VFA AND AMMONIA FROM RESIDENTIAL FOOD WASTE AS INDICATORS OF

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Abstract

2	Research was conducted to determine suitable chemical parameters as indicators of odor from
3	decomposing food wastes. Prepared food scraps were stored in 18 L plastic buckets (2 kg wet
4	weight each) at 20°C and 8°C to reproduce high and low temperature conditions. After 1, 3, 7,
5	10 and 14 days of storage, the odor from the buckets were marked to an intensity scale of 0 (no
6	odor) to 5 (intense) and the corresponding leachate analysed for volatile fatty acids, ammonia
7	and total organic carbon. A linear relationship between odor intensity and the measured
8	parameter indicates a suitable odor indicator. Odor intensified with longer storage period and
9	warmer surroundings. The study found ammonia and isovaleric acid to be promising odor
10	indicators. For this food waste mixture, offensive odours were emitted if the ammonia and

Keywords: isovaleric acid, OFMSW, odor management, kitchen waste, odor indicator parameters.

isovaleric acid contents exceeded 360 mg/l and 940 mg/l, respectively.

1. Introduction

diversion program.

The separate collection and transportation of food residues from residential waste saves landfill capacity, but presents with it the issue of malodor (Qamaruz-Zaman and Milke, 2008). The coexistence of waste treatment facilities (e.g. composting or anaerobic digestion plant) with people, particularly in urban areas, can hardly be avoided due to the consideration of transportation costs and available land. This leads to odor concerns at waste treatment facilities, and their close management. Similarly, there is a need for an understanding regarding the degree of odor from food waste stored in household bins in order to ensure a successful organic waste

Food residues generated from domestic kitchens include fruit, vegetables (including peelings), meat and bones, bread, fish bones, pasta, shellfish, rice, eggshells, coffee grounds, dairy products and table scraps (Steuteville and Karen, 1996; Gies, 1996; Shin et. al., 2000; Farrell, 2001; Viana and Schulz, 2003). These materials when decomposed may emit odor of varying intensities (Kim et al., 2009), likely to be influenced by the storage conditions, as well as by the combination of materials in the waste. For example, a high amount of meat may produce an objectionable odor that could worsen with the presence of liquid, for example gravy or stew leftovers.

The managers of organic diversion programs are faced with a number of choices in terms of materials for residents, collection equipment, and advice to residents. Bad odors from source-separated food wastes can lead to reluctance of residents to participate. Therefore, waste managers often need quick ways of evaluating whether changes to a program would increase or decrease odor problems, and also need ways to evaluate if problems are due to specific wastes or due to the segregation practices employed in individual residences. Sensitive analytical chemistry methods can be used to quantify the concentrations of individual odorous gases (Sironi et al.,

1 2007), and olfactometry methods can use a panel of invididuals to assess odor subjectively (EN

13725, 2003). Many efforts have been made to associate human sensing of odor with the

3 concentration levels of odorants measured by advanced analytical instruments(e.g., the

combination of gas chromatography with mass spectrometry) (Nagata, 2003; Kim, 2010; Kim,

5 2011).

Simple and cost-effective chemical indicators that quantify the intensity of odor emission could provide an alternative to more sophisticated and expensive methods. *E. Coli* is used as an indicator or surrogate of pathological organisms in water because *E. Coli* is commonly present when pathogens are present, and *E. coli* is easier to measure than pathogens are. For odor, the measurement of concentrations of one or more key compounds, which reflect the degradation of the main waste constituents, can be used as indicators in an analogous way to *E. coli* as a surrogate or indicator to monitor odor development and/or the determination of odor intensity. It has been suggested by Spoelstra (1980) that for good indicators: (i) the components must be products of protein (or possibly carbohydrate) degradation, (ii) the formation of the components must reflect kinetics of degradation, (iii) the components must respond in a representative way to environmental changes e.g. aeration, methane formation, and (iv) the concentrations must be suitably large for easy measurements; trace components are not suitable. The selection of

A wide variety of odorants are constantly being relased from the environment including acetaldehyde, isovaleric acid, dimethyl sulfide, buytlraldehyde, butyric acid, valeric acid and ammonia from lake sediments (Susaya et al., 2011). Ammonia is commonly high in fishery

indicators also depends on speed of analysis and the availability of equipment (Williams,

1984). With odor indicators, results can be obtained where no odor panel is available and

retrospective analysis of appropriate data can indicate the effectiveness of treatments that were

designed for other purposes (Williams, 1984).

