Title

Odor Control of Wastewater Sludge Drying and Sludge Hydrolysis Processes

Author
Adigwe, Regina

Publication Date
2024

UNIVERSITY OF CALIFORNIA

Los Angeles

Odor Control of Wastewater Sludge Drying and Sludge Hydrolysis Processes

A dissertation submitted in partial satisfaction of the requirements

for the degree doctor

of

Environmental Science and Engineering

by

Regina Nneamaka Adigwe

2024

ABSTRACT OF THE DISSERTATION

Odor Control of Wastewater Sludge Drying and Sludge Hydrolysis Processes

by

Regina Nneamaka Adigwe

Doctor of Environmental Science and Engineering

University of California, Los Angeles, 2024

Professor Irwin H. Suffet, Chair

The sludge drying process, a crucial step in treating sludge from wastewater treatment plants, significantly reduces the sludge volume. This reduction makes it more manageable for transportation, storage, and utilization as fertilizer or soil amendment. However, despite its importance, the sludge drying process is marred by the emission of odors, which not only lead to complaints from neighboring communities but also impede the use of dried sludge as fertilizer.

Given the challenges posed by the odors emitted during the sludge drying process, it becomes imperative to evaluate these odors comprehensively. The current standard method of evaluation, which focuses solely on olfactometry, falls short as it does not provide a complete understanding of the character and intensity of odors. Therefore, this dissertation delves into the use of sensory and chemical analysis to gain a more nuanced understanding of the odors emitted during various sludge drying processes and at different temperatures.

The study employed the Odor Profile Method (OPM) as the sensory method, which involved a panel of at least four individuals describing the character and intensity of the odor. The chemical analysis was conducted using a Gas Chromatography-Mass Spectrometer (GC-MS), which identified the chemicals responsible for the odorants. This combined approach provided a comprehensive pattern of odor evolution during the sludge drying process, similar to the one used to evaluate drinking water odor.

A pilot study at a WWTP in Limay, France, laid the foundation for developing the sludge drying odor wheel using a pilot laboratory roto evaporator system that emulated the sludge drying process at a drying facility. After 14 years, a follow-up study was done using sludge samples from Orange County Sanitation Districts (OCSD) in Southern California, USA. Following similar pilot laboratory procedures, the study confirmed they had similar odors but were not duplicable compared with the pilot study. The primary odors occurring in both studies were fecal, rotten vegetable, and rancid. OPM data was proven by finding the chemical concentration/OTC ratio. A concentration/OTC ratio of over 1 demonstrates that the panelists detected the odor during the study. The concentration/OTC ratio in the pilot study corresponded with the OPM data. Furthermore, with the occurrence of fecal odor in both the pilot study and the follow-up study, chromatographs indicated that indole was responsible for the fecal odor.

To better understand the process of odor evolution during sludge drying, this dissertation explored a new approach by creating artificially undigested and digested sludge recipes to observe if the same sludge drying odors could be replicated and to understand how the sludge composition affects odor during the sludge drying process. The primary odors detected were consistent with the previous study, including the burnt odor. The simulated sludge study indicated that fecal and musty/earthy odors were also significant. A caramel odor was detected as a new odor within the burnt odor category. The sludge composition contributing to each odor was more easily studied by simulating a sludge-digesting recipe. The different recipes of simulated sludge contained specific ratios of carbohydrates, proteins, and lipids. Raw sludge was added to a recipe to stimulate anaerobic digestion. However, the odors produced from the recipe with raw sludge were much closer to the actual odors from the sludge drying process than those without adding digested sludge. The observed difference in odor suggests odor variation based on varying sludge composition and microbial activity within the sludge.

Sludge drying at different temperatures can also affect the odors emitted during the process. To understand how odors change at different temperatures, thermal hydrolysis and indirect drying were carried out. The odor produced from both processes was analyzed using the Odor Profile Method (OPM) and the sludge drying odor wheel. The musty odor was the primary odor in thermal hydrolysis, while the fecal odor was the primary odor in sludge drying. Results revealed that thermal hydrolysis is less of an odor nuisance than sludge drying.

The results from the studies of this dissertation revealed a new odor category (caramel odor) that needs to be further investigated to be added to the sludge drying odor wheel. Also, the production of the caramel odor needs more exploration, as caramel odor can be the ideal odor for the sludge drying process. Additionally, the thermal hydrolysis process for sludge drying suggests a more controlled process for odor nuisance in sludge drying.

The dissertation of Regina Nneamaka Adigwe is approved.

Jennifer Ayla Jay

Sanjay K. Mohanty

Michael K. Stenstrom

Irwin H. Suffet, Committer Chair

University of California, Los Angeles

2024

Table of Contents

List of Tables

List of Figures

List of Acronyms

E.U.	European Union
DMS	Dimethyl Sulfide
DMDS	Dimethyl disulfide
DS	Dry Solids
MVOC	Malodorous Volatile Organic Compounds
PAC	Polyaromatic Compounds
OAVs	Odor Activity values
MESH	Methyl Mercaptan
WWTP	Wastewater Treatment Plant
OCSD	Orange County Sanitation District
OPM	Odor Profile Method
GC/MS	Gas Chromatography/Mass Spectrometry
ODP	Odor Detection Port
THP	Thermal Hydrolysis Process
VFAs	Volatile Fatty Acids

Acknowledgement

Ultimately, I am super grateful to God for my time in UCLA. It's been a privilege to learn and grow within the UCLA community. Next, I would like to express my gratitude to my advisor, Dr. Mel Suffet, for his guidance, support, inspiration, and motivation through my doctoral study. His kindness and willingness to support my thought process made my experience as a doctoral student more interesting. With his support, I have navigated high quality research works and the industry outside the school space which has made me a more confident professional. More importantly, I would like to express my gratitude to the other members of my doctoral committee, Professor Sanjay Mohanty, Professor Michael Stenstrom, and Professor Jennifer Jay.

Also, I am thankful to my supervisor at Orange County Water District, Christine Pham. You are a huge inspiration, and your kindness will never be forgotten. A big thank you to Harrison Levy for your immeasurable support which made navigating the ESE program easier. Thank you to all the students who have contributed and participated in my research especially Yuge Bian. Your participation in my project is invaluable and will always be appreciated.

Special thanks to my Fiancé Kenechi Orizu for your unwavering support and guidance. Thank you for helping me believe in myself and being my strength when I thought I would give up.

Finally, a very big thanks to my parents, sisters, and my brother for helping me get through one of the toughest times in my life. Distance was never a barrier to feel your love!

Biographical Sketch

Field Environmental Engineer Intern – Research and Development
Orange County Water District (Research & Development Department Nov. 2021 – Present

- Design and implement pilot column scale testing to assess absorbent for Per and Polyfluoroalkyl Substances (PFAS) in-situ treatment for groundwater management
- Perform various laboratory analysis such as permeametry analysis, wet sieve soil analysis, and total suspended solids analysis
- Assist in experimental studies related to PFAS Ex-situ treatment in groundwater using ion exchange and other absorbents
- Compile, organize, and evaluate field and laboratory data for incorporation into technical reports; Recognize and identify discrepancies in results obtained in such assignments and follow-up with senior personnel to resolve these situations
- Assist in setting up pilot experimental studies and in charge of monitoring of these pilot studies.
- Perform soil and water sample collection, data entry and analysis using Excel, GIS and R
- Perform standard or routine calculations, input data into and/or run standard or, in some cases advanced computer programs under supervision.
- Conduct scheduling and coordination of routine OMM activities for operation and compliance of groundwater extraction and treatment systems. Also perform OMM activities, conduct monitoring of system status, respond to system alarms, and evaluate operational data associated with system performance.

Research Project Oct. 2019 – Present
Dr. Mel Suffet Lab (University of California, Los Angeles)

- Led a team of four research graduates to automate NDMA analysis in water and wastewater treatment facilities using a novel direct-injection chemiluminescence-based method that is less time consuming and saves $45,000 in cost
- Manage multiple environmental projects simultaneously while providing guidance for environmental regulations compliance through working knowledge of NPDES Permits, TMDL, CEQA, NEPA, RCRA, and Environmental Management System (EMS)
- Perform analysis of storm water samples and landfill samples for odor using the Odor Profile Method (OPM)
- Observe safety procedures and controls for lab experiments
- Perform analysis of odorant concentration and intensity during sludge drying
- Perform tests for water quality, chemical contaminants, emerging contaminants (NDMA), and risk assessment

Principal Investigator
Pritzker Award Partnership with IoES UCLA
Oct. 2021– August 2022

- Investigated and reviewed "Emerging Sanitation Technologies that meet Human and Ocean Health Goals

Chapter 1

Introduction and Objectives

1.1 Introduction

The odor from sludge has been a persistent odor problem from Wastewater Treatment Plants (WWTPs). WWTPs are not considered good neighbors if odor complaints and community opposition come from the plant's surrounding area. (Hayes et al., 2014). Furthermore, the release of foul odors during the wastewater treatment processes is known to be associated with photochemical smog formation and secondary particulate contaminant emissions (Fan et al., 2020).

The production and treatment of wastewater sludges are a significant part of the odor problem, as reviewed by Fisher et al. (2019). The sludge quality is strongly tied to the wastewater treatment process. Also, the configuration and operation of the sludge treatment processes affect the characteristics of odor emission throughout the sludge process. For example, Zhu et al. (2022) have shown that low-temperature thermal drying processes and dried sludge, especially sludge without digestion or other stabilization pretreatments, have a powerful and offensive odor.

Wastewater sludge is the by-product of treating wastewater in WWTPs, which undergoes various treatments before its use as a fertilizer in forests and agriculture or disposed of in landfills (Demirbas et al., 2017). Wastewater sludges have valuable agronomic properties if sufficiently treated (e.g., supplying phosphorus and nitrogen). Domestic wastewater from homes is the most easily treated. If sludge from wastewater contains compounds that are poorly biodegradable, such as heavy metals, and toxic chemicals like pesticides, the sludge cannot be used for agricultural purposes. Therefore, improper disposal of sludge seriously pollutes water and soil resources and spreads diseases, thus adversely affecting human living conditions (Bao & Wang, 2023). Therefore, wastewater treatment plants have developed rules that limit these substances from entering a wastewater treatment plant. Thus, pretreatment of the wastewater for removal of these hazardous substances is required for industrial purposes by many WWTPs.

Economically, wastewater sludge must be reduced in volume through drying for storage and transportation before final disposal of sludge for agricultural use or to a sanitary landfill, or even for combustion (Bao & Wang, 2023). Achieving a green and sustainable world requires proper handling of the increasing amount of residual sludge from WWTPs in an economical and environmentally friendly manner. This sustainability issue remains a critical issue. (Ahmad et al., 2016).

Generally, sludge treatment costs account for about 50% of the whole wastewater treatment plant (Chang et al., 2013). Today, almost all plants built from 1938 to the seventies have been closed or upgraded due to technical and odor problems or because of economic reasons (Barber, 2020). Reducing the cost and energy associated with sludge treatment is imperative and depends upon various technologies, including sludge dewatering, drying, and thermal hydrolysis. In assessing these technologies, methods are aimed at stabilizing organic matter. The methods should include destroying pathogens, eliminating odor, and reducing volatile contents while maximizing nutrient recovery for safer disposal (Oladejo et al., 2019). However, there are many drying technologies, but most of the sludge drying technologies are costly, energy-intensive, and produce unpleasant odors during the drying process (Bratina et al., 2016).

Thermal drying is widely used with other dewatering methods for volume reduction and stabilization treatment of wastewater sludge (Zhu et al., 2022). Typical sludge drying technologies include thermal drying, which consists of A. Direct Drying, B. Indirect Drying, C. Solar Drying (Bennamoun et al., 2011), D. Microwave Technology (Mawoo et al., 2017), and E. Fry Drying

(Chang et al., 2013). and F. Thermal Hydrolysis (Barber, 2020). The selection of a drying method depends on the initial and final moisture contents, the product use, the ability to operate and maintain the system, and the nature and capacity of the sludge being handled (Chang et al., 2013).

Direct drying of sludge occurs with direct contact of the sludge with the heat source, which could be hot air or superheated steam, and acts as the conveying medium. Direct drying has the advantage of drying all types of sludge, including primary, secondary, and mixed-digested sludge. Unfortunately, the investment and operation costs of the thermal drying process are high, and the energy consumption of equipment operation is large, causing unpleasant odors (Xue et al., 2022). Although solar drying offers the advantage of being environmentally friendly and energy-saving, it presents a high odor complaint problem and can be time-consuming (Liu & Lee, 2015). Fry drying has the advantage of producing sludge with a high calorific value and uses waste oil while producing odor-free organic pellets. It also achieves waste material disposal and cost reduction and efficient removal of moisture content, but it decreases nitrogen and sulfur content as the ratio of oil used increases (Liu & Lee, 2015).

Microwave technology is widely used in heating applications, and its unique operational principle offers many advantages over conventional heating (Maiwoo et al., 2017). For instance, in contrast to conventional thermal processes, microwave energy offers benefits such as high heating rates, interior heating, energy savings, greater control of the heating process, and a higher level of safety and automation. However, at low temperatures, microwave technology cannot attain organic stabilization of sludge (Maiwoo et al., 2017).

Figure 1.1 shows the occurrence of dewatering and drying in a general sludge treatment process.

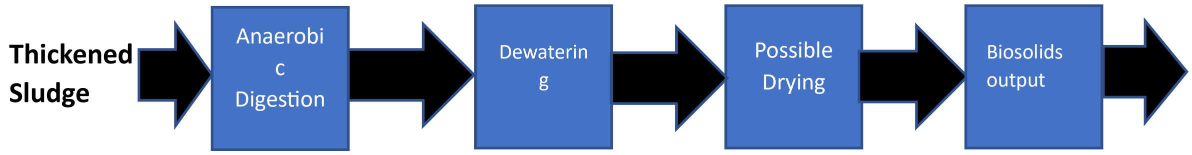


Figure 1.1. Schematic diagram of the Sludge Treatment Process

Dewatering is an energy-intensive process but an essential step after anaerobic digestion to improve waste into useful end products from wastewater sludge (Chen et al., 2013). The two dominant technologies in wastewater sludge dewatering are belt filter presses and centrifuges. Decanter centrifuges use a high-speed rotational process to achieve dewatering by applying high forces of 2.000–4,000 G's directly to the feed solids.

Dewatering plays a pivotal role in waste management, increasing the dry solids of sludge from 3-5% to 15-30% (Metcalf and Eddy, 2002). This significant increase in dry solids content leads to a corresponding decrease in the cost of handling, transporting, and storing the final product. The concentrated sludge, free from harmful chemicals, can be directly composted with green waste. Moreover, the sludge can be further thermally dried to 92–97% of dry content and transformed into sludge pellets, which can be incinerated or used as fertilizers (Collard et al., 2017). The highly dried sludge, with its increased calorific value, can also be utilized as a fuel or a co-fuel in various industries, including cement kilns, coal-fired power plants, municipal waste incinerators, and mono-incinerators (Bennamoun et al., 2013). However, it is important to note that all these dewatering processes have been shown to produce odor nuisances (Vega et al., 2015; Fisher et al., 2019). The production of odors during the dewatering process will be thoroughly evaluated in Chapters 2 and 3 of this thesis.

Today, many experts consider the thermal hydrolysis process (THP) a successful strategy for sludge treatment (Han et al., 2021). Primarily in Europe and Asia, studies show that thermal hydrolysis at 120°C to 200oC, most often with pressures up to 7 bars for one hour, could significantly improve the dewaterability of sludge by destroying microbial cells and releasing bound water (Feng et al., 2014). Unfortunately, the pattern of odor production during the Thermal Hydrolysis Process (THP) has not been extensively studied. Thus, the relationship between odor production and high temperatures used by THP including sludge content needs to be investigated.

Figure 1.2 shows a schematic diagram of THP after the dewatering of sludge (Barber, 2020). The THP involves applying heat to thickened or dewatered sludge produced during wastewater treatment at temperatures between 120°C and 200°C for a defined time. (Barber, 2020). Initially, THP was installed before anaerobic digestion, but more recently, it is used downstream of the anaerobic digestion process, as seen in Figure 1.2 (Barber, 2020). When thermal hydrolysis is used, the dewatered cake produced can achieve 60% dry solids (Barber, 2016) and, therefore, needs no further drying and can serve as a fertilizer after treatment.

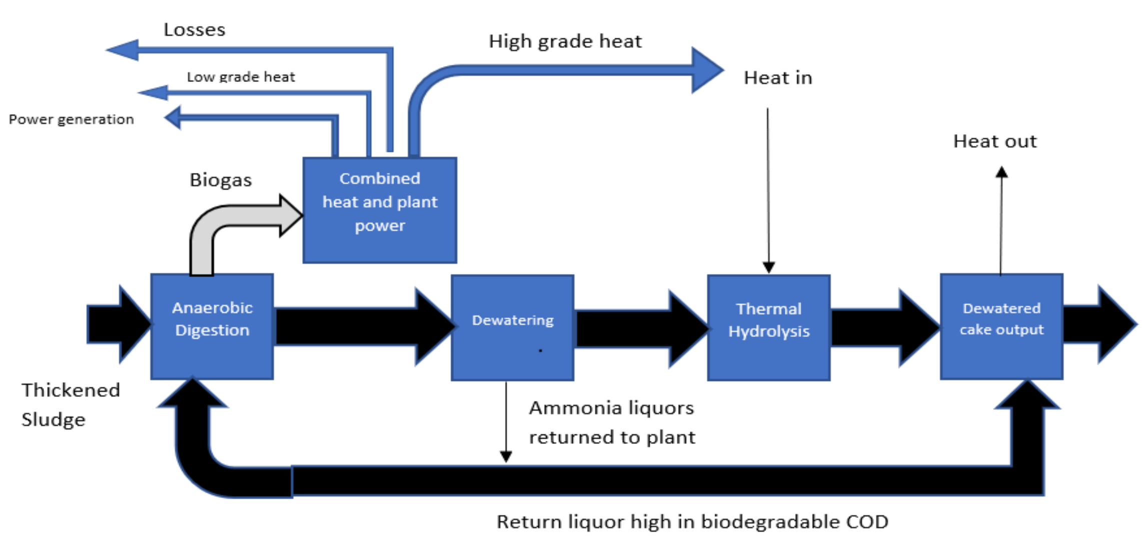


Figure 1.2. Schematic Diagram of the Thermal hydrolysis Process after Dewatering (Barber, 2020)

A significant limitation of THP is the production of potentially hazardous and odorous chemicals, such as NH3 and volatile sulfur compounds. In addition to causing unpleasant odors, these compounds may be harmful to human health when concentrations in the working environment exceed certain health thresholds (Han et al., 2021). For example, the permissible concentration-time weighted average (PC-TWA) of hydrogen sulfide (H2S) and methyl mercaptan are 1 and 0.5 ppm, respectively. When these concentrations exceed their PC-TWAs, on-site employees may experience upper respiratory tract irritation, central nervous system impairment, or even liver damage (Han et al., 2021).

