	FA 50%	FA 30%	FA 30%
	Lime 0%	Lime 20%	Lime 0%	Lime 20%
IS	IS 50%	IS 50%	IS 30%	IS 30%
	Lime 0%	Lime 20%	Lime 0%	Lime 20%
WD	WD 50%	WD 50%	WD 30%	WD 30%
	Lime 0%	Lime 20%	Lime 0%	Lime 20%

### 3.2. Gas Analysis

Gases that are produced from the respirometer set up are analyzed after 5, 10 and 15 days for CH<sub>4</sub> and CO<sub>2</sub> percentages. Carbon dioxide and methane, both of the gases are detected in a single sample. The GC is turned on according to its instructions and the three gases air, hydrogen and nitrogen are turned on and allowed to pass and warmed up through the GC before analysis. GC set up is calibrated every testing day with 100% CH<sub>4</sub> standard then again with 60% CH<sub>4</sub> and 40% CO<sub>2</sub> standard then calibration curve is prepared into the GC software each day using the same standard. Gas samples from respirometer reaction vessels are taken by using airtight 1ml syringe as shown in the figure 2 then the needle is locked using pressure lock so that the gas could not escape. The needle is then directly inserted in the sampling port of the SRI GC then the samples are then injected in SRI GC for methane and carbondioxide analysis. The retention time for methane is between 90 seconds to 120 seconds (2 minutes). Total retention time for methane and carbondioxide is approximately 4 minutes. The curve is formed in the system during gas analysis and shows methane and carbondioxide percentage, area under the curve, required peak and other statistics.

### 3.3. Solids Parameters Analysis

The biosolids samples in the respirometer reactors are analysed for pH, water content (WC) and volatile solids (VS) prior to placing the samples in the reactors and also at the end of the experiments i.e. after two weeks. These parameters are analyzed to completely understand the gas production process due to biochemical activities. The pH of biosolids is measured in solution using a pH meter [13]. Solution is prepared by stirring biosolids in deionised water using a magnetic stirrer. Water content (WC) is measured by oven drying method at 105 °C overnight to constant weight (Gravimetric Analysis - [13]). Volatile solids (VS) are measured by igniting samples in furnace at 550 °C for 30 minutes (loss on ignition).

## 4. Results and Discussion

### 4.1. Gas Production

Gas production results are shown in figure 3. Raw biosolids do not have the highest gas production but looking at gas analysis, it shows that it has maximum biochemical activity due to maximum CH<sub>4</sub> percentage. Biosolids with 30% additive without lime has highest gas production with respect to all amendments except LKD. This provides evidence that, when there is no lime, maximum biochemical activity is present. The gas production is higher than that of raw biosolids, this shows that when there is any additive added to the biosolids, there are some other reactions occur generating some gases. Looking at LKD 30% gas production, it shows that there is not much gas production when LKD reacts with biosolids. But considering gas analysis results it shows that LKD 30% has higher methane percentage than most of the other amendments. Here lesser gas production shows that, some other gases are not producing besides CH<sub>4</sub> and CO<sub>2</sub> (e.g H<sub>2</sub> & NH<sub>3</sub>). But in other amendments, there are other gases producing besides CH<sub>4</sub> and CO<sub>2</sub>. The amendments with 30% and 50% additive with 20% lime have overlapping total gas production and gas production is lesser than unlimed samples. Total gas volume of IS 30% is highest as compared to other amendments' gas production. It is even higher than the gas production of raw biosolids. This tells that besides regular biochemical activity in biosolids, there are some other reactions going on with this amendment. Based on gas analysis results, it also explains that IS 30% allows maximum biochemical activity to occur. When an amendment is added to biosolids, gas volume is more due to other gases besides CH<sub>4</sub> and CO<sub>2</sub> (except with LKD). But when lime is added with the additive, it inhibits biochemical activity, so gas volume is lesser than the additive alone with biosolids.