1	wastes (Seo et al., 2011), and has also been reported in stormwater catch basin waste (Rabii et
2	al., 2010).
3	
4	Residential food waste because of its wet nature, high organic content, and mixture of chemical
5	substances, quickly produces odors as it begins to decompose. Volatile fatty acids (VFAs) and
6	ammonia can be expected to result from the decomposition of organic matter containing
7	carbohydrates and protein. Ammonia is produced from either aerobic or anaerobic decomposition
8	of proteins and amino acids (Pagans et al., 2007). VFAs are a product of fermentative or
9	anaerobic degradation of a wide variety of complex organic compounds (Nielsen et al., 2007).
10	Carbohydrates are degraded to a limited number of VFAs (mainly acetic, propionic and butyric
11	acids), while proteins are broken down to straight chain fatty acids, and fats and oils are broken
12	down to long chain fatty acids. The longer chained acids can be hydrolyzed or biodegraded to
13	lower molecular weight acids such as acetic, propionic and isovaleric (Lyberatos and Skiadas,
14	1999).
15	
16	This research was conducted to investigate the use of odor indicators to quantify odor emission
17	from food waste kept at two different temperature conditions. Three indicators total organic
18	carbon (TOC), volatile fatty acids (VFAs) and ammonia (NH ₃) are analysed to demonstrate
19	their suitability in odor quantification. The TOC would indicate the amount of organics present in
20	the sample whereas the latter two are chosen based on the assumption that food waste

decomposition can occur, via an aerobic or anaerobic pathway, releasing VFA or NH_3 .

2. Methodology

2.1 Experimental Procedure

1 To minimise the effect of feedstock variation on odor production, a standard food waste mixture 2 as shown in Table 1 was used throughout the study. The food waste model is based on a previous 3 study (Qamaruz-Zaman and Milke, 2008) and consisted of vegetables (34% wet weight basis), 4 fruit (19%), meat (12%) and small amounts of coffee grounds, filters, teabags, rice, leftover 5 spaghetti, eggshells and bread. The waste (2 kg wet weight) was discarded into 18 L plastic 6 buckets and stored, lidded, for 14 days in temperature controlled rooms of 20°C and 8°C. 7 8 A total of five buckets were kept at each temperature conditions. After 1, 3, 7, 10 and 14 days of 9 storage, one bucket from each group was evaluated. Because the buckets were emptied during 10 evaluation, fewer replicates remained in storage as time progressed. The evaluation of buckets 11 followed the following procedure; first, buckets were weighed, then scored for their subjective odor intensity, followed by leachate analysis and finally pictures of the decomposed wastes were 12 13 taken. 14 15 2.2 Odour evaluation 16 Two individuals were used to evaluate odor. Because the waste decompostion occured in the 17 bucket, the odor volatilized within the headspace of the container. As such, odor assessment was 18 done directly on the bucket without gas sampling. The smell was marked according to the odour 19 intensity scale depicted in Table 2.3. A mark was assigned to the characteristic smell of that 20 bucket, based upon a range between 0 (no odour) to 5 (intense odor). 21 22 During the odour testing, oneassesor conducted a preliminary scaled assessment of each bucktet. 23 This judgement was then confirmed by the second assessor. In roughly half of these human odor evaluations, the two assessments did not match. To resolve this, both evaluators performed odour 24

testing together on the buckets that caused the confusion, side by side. This was repeated until all

buckets were scored to the satisfaction of both evaluators. The time taken for each bucket to be

smelt was less than a minute per bucket, after which the lid was put back on.

2.3 Leachate Analysis

The concentration of odor indicators was measured only in the liquid phase. There was a lack of leachate being produced from the decomposing wastes which then resulted in the adoption of a leachate extraction technique (Qamaruz-Zaman and Milke, 2007) to enable enough liquid samples for analysis. The idea behind the liquid extraction procedure was to wash the outsides of the wastes. This is based on the concept that odour is associated with the waste's outer layer rather than its inner portion, where the smell remains enclosed and is less volatile. The buckets were at times aerobic and at times anaerobic, much like food waste stored in households would be. Because of this, the compounds extracted can be expected to be similar to those extracted in many household food waste storage conditions.