Figure 1.2 shows the heat for thermal hydrolysis is typically supplied by live steam injection at design temperature and concomitant pressure, which is then rapidly released (exploded), although some configurations use standard heat exchange (Barber, 2016). Before thermal hydrolysis is applied, the sludge is dewatered to about 10% - 30% dissolved solids to reduce the heating demands of the thermal hydrolysis plant. The heating of sludge causes solubilization of material within it, destroys materials such as extracellular polymers in activated sludge, and sterilizes the sludge from microorganisms, making the sludge compressible, which further dewaters the sludge (Barber, 2020). Freeing cell water under hydrolysis changes the viscosity of sludge. Therefore, sludge with about 12% dissolved solids can be handled in the same way as raw sludge with 5–6% dissolved solids. This process allows for a higher sludge concentration in the digester feed, higher buffer capacity, and a stable digestion process (Keep et al., 2000). After processing, if the sludge goes to an anaerobic digester, it is removed from the plant at approximately 100oC and slightly lower dry solids due to dilution with the added stream. It is then further diluted using clean water or treated effluent to control the loading rate to the downstream anaerobic digestion plant, which reduces the sludge temperature by approximately 20 oC (Barber, 2020).

The efficacy of thermal hydrolysis in generating high-quality biosolids has been demonstrated in a Norwegian operation plant established in 1995. The plant was designed to minimize sludge volume and maximize biogas production (Barber, 2020). Presently, the plant produces digested and disinfected sludge that is highly sought after by farmers and is utilized 100% in agriculture and land reclamation (Keep et al., 2000). The decision to install thermal hydrolysis systems is often driven by market demand and geographical factors. However, the expected benefits include increased loading rates (to reduce the size of new digestion plants or maximize the use of existing facilities), improved sludge cake dewaterability (which lowers downstream transport and processing costs), increased production of renewable energy, and sludge sterilization (Barber, 2016). Table 1 summarizes global thermal hydrolysis capacity, ranked by installed capacity from suppliers.

Table 1.1: Full Scale Suppliers Worldwide of the THP Technology

Name of Manufacturers	Number	% of Total	Installed Capacity	% Installed
Cambi	65	53	6947	77
Shanghai, Pulande	6	5	432	5
Sichuan, Deepblue	11	9	324	4
Tianjin, Yuchuan	4	3	220	2
Biothelys	7	6	206	2
Beijing, Jiankunweihua	5	4	185	2
Beijing, GeeGreen	3	2	180	2
Hubei Guoxin Tianhui	4	3	146	2
Exelys	6	5	126	1
Shenzhen, Est	3	2	70	1
Turbotech	2	2	55	1
Beijing, Hinergy	1	1	40	0
Lysotherm	2	2	36	0
Haarslev	3	2	32	0
Average Total % of Total	122		8999	

Studies underscore the promising economic benefits of thermal hydrolysis technology, particularly for plants grappling with high cake disposal costs. The potential savings in cake disposal costs are increasingly recognized as a significant advantage in modern wastewater treatment plants (Svennevik et al., 2019). Moreover, the substantial enhancements in dewatering could revolutionize the design and footprint of new plants adopting thermal hydrolysis. The need for storage space for dewatered cake could be significantly reduced, leading to a decrease in the number of trucks required for cake disposal. Furthermore, the higher dryness of the dewatered cake could open up new possibilities for alternative cake disposal methods. However, to effectively incorporate these factors into the planning of new plants, more information on the expected dry solids concentration of dewatered cake after thermal hydrolysis is crucial (Svennevik et al., 2019). Additionally, the issue of odorous or harmful odor production during THP remains a significant area that requires further understanding (Han et al., 2021). Thermal hydrolysis treatments, such as the Post-Anaerobic Digestion thermal hydrolysis process (Post-AD THP), have been developed to enhance sludge dewatering, but their mode of action is not yet fully understood (Svennevik et al., 2019).

High quality of biosolids is a result of higher degradability after THP and more stabilization of its final product with a pathogen free product that reduced the risk of biosolids management which further adds value to the final product but causes odors (Sahu et al., 2022). Thus, an effective strategy to eliminate odors is needed. The strategy should identify the essential odors and chemical emissions during the thermal hydrolysis process to enable corrective treatment to be developed to remove the odors.

Deodorization remains a major objective of each sludge treatment and plays an important role as an index to measure the stabilization extent of all wastewater sludge processes. Accordingly, identification of the odor emission characteristics of sludge is important in the evaluation of sludge quality and disposal options (Gao et al., 2022). Thus, a standard method to identify the odors and the primary chemicals that produce the odors should be developed to enable the correct treatment of the odors released from these processes. This is the objective of this thesis.

1.2 Literature Review of Odorous Chemicals from Sludge Drying Processes

Measurement of odor includes sensory and instrumental analysis. Initially, olfactometry (European Standard CEN 13725, 1997; Association of French Normalization, 1997) was used to describe the "Total Odor" and the disappearance of the change of odors during treatment. However, this method did not describe the odor character and intensity of each odor.

Murthy et al. (2003) began initial studies of the odorous chemicals released from sludge drying by studying only the final odors of the digested sludge, followed by Lazanova et al. (2008) and Bouchy et al. (2009). These authors identified a series of odor descriptors that were defined in general but not described with an intensity scale for each descriptor that would define their importance to the overall odor. The specific odor characteristics and their change of intensity were not evaluated. However, of critical importance, these researchers did chemically define odorous chemicals, including trimethyl amine, a series of fatty acids, and reduced sulfur compounds during the heat drying processes that cause significant odors. These odorous chemicals were observed from the final sludge product primary and secondary solids with the effects of moisture, storage time, soil amendments and lime stabilization considered.

Lazanova et al. (2008) and Bouchy et al. (2009) evaluate the odorous chemicals of 8 different sludge drying facilities in France, primarily from WWTPs using a Belt, Paddles, Solar and a Thin layer/belt system under low to high temperatures and low to high retention times by chemical analysis and Olfactometry. Figure 1.3 shows the overall mass flow of chemicals identified comparing compost and sludge drying with 88% of the total emissions being ammonia comparing composting and sludge drying odors.

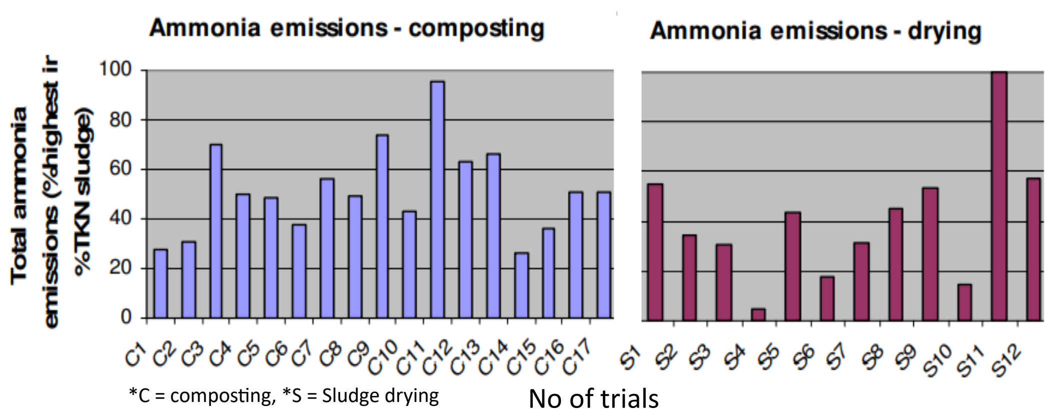


Figure 1.3 Overall mass flow of emission at a treatment plant on a daily basis with 17 trial times for composting and 12 trial times for sludge drying (Bouchy et al. 2009)

The authors point out correctly that the ammonia has a high odor threshold concentration (OTC) compared to the reduced sulfur compounds. For example, the Odor Activity Value (OAV) which equals the Odor Concentration/OTC was used to show that Trimethyl Amine (fishy odor) and the sulfides, methyl mercaptan and dimethyl sulfide (decaying vegetable odors) were the primary odors produced. Ammonia, burnt coffee, burnt rubber, manure and cabbage odor characteristics were observed. Murthy al, (2003) found medicinal, ammonia, chlorinous, fishy, sour, decay, rancid and garbage as primary odor at a different sludge drying facilities, as well.

In a previous study, Decottignies et al.,(2010) used the Odor Profile Method (OPM) to evaluate the odor character and odor intensity of each odor (Burlingame, et al., 2004). A sludge drying odor wheel was developed from the literature and this study of 33 dried sludge samples over a 6-month period from a French sludge drying facility at Limay, France. Samples were also evaluated for the first time from the air during a field study where odors were escaping from that sludge drying site by an OPA, odor panel of four. The samples generated by the dried samples and the odors observed from the on-site investigation during processing were used to produce the first sludge drying odor wheel. The 2010 sludge drying odor wheel is shown in Figure 1.4.

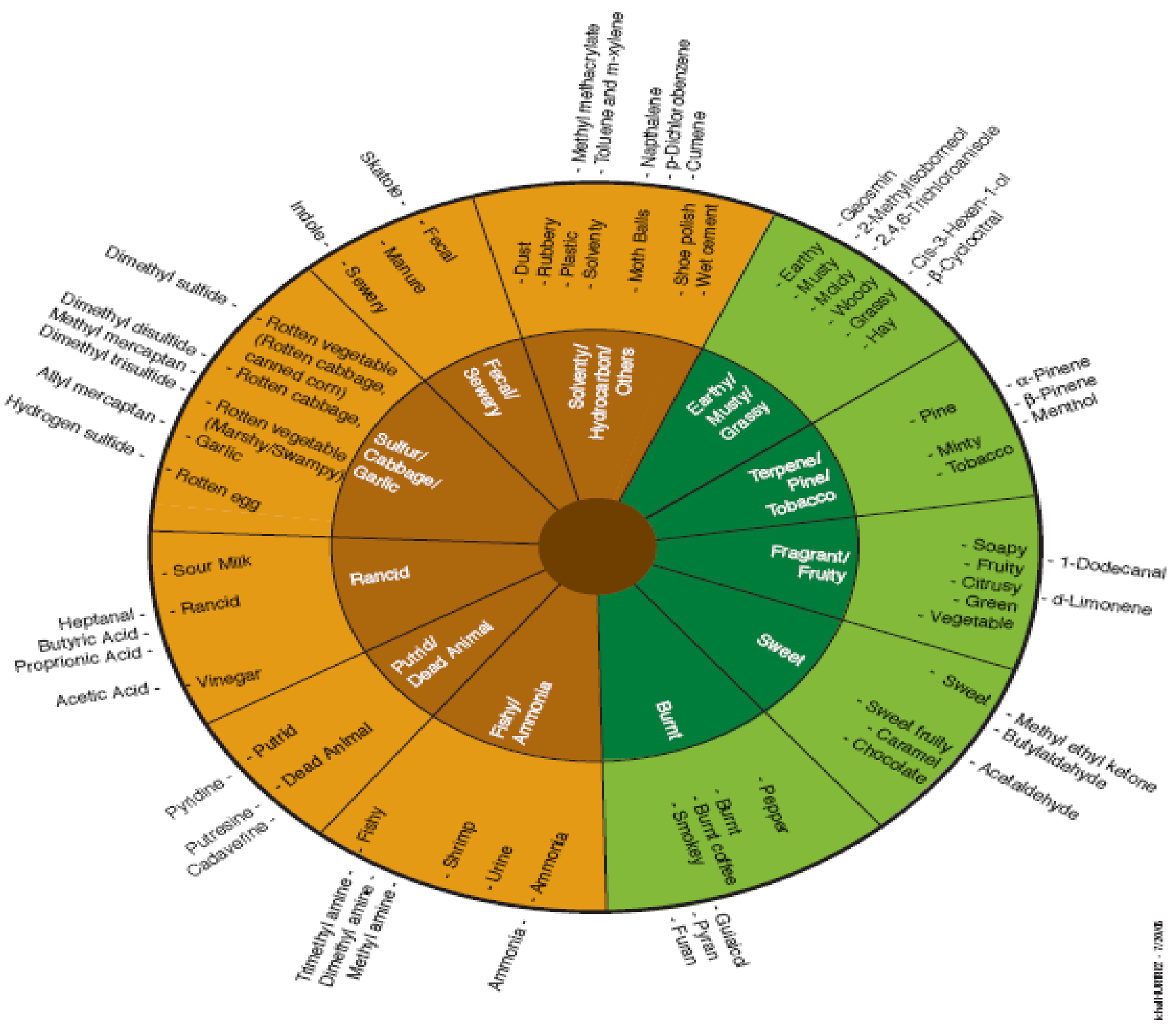


Figure 1.4. Sludge Drying Odor Wheel (Decottignies, Bruchet, & Suffet, 2010)

Decottignies et al., (2010) introduced an initial Laboratory Pilot Operation Method to observe sludge drying process as it is occurring during heating to the final temperature. The odor profile method was used to evaluate odor character. The chemical methods used to collect samples during the process was used to validate the chemical producing the odors for specific chemical groups - ammonia (Acid Bubbler-Indophenol method), trimethyl amine (GC Nitrogen/Phosphorus Detector) , specific sulfur compounds (hydrogen sulfide (H2S), methyl mercaptan (CH3SH), dimethyl sulfide (DMS), dimethyl disulfide (DMDS) and Dimethyl trisulfide (DMTS), volatile acid compounds - Butyric Acid and Isobutryic Acid (GC Flame Ionization Detector) and Acetone and Limonene (Charcoal Cartridge GC/MS).

Fisher et al. (2018) further developed the Sludge Drying Wheel as the "Biosolids Processing Odor Wheel" in 2018 after sampling sludge from 8 wastewater treatment plants from primary sludge, waste activated sludge, thickened primary sludge, thickened waste activated sludge, digested sludge, dewatered sludge, and stored sludge from 3-10 times. Fisher et al., (2018) demonstrated how the Odor Wheel with chemical and odor measurements could be used to identify major odors and the probable chemical(s) causing these odors. Dewatered biosolids from aerobic and anerobic digestion caused the greatest variety of odorants.

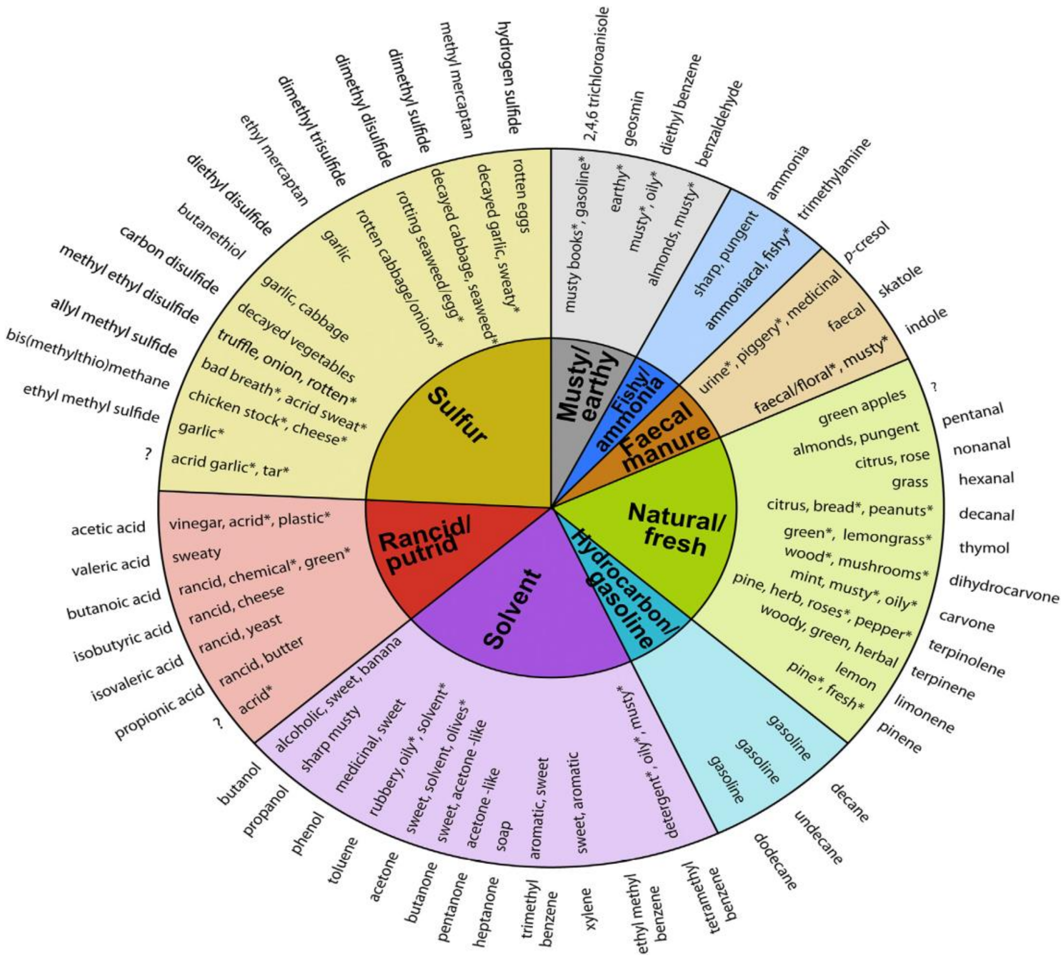


Figure 1.5. Biosolids Processing Odor Wheel (Fisher, Barczak, Suffet, Hayes, and Stuetz, 2018)

Fisher et al. (2019) reviewed the effects of wastewater biosolids stabilization processes on odor emissions and highlighted that volatile Sulfur compounds in Figure 1 were the predominant odorants, and trimethyl amine (fishy), pinene (pine), ammonia, indole (fecal), and p-cresol (medicinal) were also important odorants.

Odorants are emitted during the dewatering process as a function of time and temperature. Besides the temperature, the reduced moisture content during the process would influence the emission rate of odorants (Deng et al., 2009). It is noted that the emission of rancid, earthy, and other types of malodorous volatile organic compounds from wastewater sludge or biosolids has been observed as odors from chemicals such as volatile organic sulfur compounds (VOSCs), volatile fatty acids (VFAs), 2-MIB, and geosmin (Zhu et al., 2022). Due to their low odor threshold concentration, these compounds do not have to occur in high concentrations to cause odor nuisance and unpleasant odor characteristics. For instance, it was found that although H2S concentration was significant in the emission of sludge during thermal drying, MVOCs contributed to 90% of the overall odor emissions (Hoener et al., (2007) and Decottignies et al., (2010))

1.3 Objectives of the Study

The primary and significant goal of this dissertation is to develop advanced knowledge and innovative methods to control odors emitted during the sludge drying process. The detailed objectives, which are crucial for the advancement of wastewater treatment and odor control, include:

1. The meticulous development of a comprehensive laboratory pilot system is vital to this study. It will allow us to observe the odors produced during dewatering processes with precision. This process will be achieved by comparing the similarities and differences of odors from anaerobic sludge collected from WWTP processes in Paris, France, to the sludge produced by the Orange County Sanitation District (OCSD) in Southern California, USA. During the laboratory pilot process, the odor of the initial sludge and the final dried sludge will be compared to the emission of odorants during the process.
2. Paper 2 introduces a novel approach by developing a synthetic sludge model. This innovative model will help us better determine the chemical sources of the odors produced using the

laboratory pilot system, marking a significant step forward in our understanding of odor control.

3. Paper 3 determines the odors produced by a Sludge Hydrolysis Process at high temperatures compared to drying the same sludge at lower temperatures using the laboratory pilot system.

References

Ahmad T., Ahmad K., Alam M. 2016. Sustainable management of water treatment sludge through 3'R' concept, Journal of Cleaner Production, Volume 124, Pages 1-13, ISSN 0959-6526. https://doi.org/10.1016/j.jclepro.2016.02.073.