### 4.2. CH<sub>4</sub> and CO<sub>2</sub> analysis

Raw biosolids manifests highest methane production with time [14]. Methane is increasing with time and CO<sub>2</sub> is decreasing with time in raw biosolids as shown in table 2. Considering the additive LKD and its amendments, maximum methane production is with LKD 30% without lime. Maximum CO<sub>2</sub> production is with LKD 50%, lime 20%. CO<sub>2</sub> is very high in limed samples as compared to unlimed ones. This tells that, CO<sub>2</sub> is more produced due to lime addition. CH<sub>4</sub> is higher in unlimed samples as compared to limed ones. This reveals that lime in addition with the additive, inhibits CH<sub>4</sub> production and thus biochemical activity. Considering the additive FA and its amendments, maximum methane production is with FA 50% without lime. Maximum CO<sub>2</sub> production is with FA 50%, lime 20% and with FA 30%, lime 20%. CO<sub>2</sub> is very high in limed samples as compared to unlimed ones. This divulges that, CO<sub>2</sub> is more produced due to lime addition. CH<sub>4</sub> is higher in unlimed samples as compared to limed ones. This tells that lime in addition with the additive, inhibits CH<sub>4</sub> production and thus biochemical activity. Considering the additive IS and its amendments, maximum methane production is with IS 30% without lime. Maximum CO<sub>2</sub> production is with IS 50%, lime 20% and IS 30%, lime 20%. CO<sub>2</sub> is very high in limed samples as compared to unlimed ones. This shows that, CO<sub>2</sub> is more produced due to lime addition. CH<sub>4</sub> is higher in unlimed samples as compared to limed ones which tells that lime in addition with the additive, inhibits CH<sub>4</sub> production and thus biochemical activity. Considering the additive WD and its amendments, WD demonstrates same trend as IS that maximum methane production is with WD 30% without lime. Maximum percentage of methane is with raw biosolids but

with respect to all amendments, the maximum percentage of methane is with IS 30% without lime. This attests that IS 30% without lime allows maximum biochemical activity to occur. Minimum percentage of methane is with FA 50%, lime 20%. It can be concluded that FA 50% with lime 20% inhibits most of the biochemical activities. CO<sub>2</sub> percentage is high in sample with FA 50%, lime 20%. But as in the case of all amendments with lime, CO<sub>2</sub> is likely to be produced more due to lime addition. Methane is also produced through carbon dioxide reduction[15].

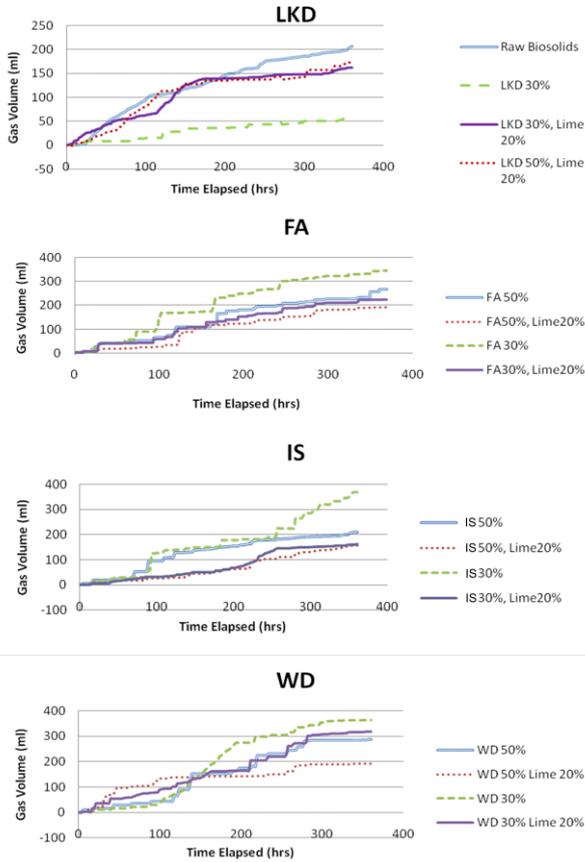


Fig. 3. Total gas production in respirometer reactors.

4.3. Changes in pH, WC % and VS %

The results as shown in table 3 indicate that all pH, WC % and VS % along with gas production are affected by the addition of different additives. pH, WC% and VS% are measured before putting the amendments into the respirometer reactors and after aborting the reactions at 2 weeks. pH analysis is not much useful itself but changes in pH affect other biochemical parameters of biosolids [16]. The pH of all the samples is decreased after 2 weeks of experiment due to acid production. Comparing initial and 2 weeks data after completion of respirometer tests, it validates that pH of biosolids with FA 50% and lime 20% has least change after two weeks and it is maximum after two weeks as compared to other amendments results i.e. 12.3. WC% of FA50% and lime 20% is least among all the amendments after 2 weeks which is 183.5%. The result also shows that maximum inhibition of biochemical activities is with this amendment. The higher the WC%, higher is the biochemical activity and lower the WC% lower the biochemical activity. WC% results also support that the amendment FA 50% with lime 20% is better than other amendments. VS% results completely go to the same direction

of supporting the above mentioned selected amendment as VS% of FA 50% and lime 20% after 2 weeks is least i.e. 38.7%. A low VS% means that there is less biochemical activity taking place with this amendment as compared to other amendments. All these results manifest that FA along with lime does not allow biochemical activity easily as compared to other amendments.