First, the bucket contents were emptied into a muslin bag which were then completely soaked with tap water for 30 minutes. The volume of tap water used was determined from the weight of the bucket contents, with 1 ml of tap water addition per g of waste. The waste-contained muslin bags were hung for another 30 minutes to recover liquid. The simulated leachate then underwent filtration to 0.45μm Milipore filter without any polymer addition or centrifugation, prior to analysis.

Liquid samples were analysed for ammonia (NH₃-N) on a Hach Spectrophotometer and total organic carbon (TOC) on a Teledyne Tekmar (USA) Apollo 9000 Analyzer. Volatile fatty acid (VFA) were determined on a HP6980 Gas Chromatograph, with, only acetic,

- 1 propionic, butyric and isovaleric acids being reported and summed as total VFA (TVFA).
- 2 Other VFA were not considered because they were found to be at much lower
- 3 concentrations and so less suitable as odor indicator parameters. The quality of the gas
- 4 chromatograph results were maintained through re-validation of an acetic acid standard
- 5 solution at the start and completion of five sample injections. Table 3 describe the basic
- 6 quality assurance parameters for all the above target components including the minimum
- 7 detection limit and the uncertainty of measurements. pH of the extracted leachate was
- 8 taken using an EDT RE357 Microprocessor pH meter, which was calibrated daily.3.
- 9 Results and discussion
- 10 3.1 Waste Decomposition

- 11 As illustrated by Figure 1, the waste was more decomposed in 20°C storage than when stored at
- 12 8°C. About 10% of the initial weight was lost in the decomposition process, where weight loss
- was higher for the warmer condition.
 - 3.2 Odor Indicator Concentrations
- 16 In general, odor intensity increased linearly with increasing storage time for food wastes stored
- at ambient (20°C) and lower temperature (8°C) conditions (refer Figure 2). Odor from the higher
- temperature was always more intense than at lower temperature. For wastes stored at the higher
- temperature, a very intense odor (scale of 5) was evident after 7 days, compared to storing at the
- 20 lower temperature which emitted only a light odor (scale of 2). An offensive odor (scale of 5)
- 21 never occurred with the latter. The findings follows that of Zhou et al. (2003) who observed
- 22 more intentense odor from a landfill in China during the summer months compared to winter,
- 23 Their study recorded traces of volatile organic compounds (VOCs) being 1-2 order of magnitude
- 24 higher during summer. This behaviour of rise in odor emissions in parellel to temperature are
- 25 likely true until a temperature setpoint of 60°C (Zhang et al., 2009). Beyond this point, odor

1 emissions decreased despite the rise in temperature. It was thought that the odor-causing -2 microorganism have stopped growing or started to die off at 60°C. 3 4 From the initial ammonia content of 100 mg/l, ammonia increased to 1370 mg/l for the higher 5 temperature after 14 days storage, while it tripled to 360 mg/l at low temperature. 6 Correspondingly, the odor intensity for the elevated temperature was very intense (scale of 5) 7 whereas it was only moderate (scale of 3) for the lower temperature. 8 9 With volatile fatty acids, an inverse relationship was observed between acetic acid and odor 10 whereby the former decreased at rising odor intensity. Where the difference between odor 11 intensity was large, the drop in acetic acid was small, implying the weakness of acetic acid to 12 indicate food waste odor intensity. Similarly, neither butyric and propionic acid were found 13 suitable as food waste odor indicators. No correlation was found with TVFA, which was 14 influenced by the non-correlative response of the major individual acids, namely, propionic, 15 acetic and butyric acids (Table 4). 16 17 Isovaleric acid was the only VFA measured that shows potential as a food waste odor indicator. 18 When the presence of odor was minimal or even none, no isovaleric acid was present in the 19 leachate. Likewise, the highest concentration of 1240 mg/l, recorded at the higher temperature 20 after twelve days, saw an odor intensity of 5. Isovaleric acid increased with time and was always 21 higher at the elevated temperature. 22 23 Unlike ammonia and isovaleric acid, no relationship was observed between odor intensity and 24 the measured total organic carbon (TOC). Despite the rise in odor intensity level for both

temperature conditions, the same pattern was not observed with the TOC measurements. The

TOC content fluctuated without any regard to a particular storage period, temperature, or odor

25

1 intensity.