Bao, B., Wang, F. 2023. Hazardous emission from thermal drying of municipal sludge and industrial sludge as well as associated odor pollution. *J Mater Cycles Waste Manag* **25**, 2187–2194. https://doi.org/10.1007/s10163-023-01675-y

Bratina B., Šorgo A., Kramberger J., Ajdnik U., Zemljič L. F., Ekart J., Šafarič R. 2016. From municipal/industrial wastewater sludge and FOG to fertilizer: A proposal for economic sustainable sludge management, Journal of Environmental Management, Volume 183, Part 3, 2016, Pages 1009-1025, ISSN 0301-4797. https://doi.org/10.1016/j.jenvman.2016.09.063

Bouchy L., Senante E., Dauthuille P., Aupetitgendre P., Harry J. P., Venot S., Rougé P. 2009. Odor creation potential of sludge during composting and drying. *Water Practice and Technology* 1 Sept; 4 (3): wpt2009061. doi: https://doi.org/10.2166/wpt.2009.061

Decottignies, V., Bruchet, A. Suffet, I.H. (Mel) (2010), Dried sludge odours : classification and case study, Paper Paper presented and in the proceedings of the Water Envronment Federation "Odor and Air Pollution Conference". IWA world Water Congress, Vienna, Sept 7-12, 2010.

Demirbas A., Edris G., and Alalayah W. M. 2017. Sludge production from municipal wastewater treatment in sewage treatment plant, Energy Sources, Part A: Recovery, Utilization, and Environmental Effects, 39:10, 999-1006, DOI: 10.1080/15567036.2017.1283551

Fan F., Xu R., Wang D., Meng F. 2020. Application of activated sludge for odor control in wastewater treatment plants: Approaches, advances and outlooks, Water research, Volume 181, 115915, ISSN 0043-1354. https://doi.org/10.1016/j.watres.2020.115915.

Fisher R.M., Le-Minh N., Alvarez-Gaitan J.P., Moore S.J., Stuetz R.M. 2018. Emissions of volatile sulfur compounds (VSCs) throughout wastewater biosolids processing, Science of The Total Environment, Volumes 616–617, Pages 622-631, ISSN 0048-9697. https://doi.org/10.1016/j.scitotenv.2017.10.282

Fisher R. M., Alvarez-Gaitan J. P., and Stuetz R.M. 2019. Review of the effects of wastewater biosolids stabilization processes on odor emissions, Critical Reviews in Environmental Science and Technology, 49:17, 1515-1586, DOI: 10.1080/10643389.2019.1579620

Han Z., Li R., Shen H., Qi F., Liu B., Shen X., Zhang L., Wang X., Sun D. 2021.Emission characteristics and assessment of odors from sludge anaerobic digestion with thermal hydrolysis pretreatment in a wastewater treatment plant, Environmental Pollution, Volume 274, 116516, ISSN 0269-7491. https://doi.org/10.1016/j.envpol.2021.116516.

Hayes, J.E., Stevenson R.J., and Stuetz, R.M. 2014 The impact of malodors on communities: a review of assessment techniques. Sci. Total Enron.500-501, 395-407, 2014.

Lazarova V., Bouchy L., Senante E., Aupetitgendre M., Harry J., Venot S., Dauthuille P. 2008. Fingerprint of odor creation potential of sludge treatment. Water Pract. echnol., Vol 3

Mawioo P. M., Garcia H. A., Hooijmans C. M., Velkushanova K., Simonič M., Mijatović I., Brdjanovic D. 2017. A pilot-scale microwave technology for sludge sanitization and drying, Science of The Total Environment, Volumes 601–602, Pages 1437-1448, ISSN 0048-9697. https://doi.org/10.1016/j.scitotenv.2017.06.004.

Murthy S., Kim H., Peot C., McConnell L., Strawn M., Sadick T., Dolak I. 2003. Evaluation of odor characteristics of heat-dried biosolids product. Water Environ. Res., 75, pp. 523-531

Oladejo J, Shi K, Luo X, Yang G, Wu T. 2019. A Review of Sludge-to-Energy Recovery Methods. *Energies*. 12(1):60. https://doi.org/10.3390/en12010060

Sahu A. K., Mitra I., Kleiven H., Holte H. R., Svensson K.(2022). Chapter 24 - Cambi Thermal Hydrolysis Process (CambiTHP) for sewage sludge treatment, Clean Energy and Resource Recovery, Elsevier, Pages 405-422, ISBN 9780323901789. https://doi.org/10.1016/B978-0-323-90178-9.00020-2. https://www.sciencedirect.com/science/article/pii/B9780323901789000202)

Vega E., Monclús H., Gonzalez-Olmos R., Martin M. J. 2015. Optimizing chemical conditioning for odour removal of undigested sewage sludge in drying processes, Journal of Environmental Management, Volume 150, Pages 111-119, ISSN 0301-4797. https://doi.org/10.1016/j.jenvman.2014.11.012.

Xue S., Ding W., Li L., Ma J., Chai F., Liu J. 2022. Emission, dispersion, and potential risk of volatile organic and odorous compounds in the exhaust gas from two sludge thermal drying processes, Waste Management, Volume 138, Pages 116-124, ISSN 0956-053X. https://doi.org/10.1016/j.wasman.2021.11.040.

Zhu X., Yang X., Gao W., Jiao R., Zhao S., Yu J., Wang D. 2022. Effect of low-temperature thermal drying on malodorous volatile organic compounds (MVOCs) emission of wastewater sludge: The relationship with microbial communities, Environmental Pollution, Volume 306, 119423, ISSN 0269-7491, https://doi.org/10.1016/j.envpol.2022.119423.

Zhu Q., Sun X., Ge S., Yu Y. 2023. Insights into the characteristics and mechanism of vacuum drying technology for municipal sludge processing. Chemosphere, Volume 310, 136729, ISSN 0045-6535, https://doi.org/10.1016/j.chemosphere.2022.136729.

Chapter 2

A Simulation Method to Evaluate and Control Sludge Drying Nuisance Odors

2.0 Abstract

A pilot study was done at Limay, France in 2006 on classifying the odors emitted during the sludge drying process. Odors such as fecal, rotten vegetable, earthy, manure were detected from the off-gases using the Odor Profile Method (OPM). Using GC-MS, nitrogenous compounds, especially ammonia, methylamine, and trimethylamine are the major contributors to the fishy / ammonia odor family. In the sulfur/cabbage/ garlic odor family, hydrogen sulfide, methyl mercaptan and three sulfides (dimethyl sulfide, dimethyl disulfide, and dimethyl trisulfide) contribute to these malodors. Also, butyric acid and isobutyric acid are volatile fatty acids which contribute to the rancid odor family. After 14 years, a follow-up study was done using a sludge sample from Orange County Sanitation Districts (OCSD) in Southern California, USA. Using similar pilot laboratory procedures, the study aimed to confirm the odors and chemicals related to sludge drying off-gases that were detected from the pilot study. Furthermore, the results add to developing a more comprehensive sludge drying odor wheel.

The follow-up study had similar odors but not duplicable compared with the pilot study. The odors emitted in the follow-study includes fecal, rotten vegetable, burnt, earthy, ammonia and rancid odor. Since the composition of the raw sludge entering Lemay plants differs from OCSD, and the operation processes are not 100% identical, the odor profile from the two studies are not the same. OPM data can be proven by finding the chemical concentration/OTC ratio. If the concentration/OTC ratio has value over 1, then the panelists should have detected the odor during the study. The concentration/OTC ratio in the pilot study corresponded with the OPM data. The fecal odor was detected in both the pilot study and the follow-up study. This time, indole was detected in the chromatograph as the cause for the fecal odor.

The burnt odor was detected in the pilot study and described as distinctive to sludge drying odors. It is again detected in the follow-up study. However, carbonyl sulfide peak was not seen from the chromatograph. For future studies, carbonyl sulfide should be tested using Olfactory GC-MS for confirmation. As indole and methyl mercaptan odor was detected from GC-MS results in the follow-up study, the concentration of these two chemicals should be analyzed and confirmed as causative chemicals for these odors.

Keywords

Odor profile method, sludge drying, sludge drying odors, sludge drying odor wheel.

2.1 Introduction

Safe disposal and management of wastewater sludge is one of the most critical issues at every wastewater treatment facility (Singh et al., 2020). Wastewater sludge can contain heavy metals, biodegradable organic matter, microorganisms (pathogenic bacteria and viruses), and organic contaminants (e.g. pesticides) and Contaminants of Emerging Concern (CEC) (e.g., pharmaceutical products, synthetic hormones, etc.) that tend to concentrate in the sludge and present risks to human and environmental health (Santos et al, 2022). Also, the emission of foul odor at each stage of handling sludge has created a negative social impact associated with reusing sludge for fertilizer application (Gherghelet al., 2019).

Initially, incineration of sludge was considered the solution, but due to high production volumes of sludge, the focus of sludge disposal has shifted towards land application and the development of other valuable products. Countries such as France, Spain, and Denmark have shown a high percentage (>50%) of sludge utilization for agriculture purposes (Singh et al., 2020). According to the Directive 2000/60/E.C. of the European Union, sludge is no longer considered a waste material but a "product" of wastewater treatment. This aligns with the idea of the circular economy framework (Santos et al., 2022; Gherghelet al., 2019). Nevertheless, the high moisture content of sludge, which causes high transport costs, malodors, and storage difficulties, hampers all sludge management operations (Gherghel et al, 2019).

It is imperative to mitigate hazards and the odor from sludge to enable its reuse as a potential resource. For example, France is one of the European Union (E.U.) that has limits that restricts the amount of metals in sludge as part of the directive of the E.U. Directive 86/278 (Kelessidis & Staniskis, 2012). The U.S. focuses on pathogen content by classifying sludge into Classes A and B under the "Title 40 Code of Federal Regulations, Part 503". Class A requires pathogen densities to be reduced to below the detection limits for biosolids applied to lawns, home gardens, or other types of land use. Title 40 states that Class B sludge have a fecal coliform density of 2 million MPN or CFU per gram total solids biosolids (dry weight basis) (Lu et al., 2012).which regulates sludge disposals and biosolids reuse (Lu et al., 2012). Public access is restricted for Class B application since Class B sewage sludge still contains considerable pathogens, while public access is not restricted to land amended biosolids that meet Class A requirements (Lu et al., 2012).

The most common fate of wastewater sludge in the U.S. is to land application as Class B biosolids, which benefits soils by adding nutrients, increasing organic matter content, and improving water holding capacity (Elektorowicz et al. 2019). Although there have been several studies on the sludge drying process, these studies mainly focus on equipment design, drying efficiency, energy recovery, and potentially hazardous volatile organic chemical emissions (Ding et al., 2015). The odor produced during the drying process and their relationship with the substances in the sludge and drying temperature have not been carefully considered.

During sludge treatment, transformations such as physical and chemical/biochemical reactions can occur, which may lead to color, odor, and texture changes of the material. The drying processes include natural (e.g., solar drying) or artificial heat. Within artificial drying, convection, conduction, and radiation are the methods to provide heat flow (Santos et al, 2022). Additionally, the odor of sludge can be affected by the properties of the sludge depending on the composition of the input effluent, seasonality factors and the treatment level at the specific Wastewater Treatment Plants (WWTP). Many wastewater treatment plants in the US have developed regulations that require the industry to pretreat the wastewater before sending to a wastewater treatment plant e.g. Orange County Sanitary District, in Fountain Valley, California. This is done for control of sludges and especially for water that are eventually further treated for water reuse projects.

The odor from sludge has been a persistent odor problem from Wastewater Treatment Plants (WWTPs). WWTPs are not good neighbors if odor complaints and even community opposition comes from the area surrounding the plant. (Hayes et al., 2014). The production and treatment of wastewater sludges has been shown to a major part of the odor problem as reviewed by Fisher et al, (2019). Figure 1 shows the occurrence of dewatering and drying in a general sludge treatment process. Direct drying of sludge occurs with direct contact of the sludge with the heat source, which could be hot air or superheated steam, and acts as the conveying medium. Direct drying has the advantage of drying all types of sludge, including primary, secondary, and mixed digested sludge. Unfortunately, the investment and operation cost of direct drying process are high, and the energy consumption of equipment operation is large causing unpleasant odors (Xue et al., 2022).

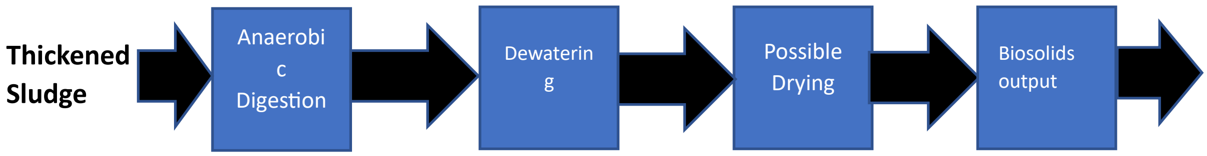


Figure 2.1. Schematic diagram of the Sludge Treatment Process

Dewatering is an energy-intensive process but an important step after anerobic digestion to improve waste to useful end products from wastewater sludge (Chen et al., 2013). The two dominant technologies in wastewater sludge dewatering are belt filter presses and centrifuges. Dewatering increases the dry solids of sludge from 3-5% to 15-30% (Metcalf and Eddy, 2002).

Thermal drying is a worldwide process used in combination with dewatering methods for sludge volume reduction and stabilization. However, practical application demonstrates the emission of intense and offensive odor as off-gases from sludge drying during low-temperature thermal drying process and dried sludge, especially for sludge without digestion or other stabilization pre-treatments (Zhu et al., 2022). Removal of odors remains a major objective of each sludge treatment and plays an important role as an index to measure the stabilization extent of all wastewater sludge processes. Accordingly, identification of the odor emission characteristics of sludge is important in the evaluation of sludge quality and disposal options (Gao et al., 2022). Thus, a scheme to identify the odors and the primary chemicals that produce the odors should be characterized to enable develop the correct treatment of the odors released from these processes.

Suffet et al., (2008) identified odors in 33 dried sludge samples from a Sludge Drying Plant in Lemay, France during 2006 and how the odors from the raw sludge changed during the simulated sludge drying process developed the beginning of a sludge drying odor wheel. A further paper in 2010 developed the Sludge Drying Odor using a laboratory pilot simulation of the sludge drying process with sludge from the Limay Plant. This paper confirmed odors and their relationship to chemicals presented in Figure 2. Thus, the odor wheel shows that the major odor detected were fishy/ammonia, sulfur/cabbage/garlic, fragrant/fruity, and burnt odors. Both methods identified other odors that are on the odor wheel which are presented as well. Also, Low-temperature thermal drying treatment was shown to change the odor categories and increase the odor intensity of some odors from high levels for raw sludges to higher levels for dried sludge.

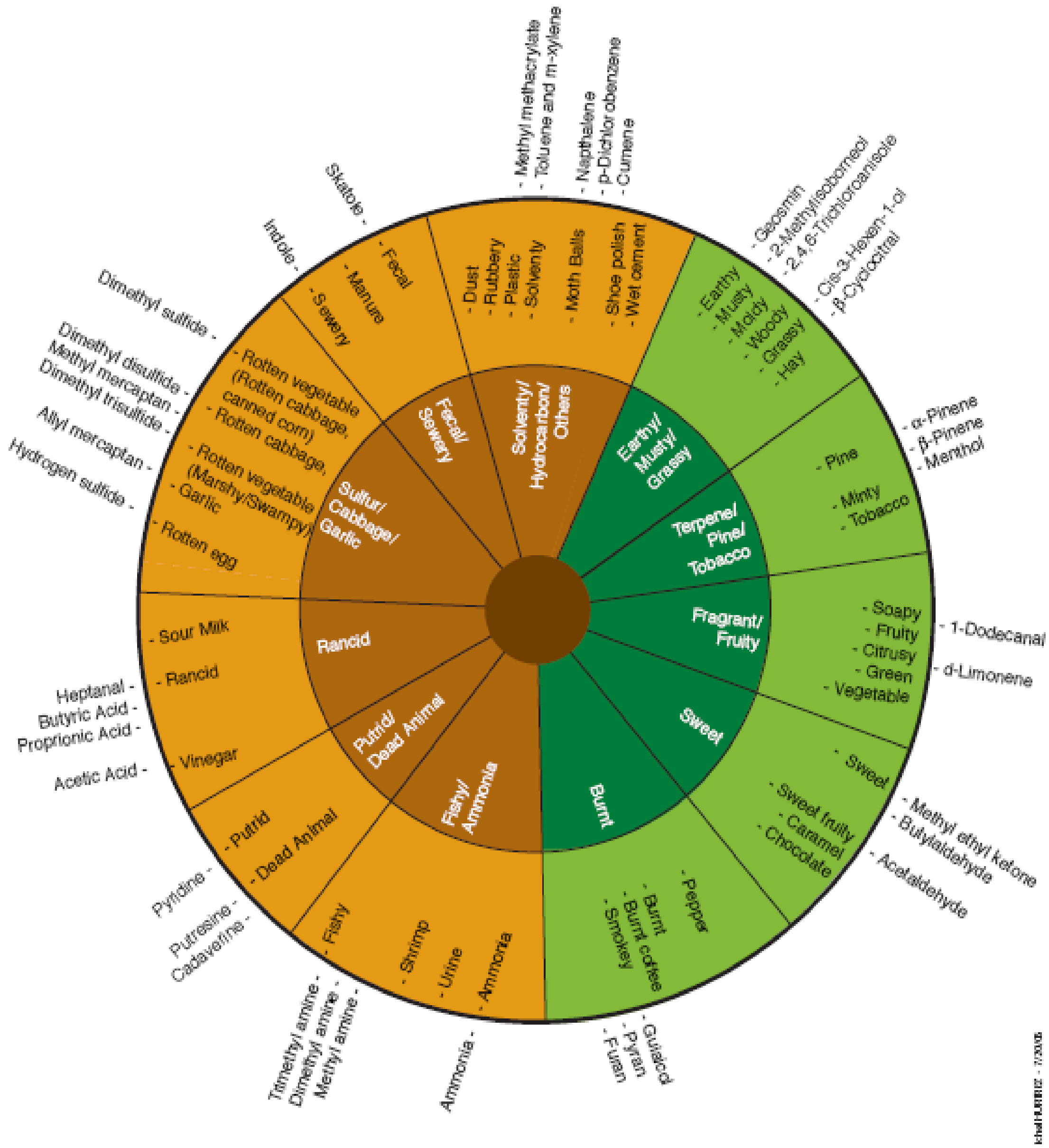


Figure 2.2. The Sludge Drying Odor Wheel (Decottignies, Bruchet, & Suffet, 2010)

Fisher et al. (2018) expanded the Sludge Drying Odor Wheel into a Biosolids Processing Wheel from a study of biosolids processing from 8 wastewater treatment plants with different unit operations in and around Syndey, Australia. Emissions from the sludge/biosolids were sampled at ambient conditions (15–25 °C) using dynamic flux hoods. For odor analysis, Odor Detection Ports

(ODP) coupled to gas chromatography– mass spectrometry (GC–MS/O), also referred to as sensory GC/MS analysis, were used to identify odorants in addition to other chemical analysis instruments. Fisher et al., (2018) further extended the odor characteristics and chemicals that could cause odors from sludge drying.