Table 2. Gas analysis results in respirometer reactors.

Set	Days	Raw Biosolids		LKD30%, Lime0%		LKD 30%, Lime 20%		LKD 50%, Lime20%	
		CH <sub>4</sub> %	CO <sub>2</sub> %						
Set 1	5	10	33	1	5	1	0.3	0	0
	10	25	24	8	8	7	2	8	1
	15	54	13	13	10	5	27	5	29
Set 2	Days	FA50%, Lime0%		FA50%, Lime20%		FA 30%, Lime 0		FA 30%, Lime20%	
		CH <sub>4</sub> %	CO <sub>2</sub> %						
	5	10	0	12	10	11	0	10	10
10	12	1	5	30	13	2	8	12	
15	12	1	4	33	11	3	7	33	
Set 3	Days	ISS0%, Lime0%		IS50%, Lime20%		IS30%, Lime 0%		IS30%, Lime20%	
		CH <sub>4</sub> %	CO <sub>2</sub> %						
	5	5	10	6	9	5	10	9	12
10	15	8	10	14	20	19	15	13	
15	26	8	10	32	30	8	16	32	
Set 4	Days	WD50%, Lime0%		WD50%, Lime20%		WD30%, Lime 0%		WD30%, Lime20%	
		CH <sub>4</sub> %	CO <sub>2</sub> %						
	5	7	5	7	10	8	4	9	11
10	11	8	10	15	10	11	10	13	
15	17	12	12	17	16	13	16	22	

5. Conclusions

Gases from the respirometer reactors are analysed for methane (CH<sub>4</sub>) and carbondioxide (CO<sub>2</sub>) as well as biosolids properties are also analysed to predict the behavior of biosolids with amendments. The amendment of biosolids with FA 50% and lime 20% illustrates least concentrations of methane throughout the experiment and at the end methane is not much produced as compared to other amendments results. FA shows high potential for the inhibition of biochemical activities within biosolids, especially when lime is added to biosolids with FA [17]. FA 50% + L 20% on the basis of dry weight of biosolids is added to wet biosolids and is found to be the most promising amendment that minimizes biochemical activities more as compared to other amendments [19]. FA resists biochemical activities within the biosolids to occur due to the presence of silicon dioxide (SiO<sub>2</sub>) and calcium oxide (CaO) in it. FA also contains non-reactive coal that allows minimum biochemical activity. When looking at the pH, WC% and VS% results from the respirometer reactors after the completion of tests, they also explain minimum bioactivity in the particular amendment as compared to other amendments. According to gas analysis results it can be concluded that FA 50% and lime 20% is the best amendment for the stabilization of biosolids. Co-disposal of FA with biosolids while FA being helpful for the inhibition of biochemical activities within biosolids would also address the disposal of FA. FA is a residue of coal combustion plants and is a harmful matter as its disposal is another environmental issue itself [20] [21].

**Table 3. Changes in pH, WC% and VS% after 2 weeks in the respirometer reactors.**

Amendments	1. pH		2. WC % (dry weight)		3. VS % (dry weight)	
	0 wk	2 wks	0 wk	2 wks	0 wk	2 wks
Raw Biosolids	8.2	8	330.3	349	68.3	67
30% IS	10.5	9.7	239.9	278	50.1	53
30% IS and 20% lime	12.6	10.4	208.6	208.5	39.8	40.6
50% IS	12	10.3	212.5	236	44.1	47
50% IS and 20% lime	12.6	10.6	186	184	35.6	39.7
30% LKD and no lime	12.1	11	246.8	251.4	50.8	50.1
30% LKD and 20% lime	12.6	12.2	207	210.5	37.5	39.1
50% LKD and 20% lime	12.6	12.2	185	184.1	32.7	33.9
30% FA no lime	8.9	9.2	261.7	258.8	52.5	49
30% FA and 20% lime	12.4	12.0	212.2	213	41.5	42.1
50% FA no lime	9.2	10	226.3	223.2	45.2	46.8
50% FA and 20% lime	12.4	12.3	184.4	183.5	37.1	38.7
30% WD and no lime	10.4	9.6	250.4	251.7	45.1	51.2
30% WD and 20% lime	12.4	10.7	210.8	210	41.7	42.3
50% WD and no lime	10.5	10.1	219.4	217.6	43.9	43.7
50% WD and 20% lime	12.4	11.1	187.2	206.6	37	41.2

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