2

- It was thought that pH could have influenced the formation of odor. The increasing pH from 5.3 to 6.9 and 6.5 after 14 days for high and low temperatures respectively, saw a subsequent rise in
- 5 ammonia from 100 mg/l to 1370 mg/l (20°C) to 360 mg/l (8°C). According to Nakasaki et al.
- 6 (2000), the increase in pH value is a result of both the production of NH₃ associated with protein
- 7 degradation and the decomposition of the organic acids. The pH results show significant
- 8 variability with a relatively small change over time. We conclude that pH appears to be a
- 9 relatively poor choice for an odor indicator.

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- 3.3 Identification of Indicator Threshold Value
- 12 Table 5 shows indicator concentrations and the corresponding odor intensities. Odor was not
- detected when ammonia and isovaleric acid were below 170 mg/l and 530 mg/l, respectively.
- 14 However, odor was very strong when the ammonia concentration rose to between 841 1400
- mg/l and isovaleric acid between 1060 1240 mg/l.

16

- 17 Ammonia and isovaleric acid have been identified among odor compounds released during the
- composting process. The former has a pungent and sharp odor characteristic and can be smelt by
- 19 the general population at 39,600 μg/m³, while the latter is more of a rancid cheese characteristic
 - with a 52.8 µg/m³ odor threshold (Epstein, 2011). Increases in ammonia are likely to be
- 21 reflective of organic nitrogen reduction to a more suitable nitrogen form for bacterial
- incorporation (Powers et al., 1999). This is important as these bacteria work to degrade protein in
- food wastes, hence fulfilling the criteria proposed by Spoelstra (1980) that odor indicators should
- be products of protein degradation.
- 25 Isovaleric acid stood out as a promising odor indicator, even though it was the VFA at the lowest

concentration of the four acids reported. Similarly, an experiment by Zhu et. al. (1997) found that some products reduced swine manure odor threshold without significantly reducing the total amount of VFAs, signifying a poor correlation. These five commercial additives; MPC, Bio-Safe, Shac, X-Stink and CPPD were based on chemical, enzymatic and bacterial interactions to reduce odor release from swine manure. Their study suggested that the products may have reduced the concentrations of long chain and branching fatty acids, and since these acids did not necessarily exist in high concentrations, they were unlikely to have made a significant difference

on the total VFA. This supports our findings with isovaleric acid.

4. Conclusions

This research has shown that ammonia and isovaleric acid have the potential as odor indicators for stored food wastes. It was not the intention to try to identify the compounds directly responsible for the odor volatilized from the waste and measure those concentrations. Rather, the intention was to utilise readily measurable, and common odor compounds, which are common end products of anaerobic and aerobic decomposition of organic wastes, and also relatively independent of the mix of food wastes. It is acknowledged that specific food wastes could lead to specific odor problems with odorous, trace gaseous compounds. An attempt was made in this study to counteract this effect by using a mix of food wastes, yet, there is still the potential that specific foods could be odorous and not be picked up with either ammonia or isovaleric acid. The leaching of ammonia and isovaleric acid found in this study could vary significantly if other food waste mixtures are used. The threshold values presented have not been tested with other food waste mixtures.

It is not advised that treatment regimes be designed to specifically target at reducing the concentrations of these indicators, as it cannot be guaranteed that by diminishing the ammonia

- and isovaleric acid in the leachate, food waste would have less odor. Instead, the unacceptable
- 2 limits of concentrations of leachate indicator compounds, would be useful as a guide for waste
- 3 managers of when the food waste odor is likely to become a nuisance.

- 5 No previous research has been reported on the use of odor indicators for residential food waste.
- 6 Further study is needed on a variety of aspects including the sensitivity of the results to variations
- 7 in food waste composition and the sensitivity to human odor assessment methods.