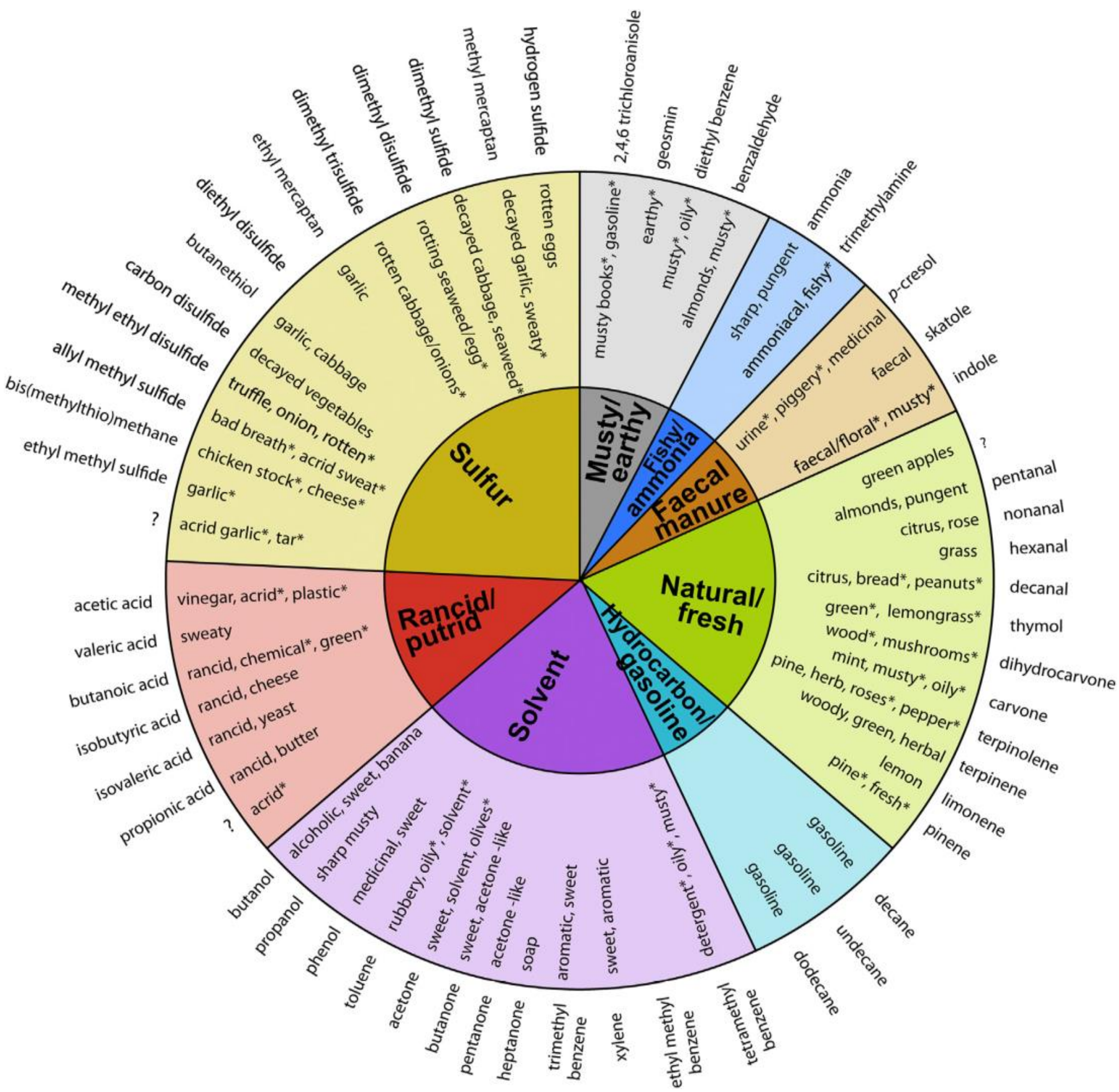


Figure 2.3. Biosolids Processing Odor Wheel (Fisher, Barczak, Suffet, Hayes, and Stuetz, 2018)

The objectives of this paper are to show an optimized laboratory pilot system to follow the evolution of specific odor character and intensity of the odors produced and identify the primary chemicals associate with those odors using gas chromatography/mass spectroscopy GC/MS instead of selected chemical methods previously used (Decottignies et al., 2010). Also, the initial sludge and the final product sludge was able to be monitored as well.

A comparison of the updated laboratory pilot sludge dewatering process was completed on sludge from the Orange County Sanitation District (OCSD) in Southern California, USA. During the laboratory pilot process, the odor of the initial sludge and the final dried sludge was compared to the emission of odorants during the process. For maximum reuse of the sludge pretreatment of the wastewater entering the OCSD WWTP, industrial uses must pretreat their waste for toxic metals and specified organic chemical pollutants.

This study will compare the similarities and differences of odors produced during and after the anerobic sludges from WWTP processes in Paris, France (Decottignies et al., 2010) to the sludge produced from the OCSD in Southern California, USA. During the updated laboratory pilot process, the odor of the initial sludge and the final dried sludge will be compared to the emission of odorants during the process.

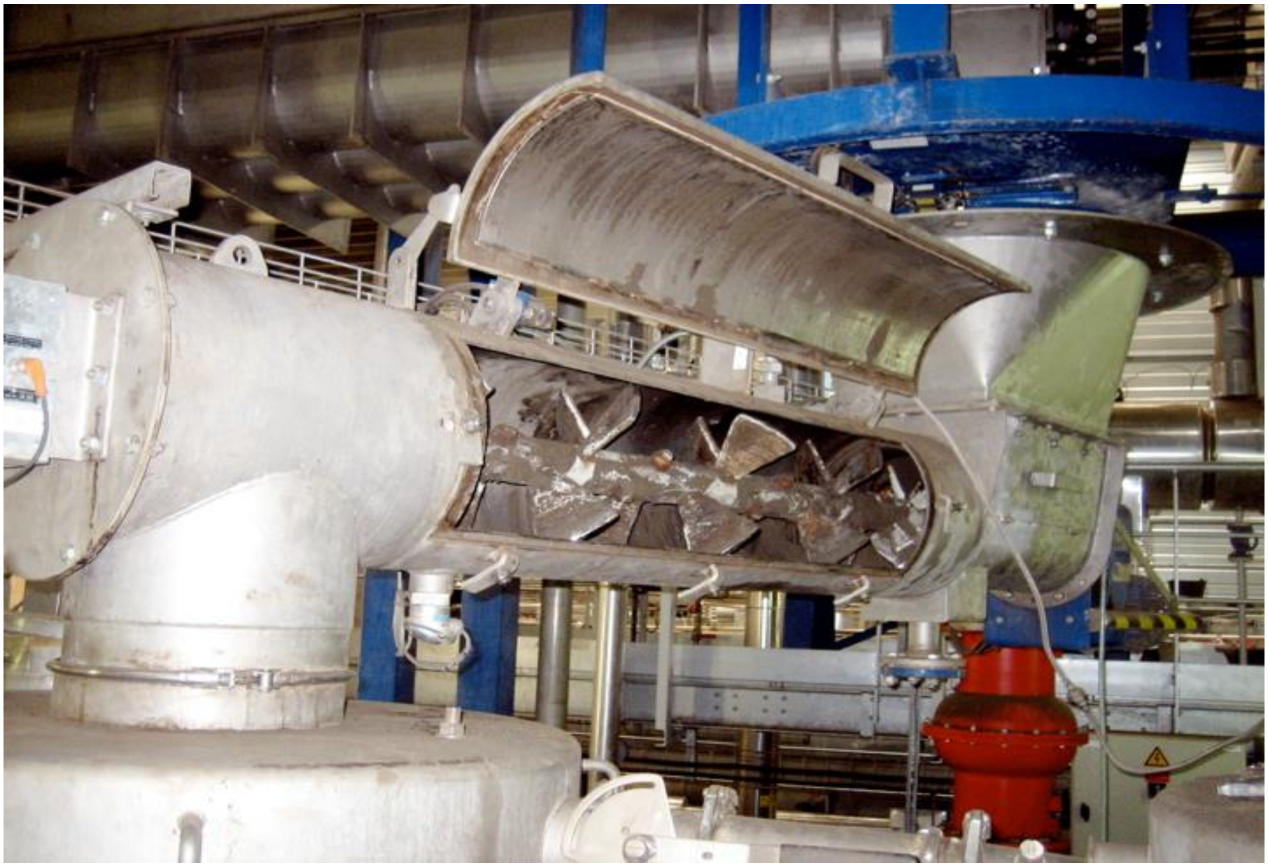


Figure 2.4. The Lemay "Screw Type" Sludge Drying Process

The emission of rancid, earthy, and other types of malodorous volatile organic compounds from wastewater sludge or biosolids has been associated with, for instance, volatile organic sulfur compounds, volatile fatty acids, 2-MIB, and geosmin (Zhu et al., 2022). Malodorous Volatile Organic Compounds have the capacity for increased odor emission in dried sludge. The major odorants of sludge after drying processes included 2-MIB, geosmin, dimethyl disulfide (DMDS), dimethyl trisulfide (DMTS), hexanal, and decanal. These compounds do not have to occur in high concentrations to cause odor nuisance due to their low Odor Threshold Concentrations (OTCs) and unpleasant odor characters (Zhu et al., 2020).

The measuring of odor includes sensory analysis and instrumental analysis. Sensory analysis can be used to identify the odor concentration or odor intensity and odor characters subjectively reflecting the degree of odor impact (Gao et al., 2022). The sensory analysis involves using the Odor Profile Method (OPM). The OPM is the primary odor detection method. Then the chemical analysis by gas chromatography/mass spectroscopy (GC/MS), as well as the use of a sniffing port as a Sensory Analysis detector (GC-Sensory Analysis) are used to identify the odorous chemicals from the sludge drying process. The results from OPM were compared with GC/MS and GC-Sensory analytical results to determine the chemicals causing the primary odors.

2.1.1 Objectives of the Study

The primary goal of this dissertation is to develop a better knowledge that can control odors emitted during the sludge drying process. The objectives of this paper is the development and evolution of the laboratory pilot systems to simulate and evaluate the odors produced during the sludge drying process. During the laboratory pilot process, a comparison of the odor of the initial sludge and the final dried sludge can also be compared to the emission of odorants during the process. This paper compares the odors produced from the anerobic sludge from WWTP processes in the Lemay Sludge Drying Plant outside of Paris, France to the sludge produced from the Orange County Sanitation District in Southern California, USA.

2.2 Materials and Methods

2.2.1 Odor Profile Method (OPM) to Determine Odor Character and Odor Intensity

It is necessary to have a consistent language for odor definition and an understanding of the possible chemical compounds responsible for controlling odor nuisances. The OPM was used to categorize typical odors for the odor wheel and their intensities by odor panels as well as to help identify the chemical odorants that define those odors characteristics by chemical analysis methods. (Burlingame, 2004 and 2009). The OPM analysis is not a threshold technique. The OPM scale is based upon the Flavor Profile Analysis in Standard Method #2170, (APHA, 2012) that is used as sensory panel methods in drinking water odor control studies (Suffet et al., 2019). The ambient OPM consists of at least 4 well-trained people who sniff odorous gas samples collected in Teflon bags generated during the sludge drying process. The odor panelist works independently at first. A discussion period is used to develop a panel consensus afterward. Both individual results and the consensus are recorded. The odor panel used the OPM with the Sludge Drying Odor Wheel (Figure 2) to describe odor characters and the Odor Intensity Scale for each odorant to report odor intensity (Burlingame, 1999; Curren et al., 2014).

The Weber-Fechner curves which relate the OTC in the air to the odor intensity of a chemical concentration must be defined to evaluate the odor nuisance for the OPM. Weber observed that increased stimuli are relative to the previous amount experience and his student, Fechner (1859) described this relationship that has been used by Suffet et al., (1995a) and Greenman et al., (2005). The intensity of an odor characteristic is a measure of its odor strength, which is related to the log of its concentration via the Weber-Fechner Equation (1):

$$I = k \, \mathrm{Log}(C/C_o) \qquad (1)$$

The intensity (I) is proportional to the logarithm (base 10) of the concentration (C) relative to a previous concentration (C_o). Figure 5 shows the Weber–Fechner Curve. For each odor character, the OPM method develops an odor intensity that is related to the log concentration of the odorous chemical. The odor intensity scale has 7 points from 1 to 12; 1 (threshold), 2, 4 (recognition), 6, 8, 10, and 12. At the odor threshold intensity of 1, an odor panelist can state that the odor is different than clean air. Only at an odor intensity of 4 can the panelist recognize odor characteristics on the odor wheel, Figure 3. The OTC is formally described as the point where 50% of a population (minimum of 4 panelists) can define a difference between pure air and air containing an odorant. The odor recognition threshold is where 50% of an odor panel can define the type of odor from the odor wheel.

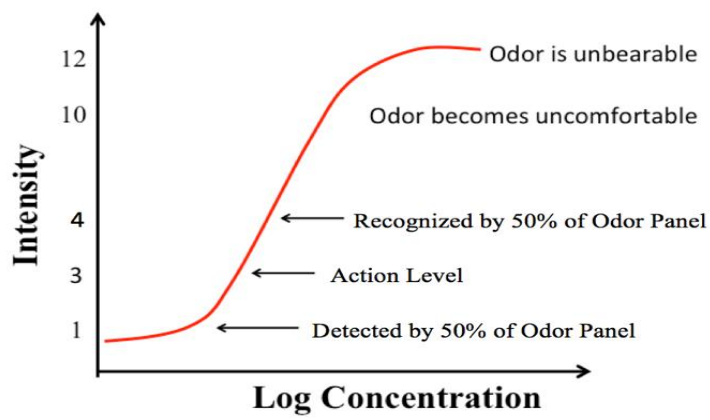


Figure 2.5. Intensity Scale for Each Odor by Weber-Fechner Curve

2.2.2 *Analytical Methods*

Chemical analyses of off-gases from the laboratory pilot model for sludge drying (Decottiginies et al., 2010) included GC-NPD for volatiles amines, GC-Electrochemical detection for sulfur compounds, GC-MS for volatile organic compounds and GC-FID for volatile fatty acids and GC/MS with GC/Sensory Analysis for Broad Spectrum analysis. From previous sludge odor studies, some chemicals have been identified with its odor. Thus, it is easier to confirm if those chemicals are present in this study.

2.2.3 Experimental Set Up

The pilot study that used the Lemay plant's sludge drying temperature to understand odors that are produced from the sludge drying process (Decottiginies et al, 2010) were replicated in the present study. The final temperature of the sludge driers averages 102°C with a range of (95 – 112). Three runs of the drying processes were performed for the sludge sample on Aug 11th, Aug 16th, and Aug 17th, 2020. The present follow-up study used the sludge collected from OCSD, California on May 17th, 2020. The sludge was refrigerated at 4ºC until used. Three runs of 210 minutes drying process were performed each time for the sludge samples. Seven off-gas samples were collected by connecting an air pump to a 3-liter Teflon bags each 30 minutes to 210 minutes. Teflon bags were used because of their stability for chemical storage (Zhou, 2017). There is formation of condensation in the bag which can affect the odor panel results. Therefore, the condensate was separated from the off-gases before collecting the air samples and the total condensate for the complete sludge drying simulation process was analyzed as well.

Figure 2.6 shows the rotary-evaporator system that was used to obtain off-gas and sludge product odor information for both the Lemay and OCSD studies. It consists of a rotating flask containing the sludge in a temperature adjustable oil bath. The highest temperature reached was 125°C, which is the temperature close to the highest temperature attained from Lemay Plant. A typical temperature curve for the sludge drying process was developed as shown in Figure 2.7. The weight of the sludge used was 60 g and the sludge was placed in a 500 ml round bottom flask.

A team of well-trained OPM panelists sniffed the off-gas samples and dried sludge samples for odor character and determined the odor intensity of each odorant, on the same day as each experimental run. A Varian 220MS-450GC were used for GC/MS analysis. A solid-phase micro-extraction (SPME) method was used to identify and quantify odorous chemicals (Godayol et al., 2013). A SPME fiber of DVB/CAR/PDMS was injected into the 3-L sample bags and exposed for 20 minutes. The SPME fiber was injected onto a Varian 450 GC (Varian Inc., Palo Alto, California) through an #1177 liquid-injector port (Zhou et al., 2017).

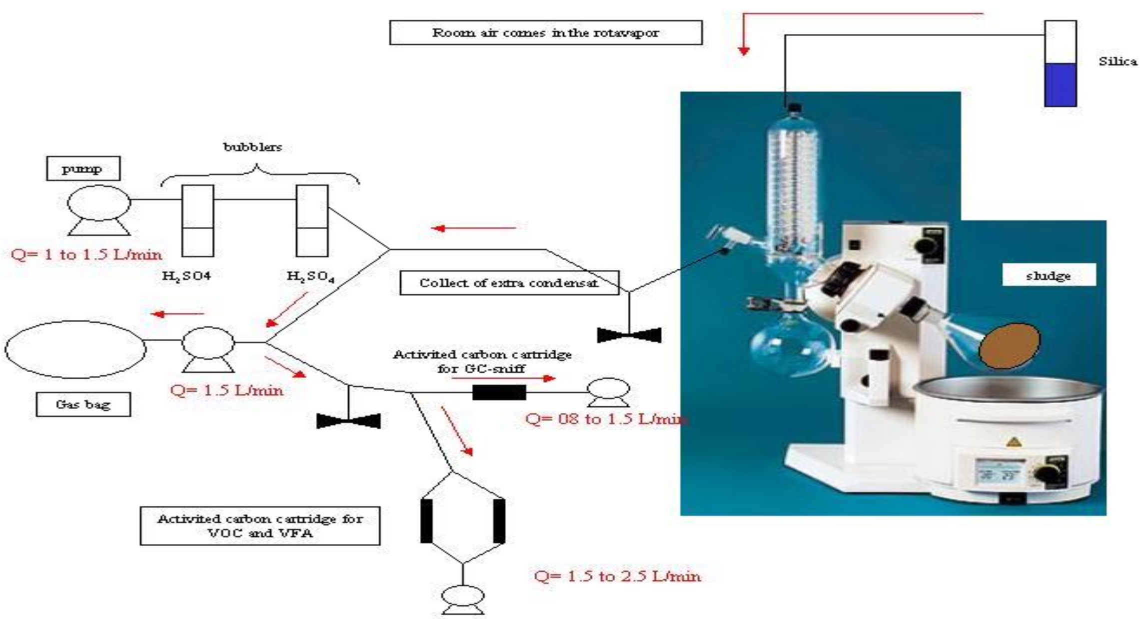


Figure 2.6. Pilot Laboratory Equipment Setup used at Limay, France

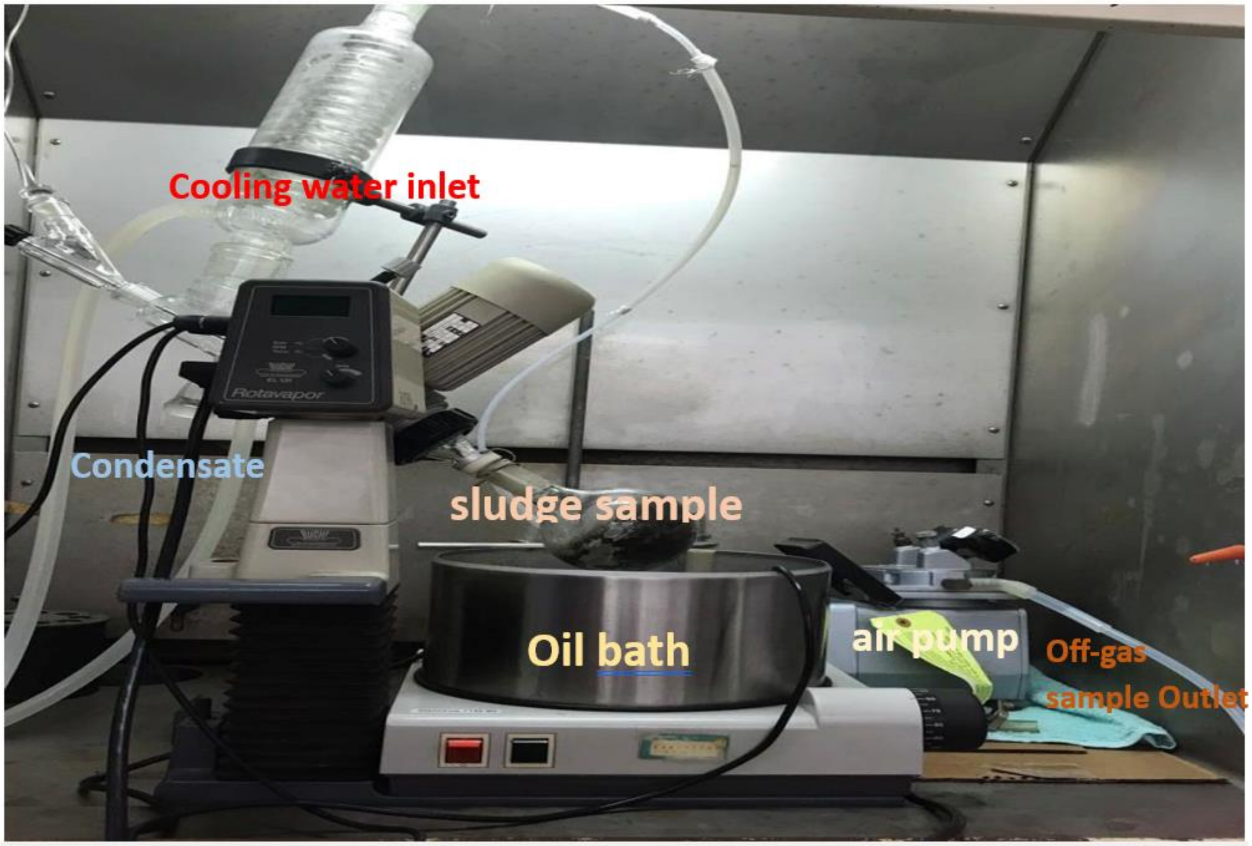


Figure 2.7. The Laboratory Rotary-Evaporator Sludge Drying System used at UCLA

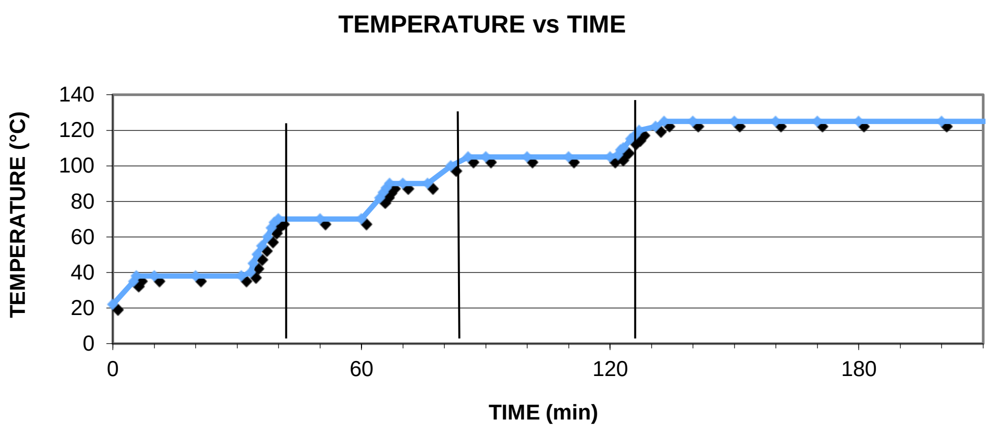


Figure 2.8 Temperature-Time Curve for the Sludge Drying Process in Figure 2.7.

2.3 Results and Discussion

2. 3.1 OPM Data of the Off-Gases from the Sludge Drying Process at Limay (Decottignies et al., 2010)

The primary odors observed from the off-gases at different times from the Limay sludge drying samples in 2006 were:

1) Rotten Vegetables in all three trials.

2) Fecal, Manure, Burnt and Ammonia in 2 of the 3 trials and

3) Earthy, Rancid and Grilled Meat in 1 of the 3 trials (Table 1-3).

As indicated on the Odor Intensity Scale, Figure 3, the OPM odor intensity data that is near or larger than 3 indicates a potential odor nuisance to neighbors of the Lemay Plant. This can be observed to occur though out the data set.

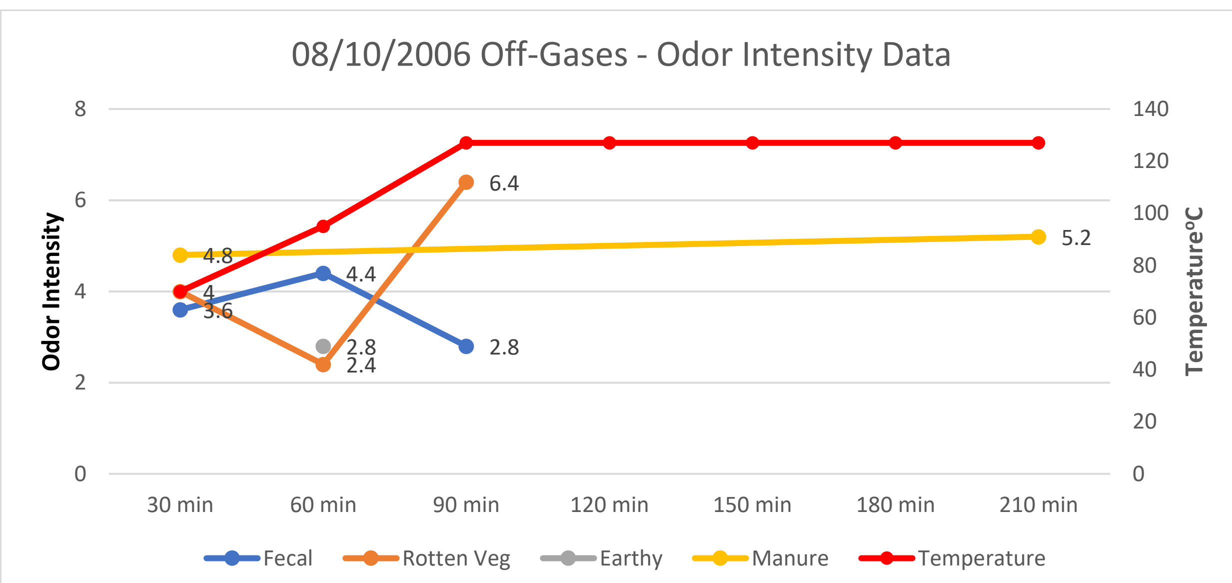


Figure 2.9. 08/10/2006 Off-Gases – Odor Intensity

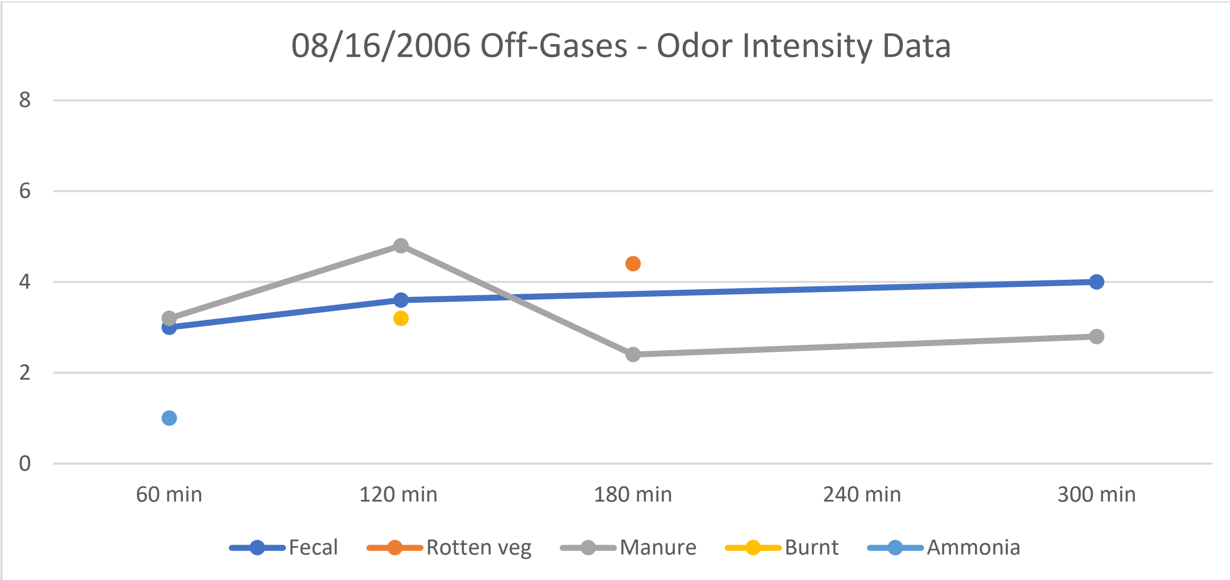


Figure 2.10. 08/16/2006 Off-Gases – Odor Intensity

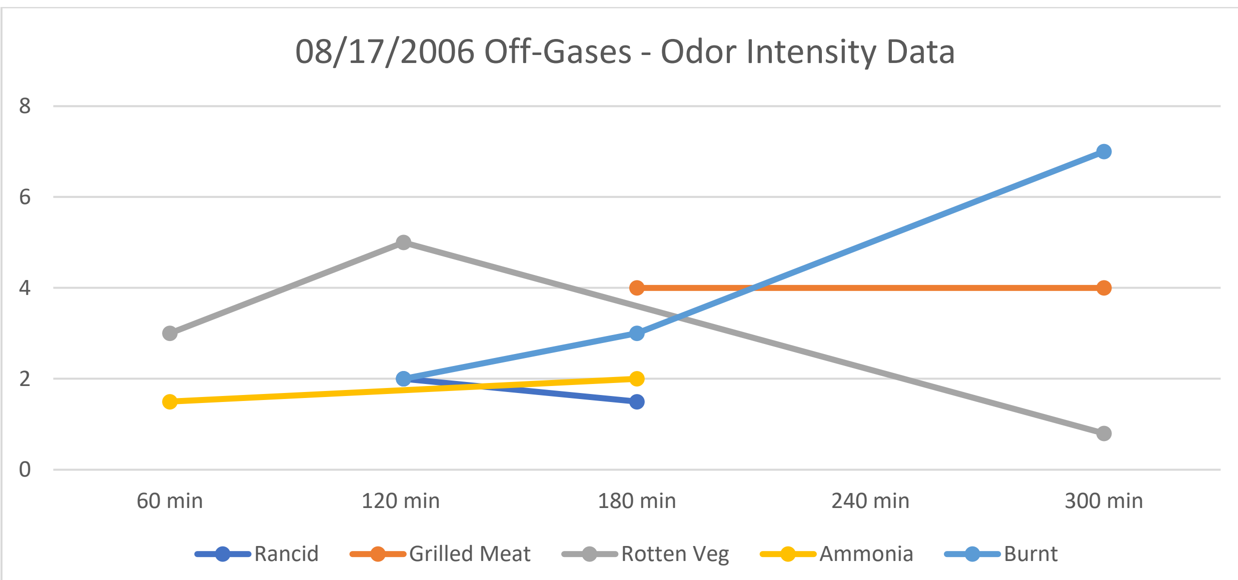


Figure 2.11. 08/17/2006 Off-Gases – Odor Intensity

2.3.2 OPM Data of the Off-Gases from Drying Process of Sludge Sample from OCSD in 2020

The primary Odors observed from the off-gases at different times from the OCSD sludge samples in 2020 were:

1) Fecal and Burnt in all three trials;

2) Rotten Vegetables, Earthy and Caramel in 2 of the 3 trials

3) and Sweet, Ammonia and Rancid in only 1 of the 3 trials (Table 4-6).

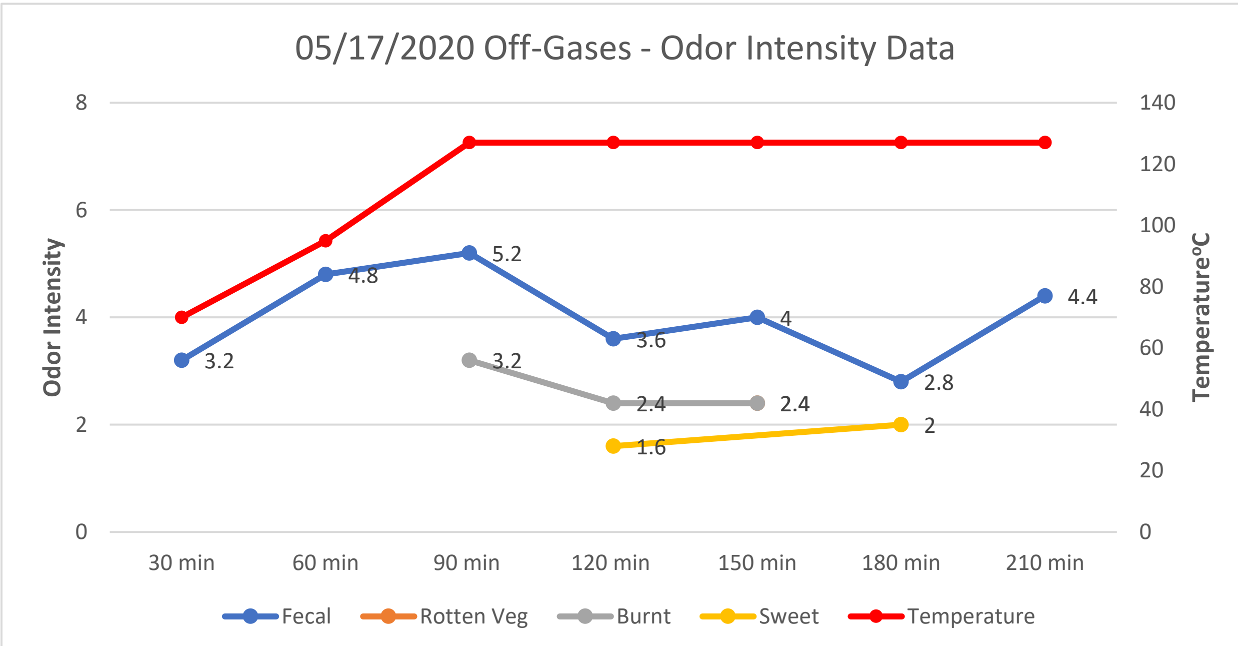


Figure 2.12. 05/17/2020 Off-Gases – Odor Intensity

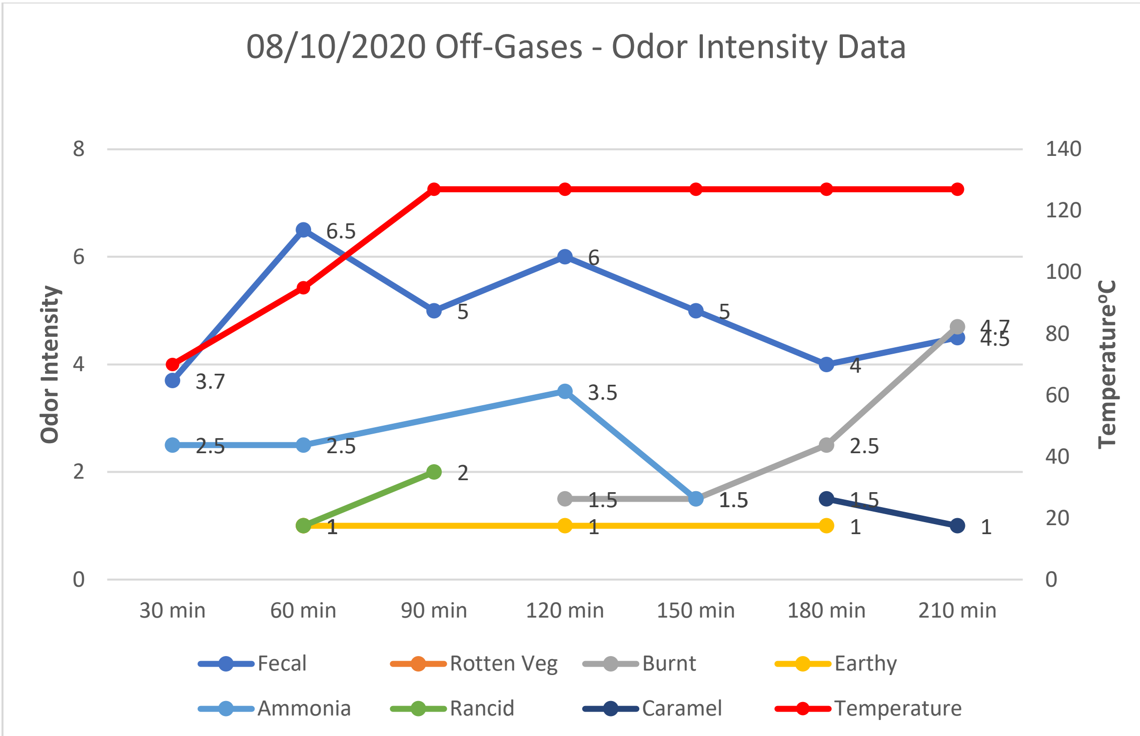


Figure 2.13. 08/10/2020 Off-Gases – Odor Intensity

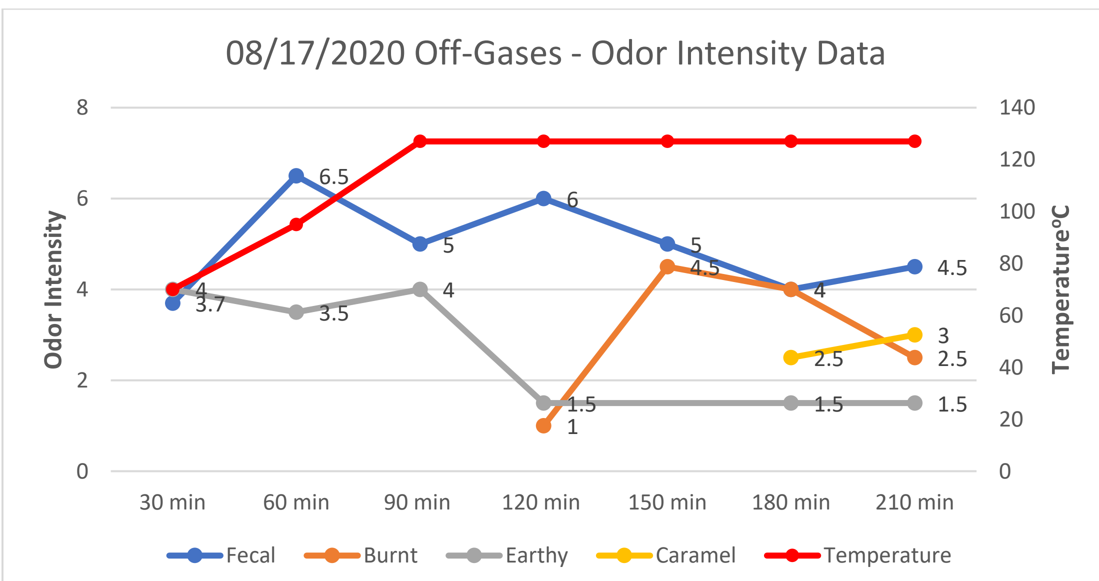


Figure 2.14. 08/17/2020 Off-Gases – Odor Intensity

2.3.3 Similarities of Odors Occurring in Both Studies

Rotten vegetable, fecal and burnt odor were the primary similar odors occurring in both studies. Rotten vegetable occurred in three trials while fecal and burnt odor occurred two trials of the pilot study. While in the follow- up study, fecal and burnt odor occurred in all three trials and rotten vegetable in two of the trials for the study. Other odors that were similar in both studies include: earthy, ammonia and rancid. Odors unique to each plant were:

1. Lemay Plant: Manure and grilled meat odor
2. Orange County Sanitation District: Sweet and caramel odor

Caramel and sweet odors are desirous odors for the sludge drying process and should be further investigated to understand its production process and compounds responsible for the odors. It should be noted that the burnt category was distinctive to sludge drying and not observed as a primary odor in wastewater, compost, or any other environmental odor wheel (Suffet et al, 2007).