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1 Table 1. The food waste content (in wet weight percentages) used in the experiments

<u>Item</u>	Percentage (%)
Carrot skin	0.5
Apple skin	1.5
Apple seed	1.5
Pumpkin seed	2.0
Teabags	2.6
Bread	3.0
Pumpkin skin	3.5
Potato skin	4.0
Mixed vegetable (stir fry)	4.0
Chicken skin (raw)	4.0
Eggshells	4.0
Coffee ground and filter	4.5
Mashed potato	5.0
Spaghetti leftover	5.0
Boiled pumpkin	5.4
Broccoli stalk (raw)	6.6
Cooked rice	7.0
Orange peel	7.0
Chicken bone (raw)	8.0
Banana skin	8.5
Salad	<u>12.5</u>
	100.0

Table 2. The odor intensity rating table (source: Table 2.2 (Blackford et. al., 1998))

Scale	1	3			5	
			+			
Intensity	Very light	Light	Moderate	Strong	Very strong	
level						
Description	- activates the	- activates the	- easily	- objectionable	- so strong it is	
	sense of smell	sense of smell	activates the	- cause a	overpowering	
	- characteristics	-	sense of smell	person to	and intolerable	
	may not be	distinguishable	- very distinct	attempt to	for any length	
	distinguishable	and definite	and clearly	avoid it	of time	
		- not necessarily	distinguishable	completely	- could tend to	
		objectionable in	- may tend to be	- could indicate	easily produce	
		short durations	objectionable	a tendency to	some	
			and/or irritating	possibly	physiological	
				produce	effects	
				physiological		
				effects during		
				prolonged		
				exposure		
Scale of 0: od	or not detectable					

Table 3 The MDL and uncertainty of the VFAs, TOC and NH_3 -N

Compound	Minimum detection limit	Uncertainty (g/l)		
	(MDL) mg/l			
Isovaleric acid	530	0.54 ± 0.07		
Acetic acid	2124			
Propionic acid	2244			
Butyric acid	1751	6.5 ± 0.8		
Total organic carbon	1000	1 ± 0.03		
Ammonia	0.4	0.0115 ± 0.0004		

Days in

storage

20°C storage

8°C storage

Figure 1. The decomposition of food wastes at high and low temperature

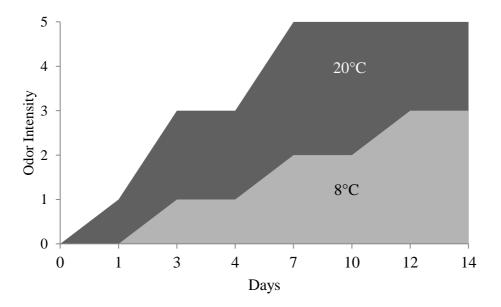


Figure 2. Odor intensity of food waste stored at different temperatures

Table 4. The odor intensity and corresponding leachate characteristics at separate temperature conditions during 14 days storage

Day	Temperature	O.I*	pН	TOC	NH ₃ -N	Acetic	Prop ^a	Butyric	Iso-
				(mg/l)	(mg/l)	(mg/l)	(mg/l)	(mg/l)	Valeric
									(mg/l)
1	High	1	5.9	3320	170	n/a	n/a	n/a	n/a
	Low	0	6.3	3580	100	2900	3600	4100	530
3	High	3	5.4	1180	300	3200	3800	3800	530
	Low	1	5.9	2430	190	3100	3500	2600	530
7	High	5	5.9	2240	830	3000	3400	2500	710
	Low	2	6.4	1490	230	3000	3600	3300	530
10	High	5	6.6	1070	1040	3100	3600	2700	1070
	Low	2	6.4	1790	260	3000	3400	2400	680
14	High	5	6.9	1250	1380	3100	3500	2700	1240
	Low	3	6.5	1800	360	2900	3500	2500	950

^{*} O.I: odor intensity, ^a: Propionic

Table 5. Suggested threshold ammonia and isovaleric acid values for varying odor intensities

Odor	Ammonia	Isovaleric		
intensity	(mg/l)	Acid (mg/l)	Description of odor	
0	100 - 170	≤ 530	No odor: undetectable, unnoticeable	
1	170 - 240	530 – 670	Faint: slight occasional wafts, undistinguishable odor	
2	240 - 300	670 – 700	Light: slight and constant, distinguishable odor	
3	300 - 360	700 - 940	Moderate: Distinguishable odor and sometimes irritating	
4	360 - 840	940 - 1060	Strong: Unbearable odor but causes no physiological effects	
5	840 - 1400	1060 - 1240	Intense: Intolerable odor for any length of time and can produce physiological effects	