2.3.4 Odorous Chemicals Related to Sludge Drying Process

Many of the odorous chemicals were associated with odors from previous odor studies at different facilities, e.g. wastewater, compost and landfill. However, it is still necessary to confirm which chemicals contributes to the sludge drying odors by chemical analysis of the sludge samples in the sludge drying process. Figure 2 shows the chemicals associated with each of the odors on sludge drying wheel.

From the pilot study and the follow-up study, some of the chemicals were identified from the off-gases samples as the odor-causing chemicals (Table 7). Odorous off-gases samples were analyzed by sensory-GC and GC/MS for the verification of the odorous chemicals. Some of the target odorous chemicals determined included indole (fecal), and methyl mercaptan, dimethyl disulfide, dimethyl trisulfide (rotten vegetable), the primary odors at both plants. Hydrogen sulfide, butyric acid, ammonia, limonene, acetic acid, and 2-butanone were also defined and are relate to other odors on the Sludge Drying Odor Wheel, Figure 2.1.

Table 2.1. Chemical detected from sludge drying off-gases

Primary Chemical Reported in the literature that is associated with the odor	Related Odor	Chemically Detected in Limay Pilot Study	Chemically Detected in OCSD Pilot Study	Odor Perceived in Study
Indole	Fecal	YES	YES	YES
Methyl Mercaptan	Rotten Vegetable	YES	YES	YES
Dimethyl Disulfide	Rotten Garlic – Egg			NO
Dimethyl Trisulfide	Rotten Vegetable	YES	YES	YES
Hydrogen Sulfide	Rotten egg			NO
Dimethyl Sulfide	Rotten Vegetable	YES	YES	YES
Butyric acid	Rancid	YES	YES	YES
Isobutyric acid	Rancid	YES	YES	YES
Ammonia	Ammonia	YES	YES	YES
Trimethylamine	Fishy	NO	NO	NO

Limonene	Lemon	NO	NO	NO
Acetic acid	Vinegar	NO	NO	NO
2-butanone	Sweet, sweet-solvent	YES	YES	YES
Carbonyl sulfide	Burnt	YES	YES	YES
Acetone	Solvent	NO	NO	NO

Most of the results were also similar to other studies. According to Zhu et al., (2020) they found that dimethyl sulfide (DMS) and dimethyl disulfide (DMDS) were the most significant MVOCs for the mixture of primary and secondary thickened sludge with concentrations of 7.9–28.5 and 7.1–496.1 μg/m^3. The concentration of DMS and DMDS in sludge dewatering workshops could reach 3.9–103.8 and 5.0–32.5 μg/m^3, respectively. The odor concentrations increased 2.3–28.4 times when the mixture of primary and secondary thickened sludge was pre-dewatered by centrifugation (Zhu et al., 2022). The emission of MVOCs during sludge conditioning with PAC, $FeCl_3$+CaO, acidizing, or Fenton reagents increased independently and was attributed to the soluble extracellular polymeric substances content. Based on the frequency of detection and odor activity values (OAVs), methyl mercaptan (MeSH), p-cresol, and butanoic acid were identified as the key odorants from wastewater sludge after dewatering and during storage (Zhu et al., 2022).

In a study by Gao et al., (2022), a total of 20 odorants were identified and quantified, including 6 groups of chemicals, among which volatile sulfur compounds (VSCs), indole, 3-methylindole and geosmin were identified as key odorants. The odor of the dewatered digested sludge was improved by means of changing the odor character from fecal/sulfide to earthy odor due to the reduction in VSCs concentration (Gao et al., 2022).

Since sludge drying off-gas samples are very complex, not all odor related chemicals were detected. The chemical might be below the GC/MS instrument detection limit. Therefore, many chemicals that were believed to be related to some of the sludge drying odors have not been confirmed yet.

2.3.5 Chemical Analysis to Confirm Odor for Lemay Pilot Study

The Sulfur gases that produce the Rotten Vegetable Odor, (methyl mercaptan, dimethyl sulfide, dimethyl disulfide, and dimethyl trisulfide) were qualitatively identified at Limay and OCSD. The study at Limay quantified these chemicals over the heating of the sludge from 20ºC to 120ºC as shown in Figure 6. The rotten vegetable odor perfectly corresponds with the OPM data from the Limay pilot study where the rotten vegetable odor was perceived in all three trails. However, panelists could not distinguish rotten vegetable odor with rotten egg or rotten garlic odor of the sulfur odors. The sulfur compounds start to evolve from the sludge drying process at 60-80 mins probably because of their slow formation and volatility.

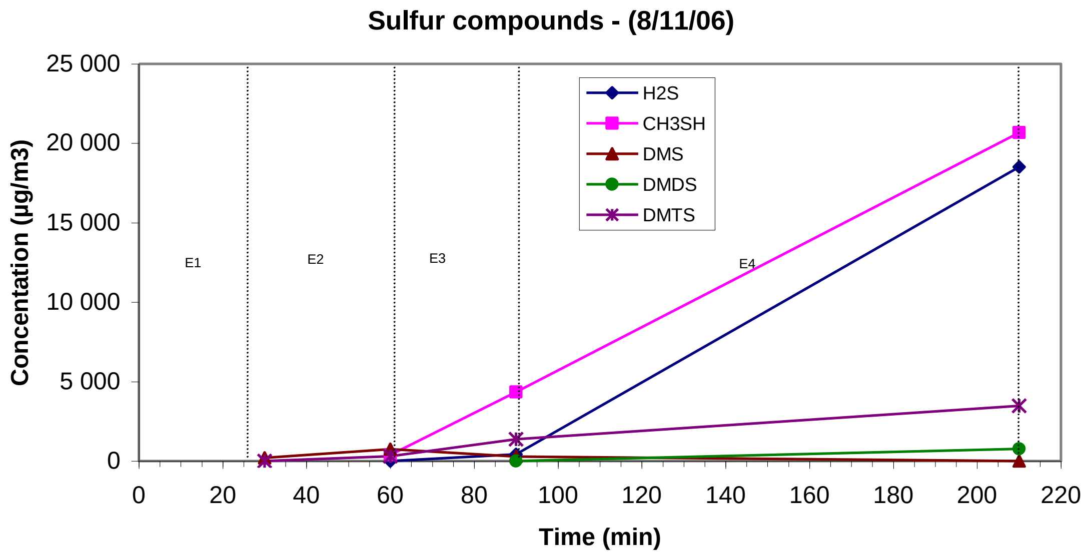


Figure 2.15. Evolution of five Sulphur compounds during the pilot study at Limay.

2.4 Conclusion

The primary goal of this research was to confirm odors detected from pilot sludge drying simulation process from the sludge drying plant at Limay, France in 2006 could be reproduced in 2020 with OCSD sludge. This study first examined the consistency of odor character of the sludges, the relationship between typical malodors and drying temperature of the sludge. This study is unique because the pattern of the change of odors during the drying process was observed and how the odors change over drying time.

The primary odorants found in at least 2 of 3 studies at each plant were consistent: Rotten Vegetables, Fecal and Burnt. Other odors were observed such as Earthy, Rancid and Ammonia odorants were found at both plants a total of 3 times out of 6 times. Manure and Grilled Meat (Limay) while Caramel and Sweet (OCSD) were distinct to each plant, probably due the source of the waste sludges. It should be noted that the burnt category was distinctive to sludge drying and not observed as a primary odor in the wastewater, compost, or any other environmental wheel (Suffet et al, 2007).

Indole was the primary chemical confirmed that causes the fecal odor. The primary sulfur compounds (methyl mercaptan, dimethyl sulfide, dimethyl disulfide, and dimethyl trisulfide) were confirmed to develop the rotten vegetable type odors. However, some of the odors can be masked if another odor is very strong. For example, the rancid odor was not perceived in many samples, yet chemical analysis showed many rancid chemicals were observed by GC/MS. This was probably an odor masking phenomenon. Further studies could investigate the chemicals responsible for caramel and sweet odor and the evolution of these chemicals during sludge drying.

To reduce odor emission during the sludge drying process, this study recommends that WWTPs conduct a pilot system weekly to monitor odors emitted during the sludge drying process and any changes in sludge source arriving at the drying plants. Optimally, monitoring the primary and secondary odors through a pilot lab system can help drying plants control or reduce temperature or drying time to effectively reduce odors emitted. Moreover, sludge drying plants unable to run a pilot operation would collect samples from different time periods and run the Odor Profile Method as described in this paper. The OPM helps describe both primary and secondary odors occurring which would help with odor management.

Reference

APHA (American Public Health Association), 2012. Standard methods for the examination of water and wastewater: Method 2170, 22nd ed. APHA, the American Water Works Association (AWWA), and the Water Environment Federation (WEF), Washington, DC, USA

Burlingame, G. A. 1999. Odor profiling of environmental odors. Water Sci. Technol. 40(6), 31-38.

Burlingame, G. A. 2009. A practical framework using odor survey data to prioritize nuisance odors. Water Sci.Technol. 59(3), 95-602.

Burlingame, G.A., Suffet, I.H., Khiari, D. and Bruchet A.L. (2004). Development of an odor wheel classification scheme for wastewater. Wat. Sci. Tech., **49**(9), 201-209.

Curren, J., Snyder, C.L.C., Abraham, S., Suffet, I.H., 2014. Comparison of two standard odor intensity evaluation methods for odor problems in air or water. Water Sci. Technol. 69, 142-146.

Decottignies, V., Bruchet, A. Link, M. Aupetitgendre, M. and Suffet, I.H., 2010. Dried Sludge Odors: Classification and Case Study. Proceedings of the Water Environment Federation. Odors and Air Pollutants 2010, pp. 72-82

Decottignies, V., Bruchet, A. Suffet, I.H. (Mel) (2010), Dried sludge odours : classification and case study, Paper Paper submitted to Water Envronment Federation “Odor and Air Pollution Conference”. IWA world Water Congress, Vienna, Sept 7-12.

Fechner, G. (1859). Elemente de Psychophysik, Leipzig University, Germany.

Gao W., Yang X., Zhu X., Zhao X., Yu J., Wang D., Yang M. (2022). The variation of odor characteristics of wastewater sludge treated by advanced anaerobic digestion (AAD) and the contribution pattern of key odorants. Science of The Total Environment, Volume 840, 2022, 156722, ISSN 0048-9697. https://doi.org/10.1016/j.scitotenv.2022.156722.

Gherghel A., Teodosiu C., De Gisi S. (2019)A review on wastewater sludge valorisation and its challenges in the context of circular economy, Journal of Cleaner Production, Volume 228, Pages 244-263, ISSN 0959-6526, https://doi.org/10.1016/j.jclepro.2019.04.240.

Godayol,A., Alonso, M., Sanchez, J.M., Anticó E 2013. Odour-causing compounds in air samples: gas e-liquid partition coefficients and determination using solid phase microextraction and GC with mass spectrometric detection. J. Sep. Sci. 36, 1045-1053.

Greenman, J., El-Maaytah, M., Duffield, J., Spencer, P., Rosenberg, M., Corry, D., Saad, S., Lenton, P., Majerus, G., Nachnani, S., 2005. Assessing the relationship between concentrations of malodor compounds and scores from judges. JADA. 136, 749-757.

Hayes, J.E., Stevenson, R.J. and Stuetz, R.M., 2014. The impact of malodour on communities: A review of assessment techniques. Science of the Total Environment, 500, pp.395-407.

INRS (2005). Comparaison des seuils olfactifs de substances chimiques avec des indicateurs de sécurité utilisés en milieu professionnel, *Note documentaire ND 2221-198-05*

Kelessidis A., Stasinakis A. S. (2012*) Comparative study of the methods used for treatment and final disposal of sewage sludge in European countries, Waste Management, Volume 32, Issue 6, Pages 1186-1195,ISSN 0956-053X*, https://doi.org/10.1016/j.wasman.2012.01.012.

Lu Q., He Z. L., Stoffella P. J. (2012) "Land Application of Biosolids in the USA: A Review", *Applied and Environmental Soil Science*, vol., Article ID 201462, 11 pages, 2012. https://doi.org/10.1155/2012/201462

Mallevialle J. and Suffet I.H., 1987. Identification and Treatment of Tastes and Odors in Drinking Water, ISBN 0-89867-392-5. AWWA Research Foundation and Lyonnaise Des Eaux Cooperative Research Report, American Water Works Assoc., Denver, Colo., USA.

Rosenfeld, P.E., Clark, J.J., Hensley, A.R. and Suffet, I.H. (2007) The use of an odour wheel classification for the evaluation of human health risk criteria for compost facilities. Wat. Sci. Tech., **55**(5), 345-357

Santos A. F., Gomes L. A, Góis J. C., Quina M. J. (2022). Sewage sludge drying to produce soil amendments using adjuvants: Toward a circular economy, Editor(s): Maulin P. Shah, Susana Rodriguez-Couto, Nidhi Shah, Rajarshi Banerjee, Development in Waste Water Treatment Research and Processes, Elsevier, Pages 433-456, ISBN 9780323855846, https://doi.org/10.1016/B978-0-323-85584-6.00024-8.

Singh V., Phuleria H. C., Chandel M. K. (2020) Estimation of energy recovery potential of sewage sludge in India: Waste to watt approach, Journal of Cleaner Production, Volume 276,122538,ISSN 0959-6526, https://doi.org/10.1016/j.jclepro.2020.122538.

Suffet, I.H. (Mel), S. Braithwaite, Y. Zhou. Bruchet, A. “The Drinking Water Taste-and-Odor Wheel After 30 Years, Chapter 2. *In “Taste and Odour in Source and Drinking Water: Causes, Controls, and Consequences” Editors*: T-F. Lin, S. Watson, and I.H. (Mel) Suffet, Publisher, International Water Association.

Suffet, I.H. (Mel), Decottignies, V., Senante, E., Bruchet, A. (2008). Assessment and characterization of odor emission during sludge drying processing. Paper submitted to the

Suffet, I.H., Mallevialle, J., Kawczynski, E., 1995. Advances in taste-and-odor treatment and control. American Water Works Association.

Van Gemert L.J. (1999) Compilations of odour threshold values in air and water. Boelens Aroma Chemical Information Services (BACIS), The Netherlands

Zhou, Y., Vitko, T., Suffet, I.H., 2017. Identification of Musty Odor Nuisance at Wastewater Treatment Plants. In Review

Zhu X., Yang X., Gao W., Jiao R., Zhao S., Yu J., Wang D. (2022) Effect of low-temperature thermal drying on malodorous volatile organic compounds (MVOCs) emission of wastewater sludge: The relationship with microbial communities, Environmental Pollution, Volume 306,119423, ISSN 0269-7491,https://doi.org/10.1016/j.envpol.2022.119423.

Zhou, Y., Thesis, UCLA, Los Angeles CA (2017): The Identification, Analysis, and Treatment of Odor Nuisance Released from Wastewater Treatment Plants

Chapter 3

Understanding Odor Emissions from the Sludge Drying Process and Byproduct Reuse Potential

3.0 Abstract

Sludge drying from wastewater treatment can cause an odor nuisance for neighboring communities. This study characterized the nuisance odor emissions to develop odor control strategies to minimize the malodor emission during a sludge drying process of digested wastewater sludge. The digested sludge used in this study was from the Orange County Sanitation District (OCSD) Wastewater Treatment Plant (WWTP), at Fountain Valley, CA. Standard experimental methods were carried out to examine the consistency of odor character, analyze the relation between typical malodors and drying temperature and determine the pattern of the change of odors during the drying process. A new approach to understanding the odor produced by the sludge drying process was simulated by creating artificial undigested and digested sludge recipes to see if the same sludge drying odors could be replicated.

The primary odors detected were consistent with a previous study, including the burnt odor. This study indicated that fecal and musty/earthy odors were also important odors. A caramel odor was detected as a new odor within the burnt odor category. The sludge composition that contributes to each odor were more easily studied by simulating a sludge digesting recipe. Digested sludge (described as raw sludge) that was added into a sludge recipe containing fresh fats, protein, and carbohydrates successfully simulated the anaerobic digestion processes of the sludge. The odors produced were much closer to the actual odors from the sludge drying process than the recipes without adding digested sludge. The overall data for WWTP sludge shows odor variation due to the varying sludge composition and microbial activity within the sludge.

3.1 Introduction

Rising population growth and rapid urbanization has made efficient wastewater and sludge management a primary goal of public and government environmental organizations (Nunez et al, 2022). Sludge, an inevitable by-product of wastewater management accounts for 50% of the carbon footprint and over 50% of the cost of running a wastewater facility (Gherghel et al., 2019). Tendencies in environmental management have evolved from eliminating sludge wastes to recycling and valorizing them in the pursuit of circular economy (Gherghel et al., 2019).

Sludge is prone to decaying and emitting odors even under natural environmental conditions because the major content of sludge is organic substances (Cui et al. 2022). Approximately 40% of the total dry weight of the sludge are proteins (Cui et al. 2022). The emission of odors affects both the air quality of the surrounding environment and endangers human health. Primary odorants such as hydrogen sulfide (H_2S) and methyl mercaptan (MeSH), can cause symptoms such as dizziness and nausea, at high concentrations and at lower concentration breathing and digestion problems as well as affecting endocrine and nervous systems (Cui et al., 2022). Published literature about the sludge drying processes has focused on equipment design, drying efficiency, energy recovery, and potentially hazardous volatile organic chemical emissions (Ding et al., 2015). While the odor produced during the sludge drying process and their relationship with the composition of sludge and drying temperature have not been extensively evaluated.

Thermal drying of sludge is a necessary intermediate disposal method, as it makes it possible to stabilize the sludge, reduce its volume and sterilize the product (Deng et al., 2020). Thermal conversion of sewage sludge has the beneficial opportunity of being a ‘waste to energy option’ enabling sustainable waste disposal. Among the different methods of waste to energy conversion, the thermochemical route has many advantages over the other methods, such as high reduction in volume of sludge and lower process time. However, sewage sludge is a non-conventional type of biomass whose characteristics and behavior under various operating conditions are quite different because of its distinct composition. The various complex reactions occurring during the thermochemical conversion process depend on the physical and chemical properties of the sludge. It is necessary to have a proper understanding of the physical as well as chemical characteristics of sludge for better processing and utilization. (Raveendravarrier et al., 2020).

Understanding the chemical and biological composition of sludge is crucial to study the relationship between sludge composition and odor emissions. The major organic materias in sludge are complex mixtures of fats, proteins, carbohydrates, humic materials, fatty acids and other dissolved organic matter (DOM) (Xue et al 2022). Studies have reported that the protein in sludge can be used to prepare protein feed, organic fertilizer, biodegradable foaming agents and foam fire extinguishing agents ((Su et al. 2015; Collivignarelli et al. 2017; Gao et al. 2020b). Therefore, sludge is a potential resource, and there are reports on sludge protein extraction technology (Yan et al., 2020). The high carbon and oxygen content in sludge is mainly due to the large number of organic components, such as protein, fat, and cellulose. Protein is primarily composed of amino acids. Therefore, the sludge contains nitrogen compounds that transform into ammonia, amines, NOx, and nitrates during the sludge treatment process (Xue et al., 2022). The thermal conversion of sludge proteins/amino acids is influenced by their existing form and the co-presence of other substances such as carbohydrates (mainly cellulose, hemicellulose, and lignin). Specifically, carbohydrates significantly affect protein reactions. Different carbohydrate types have different effects on NOx production, and the conversion of biomass fuel nitrogen can be viewed as the superposition of cellulose, hemicellulose, lignin, and protein (Guo et al., 2021).

Xue et al., (2022) conducted a study using indirect and direct drying. Indirect drying showed that H2S and NH3 were the dominant odorous gases, while acetones, benzene, acetic acid, and diethylamine were other identified VOCs. The direct drying method produced a larger amount of waste gas. SO2 was the primary gas observed, while aromatics and chlorinated compounds were the dominant VOCs. Non-carcinogenic and carcinogenic risks were mainly attributed to hydrogen sulfide, methyl mercaptan, and methyl sulfide, which were released from the sludge. SO2 and 2-heptanone were also generated. Additionally, It has been reported that the NH_3 emitted from sludge drying was formed through hydrolysis of protein. When the protein in sludge dissolves, it hydrolyzes to form many peptides, dipeptides and amino acids. The amino acid further hydrolyzes to form organic acid, NH_3 and CO_2 (Deng 2009). NH3 is a toxic and corrosive air pollutant with an extremely pungent odor and a systemic inhalation irritant (Xue et al., 2022).

Primarily, available studies on drying temperature and the composition of sludge only focus on sludge transformation at high temperature (Xue et al., 2022). Studies focused on odor character and intensity of the VOCs emissions at much lower temperature during sludge drying are still lacking. Reimann (1989) showed that thermal conditioning of sludge led to partial release of its ammonia with water vapor, and Gostelow et al. (2001) reviewed odorants measurement and listed 39 typical kinds of odorants associated with sewage treatment works, including reduced sulfur bearing compounds, amine compounds, organic acids, aldehydes and ketones. However, detailed research on the actual odors emitted from sludge drying process at WWTPs and the associated VOC species during the sludge drying process were not sufficiently investigated (Xue et al., 2022).

Many studies on the odor of “final sludge dried material” have shown the highly odorous compounds present as reviewed by Fisher et al, (2019) and shown on the Odor Wheel, Figure 3.1. An initial development of a laboratory pilot system for observing the odors produced during dewatering processes of incoming sludge compared the similarities of the sludge drying process and was included in an initial Odor Wheel, (Decottignies et al., 2010). Chapter 2 shows differences of odors produced from the anerobic sludges from WWTPs in Paris, France and at Orange County Sanitation District’s WWTP using a laboratory dewatering pilot method at lower temperatures. It was observed that there were variabilty of odors that occurred from the different sources of sludge. The similar odors emitted include fecal, rotten vegetable, earthy, burnt, rancid and ammonia. The composition of the raw sludge entering Limay plants differs from that at OCSD as odors of sweet and caramel odor were observed during the pilot plant tests at OCSD. The composition of the raw sludge entering Limay plants differs from that at the OCSD as odors of manure and grilled meat odor were observed during the pilot plant tests at the Limay Plant only.

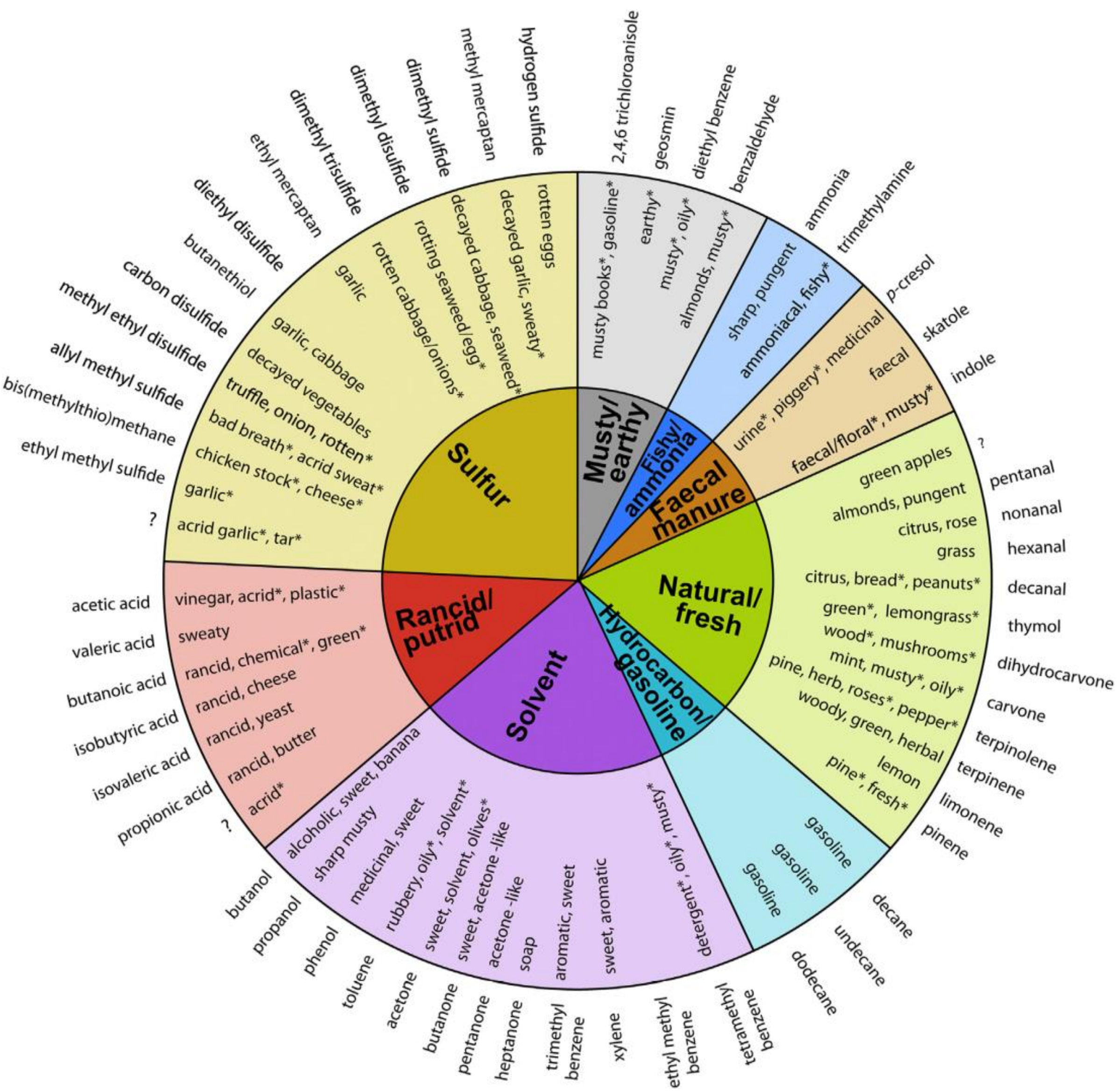


Figure 3.1. Biosolids Processing Odor Wheel (Fisher, Barczak, Suffet, Hayes, and Stuetz, 2018)

The objective of this research was to understand the mechanisms of how the “nuisance odors” were generated during the sludge drying process. Then minimization strategies to reduce odors can be instituted. Various artificial sludge recipes were created to understand how the composition of sludge evolves during drying at low temperatures and how the odors emitted are transformed. This work is needed before process changes and/or subsequent air treatment methods can be developed to control odor nuisances. A byproduct of the work could further verify the value of the laboratory simulation process to test if a sludge drying process could be used for a particular sludge.

3.2 Experimental Method

3.2.1 Odor Profile Method (OPM) to Determine Odor Character and Odor Intensity

To control odors, it is necessary to have a consistent language for their definition and an understanding of the possible chemical compounds responsible. The Odor Profile Method (OPM) was used to categorize typical odors from the odor wheel and their intensities by odor panels as well as to help identify the chemical odorants that define those odors characteristics by chemical analysis methods. (Burlingame, 2004 and 2009). All trained panels use the OPM and the sludge drying odor wheel of Figure 3.1 to identify the odor character.

The Weber-Fechner curves relates the OTC in air to the odor intensity of a chemical concentration in air to evaluate the odor nuisance for the OPM. Weber observed that increased stimuli is relative to the previous amount experience and his student, Fechner (1859) described this relationship that has been used by Suffet et al., (1995a) and Greenman et al., (2005). The intensity of an odor characteristic is a measure of its odor strength, which is related to the log of its concentration via the Weber-Fechner Equation (1):

$$I = k \text{ Log}(C/C_o) \qquad (1)$$

The intensity (I) is proportional to the logarithm (base 10) of the concentration (C) relative to a previous concentration (C_o).

The odor panel used the OPM with the Sludge Drying Odor Wheel to describe odor characters and the Odor Intensity Scale for each odorant to report odor intensity (Burlingame, 1999; Curren et al., 2014). The OPM scale is based upon the Flavor Profile Analysis in Standard Method #2170, (APHA, 2012) that is used as sensory panel methods in drinking water odor control studies (Suffet et al., 1987). A minimum of 4 trained panelists were used to analyze all odorous air samples.

Figure 3.2 shows the Weber–Fechner Curve. For each odor character, the OPM method develops an odor intensity that is related to log concentration of the odorous chemical. The odor intensity scale has 7 points from 1 to 12; 1 (threshold), 2, 4 (recognition), 6, 8, 10, and 12. At the odor threshold intensity of 1, an odor panelist can state that the odor is different than clean air. Only at an odor intensity of 4 can the panelist recognize odor characteristics on the odor wheel, Figure 1. The OTC is formally described as the point where 50% of a population (minimum of 4 panelist) can define a difference between pure air and air containing an odorant. The Odor Recognition Threshold (ORC) is where 50% of an odor panel can define the type of odor from the odor wheel.

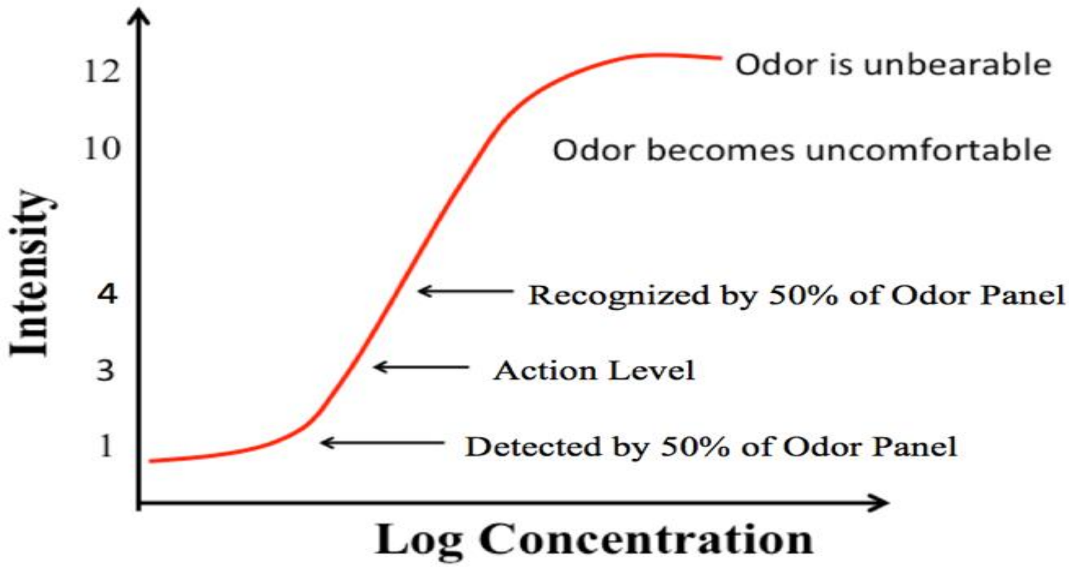


Figure 3.2. Intensity Scale for Each Odor by Weber-Fechner Curve

3.2.2. *Experimental Set Up*

The laboratory experiment method was constructed following the design of Decottignies et al. (2010) modified in modified in chapter 2. The laboratory pilot plant was designed to be like an industrial sludge drying process. Figure 3.3 shows the rotary-evaporator system that consists of a rotating flask containing the sludge in a temperature adjustable oil bath. The highest temperature reached was 125°C, which is the temperature close to the highest temperature attained during a typical sludge drying facility. In this pilot study, a typical temperature curve for the sludge drying process was developed as shown in Figure 4 similar to the one in Decottignies et al (2010).

3.2.3 Sludge Drying Repetitive Runs

Two different sludge samples were obtained from the OCSD. A team of well-trained OPM panelists sniffed the off-gas samples and dried sludge samples for odor character and determined the odor intensity of each odorant, on the same day as each experimental run.

Set I: Sludge sample A was obtained from OCSD Plant I on Feb 27th, 2021 and refrigerated at 4^{o}C until used. A 210-minute simulation of the drying process for Sludge A was completed on April 7, 2021. Seven off-gas samples were collected by connecting an air pump with the 3-liter Teflon bags of 9,000 mL/min from 30 minutes to 210 minutes, respectively. Since, the formation of condensation in the bag can affect the odor panel results, the condensate was separated from the air before collecting the air and the total condensate was analyzed as well.

Set II: A new sludge sample B was obtained from OCSD Plant I, on May 10, 2021. The same procedures were used as described for Sludge A, on May 17, 2021.

Set III: A series of sludge dryings processes were performed on sludge B to determine the odor of the sludge itself after different drying periods of 30, 60, 90, 120, 150, 180 and 210 minutes. Each dried sludge sample was contained in the glass flask covered by aluminum foil. After cooling to room temperature, each dried sludge sample was sniffed by the OPM odor panel for odor character and intensity.

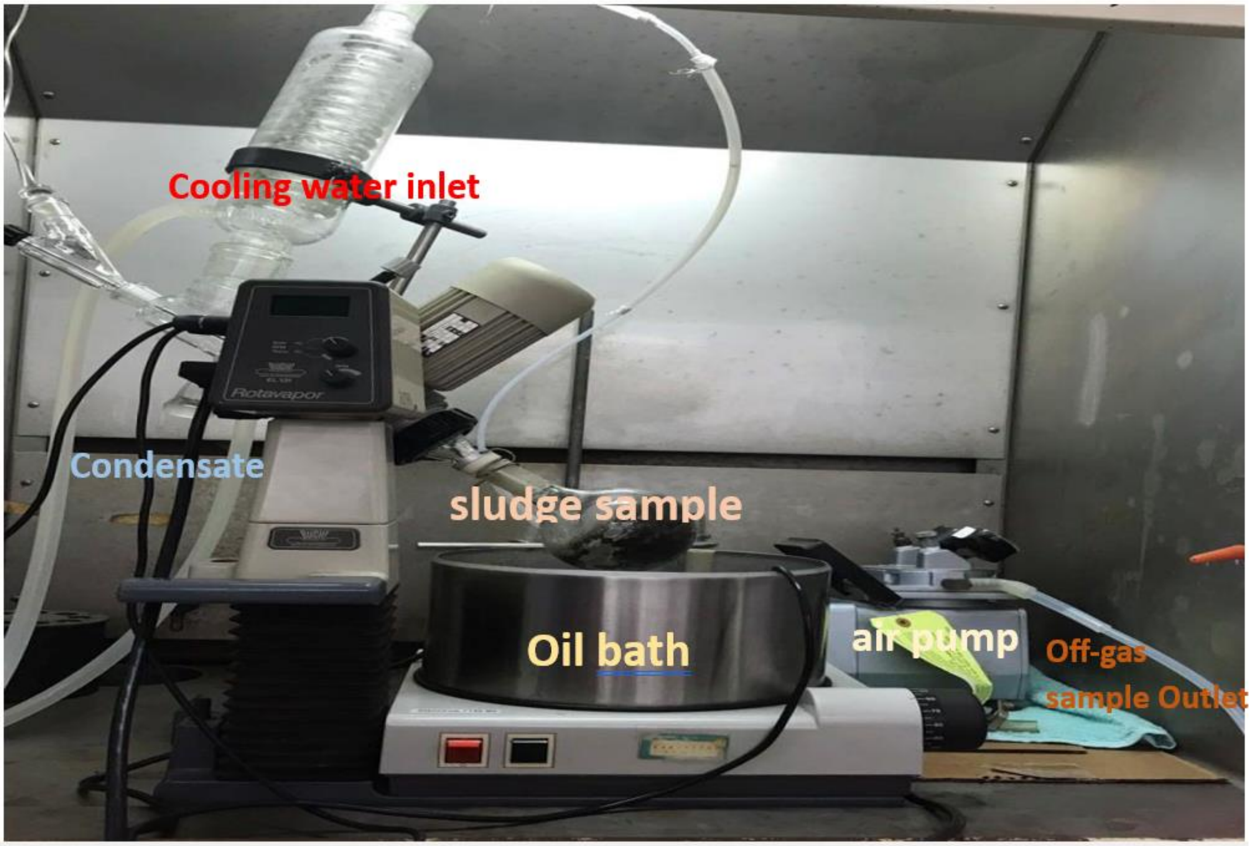


Figure 3.3. The Laboratory Rotary-Evaporator Sludge Drying System (Decottignies et al., 2010)

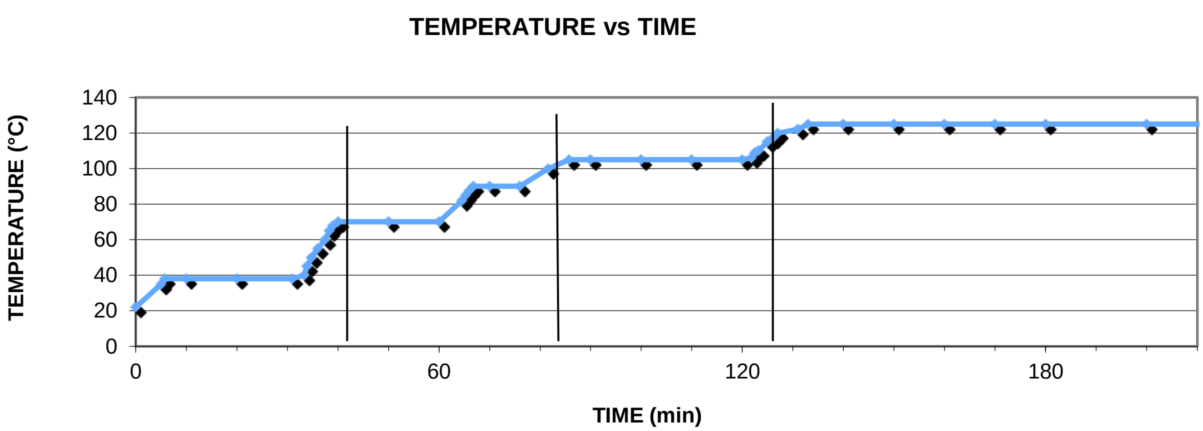


Figure 3.4. Temperature-Time Curve for the Sludge Drying Process in Figure 3.

3.2.4 Sludge Simulation

A set of sludge simulations were developed to determine which component in the sludge caused the primary odors observed during the sludge drying process. The basis for selecting components was using natural soil or soil components, e.g. tannin and lignin, and a representative protein, fat and carbohydrate. It was hypothesized that if this is known then one can determine the total proteins, carbs and fats in a sludge sample as one does for food labelling. Then maybe the digestion process and the sludge dryng methods can be optimize for control of those processes and as well control the odor.

Set IV: A simulated sludge was made by adding together 5 grams of tannin (Tannic Acid, Fisher Scientific Corporation, LOCATION), 5 grams of lignin (Lignin, TCI Corporation, Location) to 10 grams of natural topsoil from a southern California natural garden that had no fertilizer added, (366 23rd Street, Santa Monica CA) to observe the odors from background soil components of sludge. The same 210-minute sludge drying process was performed on April 20, 2021 on this synthetic sample. The samples were collected and analyzed same as Sets I-III.

Set V: A simulated sludge was made by adding together 9 grams of whey protein (Unflavored whey protein, Jarrow Formulas (SOURCE?) 9 grams of lard (i.e. fat) (Premium Manteca, Farmer John Corporation), and 9 grams of brown sugar (i.e. carbohydrates) (Pure Cane Sugar, C&H Corporation) with 33 grams of natural soil. The same 210-minute sludge drying process was performed on May 19, 2021. The samples were collected and analyzed same as Set I-IV.

Set VI: A simulated sludge was developed for this study by adding 9 grams of whey protein, 9 grams of lard, 9 grams of brown sugar and 3 grams of raw sludge into 33 grams of natural topsoil. The simulated sludge was set in the hood at room temperature in a closed container to allow the protein, carbohydrates, fats, to anaerobically decay as it would in the sludge from a WWTP for 11 days. The same 210-minute sludge drying process was performed on July 13, 2021. The samples were collected and analyzed just as Set I-V.

3.3 Discussion and Results

3.3.1 Consistencies of Odor Types Determined from Sludge A and B from OCSD vs. Sludge from Paris France (Chapter 2).

The primary odors detected from the off-gases of Sludge A and B fall into the following groups:

- Fishy /ammonia
- Sulfur /cabbage /garlic
- Rancid
- Fragrant fruity
- Burnt

The above results confirm the results by Decottignies et al., (2010). It should be noted that the burnt category was distinctive to sludge drying and not observed as a primary odor in wastewater, compost, or any other environmental wheel (Suffet et al, 2007). The release of ammonia in sludge is determined by the drying temperature. Weng et al., (2011) observed that above 220 °C, the release of ammonia increases and at 100 °C, more than 80% of the released gases are chain alkanes, followed by aromatic hydrocarbons and cycloalkanes. From the final dried sludge, Decottignies et al., (2010) also determined odors similar to shrimp, burnt coffee, and smoky. However, in this experiment, one other group were noted as primary odors that was not found in Decottignies' study such as: Sweet/fragrant.

Thus, it was concluded that using a different sludge source could cause variability in the type of odor emitted. This could be a result of sludge composition. The ratio of each component of sludge might affect the odor given off during sludge drying.

3.3.2 Consistency of FECAL Odors Intensity Between the Off-gases of sludge Samples A & B

Figure 5 shows that in Sludge A and B, the fecal odor tends to be more intense during the first 90 minutes of the drying process. The fecal odor also has a higher intensity in the condensate and is relatively low in the final dried sludge. This indicates that the chemicals possible indole causing the fecal odor are removed by volatilization but not destroyed in the sludge drying process. Thus, an air treatment for chemicals such as indole would be needed to help mitigate the fecal odor at a sludge drying operation.

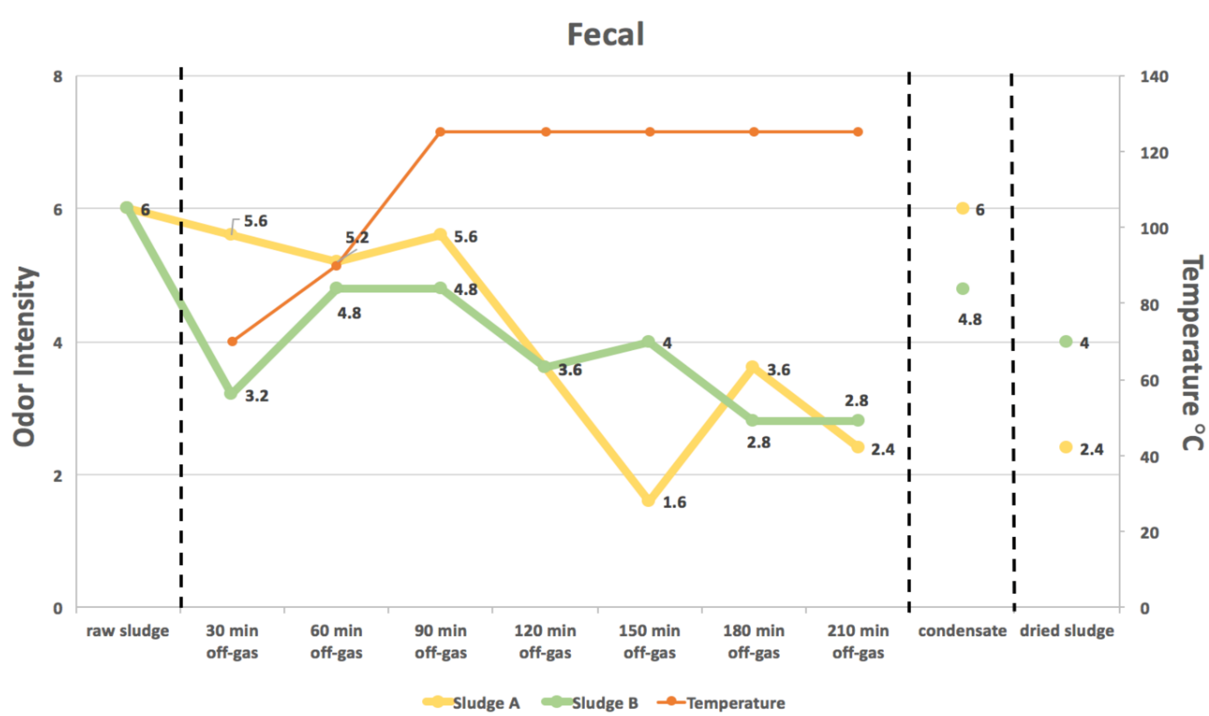


Figure 3.5. The Change of Fecal Odor in Sludge Drying Off-Gases for Sludge A & B
*Note: The number labels are average intensity values for the off gas at corresponding time. The red line represents the temperature change during the sludge drying process.

The fecal odor in the original sludge A dropped from an average Intensity of 6 to < 2.5 after the sludge drying process. However, in sludge B it only dropped from 6 to 4. The fecal odor decreased after the drying process for both sludge samples. In condensates, both sludge A and B have a high fecal odor intensity compared to the odor intensity in dried sludge. Hypothetically, as the drying process proceeds, the fecal odor escapes from sludge pile into off-gas; when off-gas encounters cold surface, the fecal odorant chemical gets transferred into the condensate.

Apparently, sludge B would need a longer processing time. This shows how a sludge batch can be adjusted for processing by slower rate of heating or a longer heating time at the highest temperature by a lab experiment. If this lab experiment is done on a site-specific basis over a few months to see the trend occurring, the optimum approach for fecal odor removal can be optimized. The reason sludge A drops to a low level at 150 minutes needs more research when optimizing the sludge drying process.

3.3.3 Consistency of BURNT Odors Intensity Between the Off-gases of Sludge Samples A & B

Figure 3.6 shows that in both sludge A and B, the burnt odor tends to appear after 90 minutes, as the sludge drying temperature reaches it maximum of 125°C during the drying process. Gao et al., (2022) suggested that burnt odors could be caused by compounds such as p-cresol, m-cresol, toluene, and xylene. The burnt odor has a high concentration in the dried sludge itself. The original sludge has no burnt odor. Thus, a biosolid treatment for burnt odor should be completed after the sludge drying process.

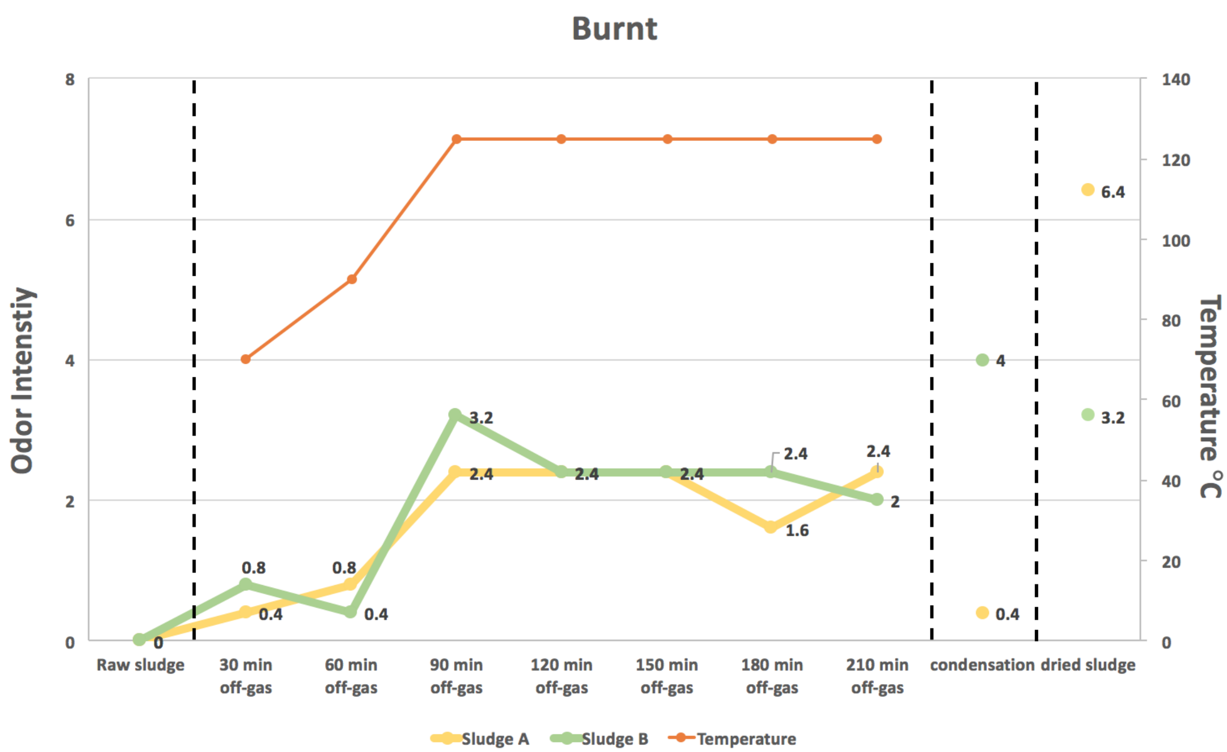


Figure 3.6. The Change of the Burnt Odor in Sludge Drying Off Gases for Sludge A & B
*Note: The number labels are average intensity values for the off gas at corresponding time. The red line represents the temperature change during the sludge drying process.

The burnt odor of the dried sludge increases over the drying time to 6.4 for sludge A. However, for sludge B, the burnt odor only increased to 3.2. Also, the intensity in the condensate is different after the drying process for Sludge A and B. Despite the differences of burnt odor in condensate, burnt odor is formed through the drying process, especially after a temperature around 125°C. For sludge A, the burnt odor is very high in dried sludge, and lower in the condensate. On the contrary, for sludge B, the opposite is true. Thus, further studies are needed to better understand the production of the burnt odor.

3.3.4 Consistency of CARAMEL Odors Intensity Between the Off-gases of Sludge Samples A & B

Figure 3.7 identified a definite caramel odor as a new odor that falls under the burnt odor category. Caramel odor is not significant throughout the drying process, but has a relatively higher intensity in the final dried sludge samples for sludge A and B. The caramel odor in dried sludge A has a slightly higher intensity of 3.6 compared to sludge B, where the caramel odor intensity is 3.2 in dried sludge. Caramel is apparently produced in the sludge drying process in the residual sludge.

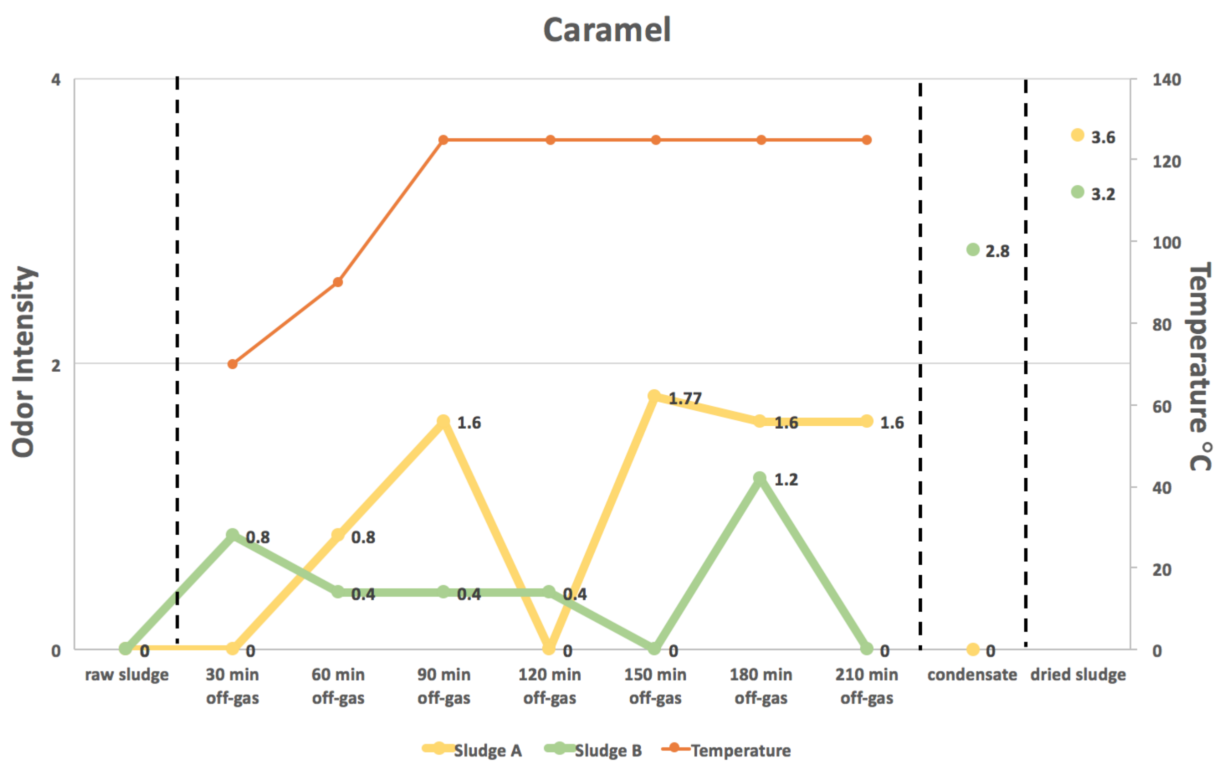


Figure 3.7. The Change of Caramel Odor in Sludge Drying for Sludge A & B
*Note: The number labels are average intensity values for the off gas at corresponding time. The red line represents the temperature change during the sludge drying process.

With temperature increasing, the caramel odor in sludge B appears earlier than sludge A. At 210 minutes of drying process, sludge A has an odor intensity of 1.6 in the off gas but has no odor in the condensate. For sludge B, there is no caramel odor in 210 minutes off-gas, but the condensate has a high caramel odor. Apparently, the caramel odor in sludge A did not condense, while for sludge B the off-gas condensed and transferred caramel odorant into the condensate. Even though the pattern for off-gas caramel odor in sludge A and B is not consistent, both dried sludge A and dried sludge B had a high caramel odor. Therefore, the main focus for caramel odor should be as a treatment of dried sludge cakes. From this perspective, caramel odor and burnt odor share a similar characteristic. The chemical causing the caramel odor is yet to be identified. Identifying the chemical causing the caramel odor is paramount so it can be used as an air treatment when drying sludge.

3.3.5 Consistency of the Intensity of OTHER Odors Between the Sludge Samples A & B

Table 1 shows the other odors and their intensities that appear in the sludge drying process for sludge A and B. For these odor types, the pattern between sludge A and B are not consistent. Fishy odors are only significant in sludge A. From an average intensity level of 4 in the off gas after 30 minutes of drying to zero in dried sludge. In sludge B, fishy odor is not significant. A sweet odor is not significant for sludge A but occurs in Sludge B. Earthy/musty odor that was observed in sludge A is only significant in the 180 minutes off gas. However, in sludge B, the earthy/musty odor was significant in both raw sludge and dried sludge. Rancid odor is only significant in raw sludge for sludge A, but no rancid odor is significant for Sludge B. Earthy/musty odor is associated with VOCs while rancid odors are caused by Volatile Fatty Acids (VFAs)

Sludge Type	Odor Type	raw sludge	30 min off-gas	60 min off-gas	90 min off-gas	120 min off-gas	150 min off-gas	180 min off-gas	210 min off-gas	coondensate	dried sludge
A	fishy		4	4.8	2.4	2	1.6		0.4	2	
B	fishy	1	0.8	2.4	2				0.4		
A	sweet					0.4		0.4	1.6	0.8	
B	sweet		1.2	0.8	0.4	1.6	0.8	2	2	3.2	4
A	earthy/musty						1.2	2		0.8	1.6
B	earthy/musty	3	0.8	0.8	1.2	1.2		1.2	0.4		1.6
A	rancid	5.5									
B	rancid	1	0.8	1.6	0.8	0.8	1.2	1.2	0.4	0.8	

Table 3.1. The Change of Other Odor in Sludge Drying for Sludge A and B *Note: number indicates average odor intensity.

In summary, there appears to be variability of odors for sludge from the same plant at different times even though domestic wastewater is always treated together. Thus, a pilot study would be valuable to complete out in the field to determine the primary and secondary odors present that would need odor nuisance control at a particular sludge drying facility at different times.

3.3.6 Fecal and Burnt Odors in the Sludge at Seven Different Drying Times for Sludge B

Figure 3.8 shows the fecal odor retained in the sludge during the drying process. Raw sludge has a fecal odor intensity of 6 which is lowered to an intensity of 2 after 90 and 120 minutes at 125°C. However, after 150 minutes or a longer drying time, the sludge residual tends to produce a higher fecal odor with intensity of 4 at 180 and 210 minutes. The burnt odor in dried sludge tends to increase with increasing drying time, to 150 minutes from an intensity of 1 to 3.5.

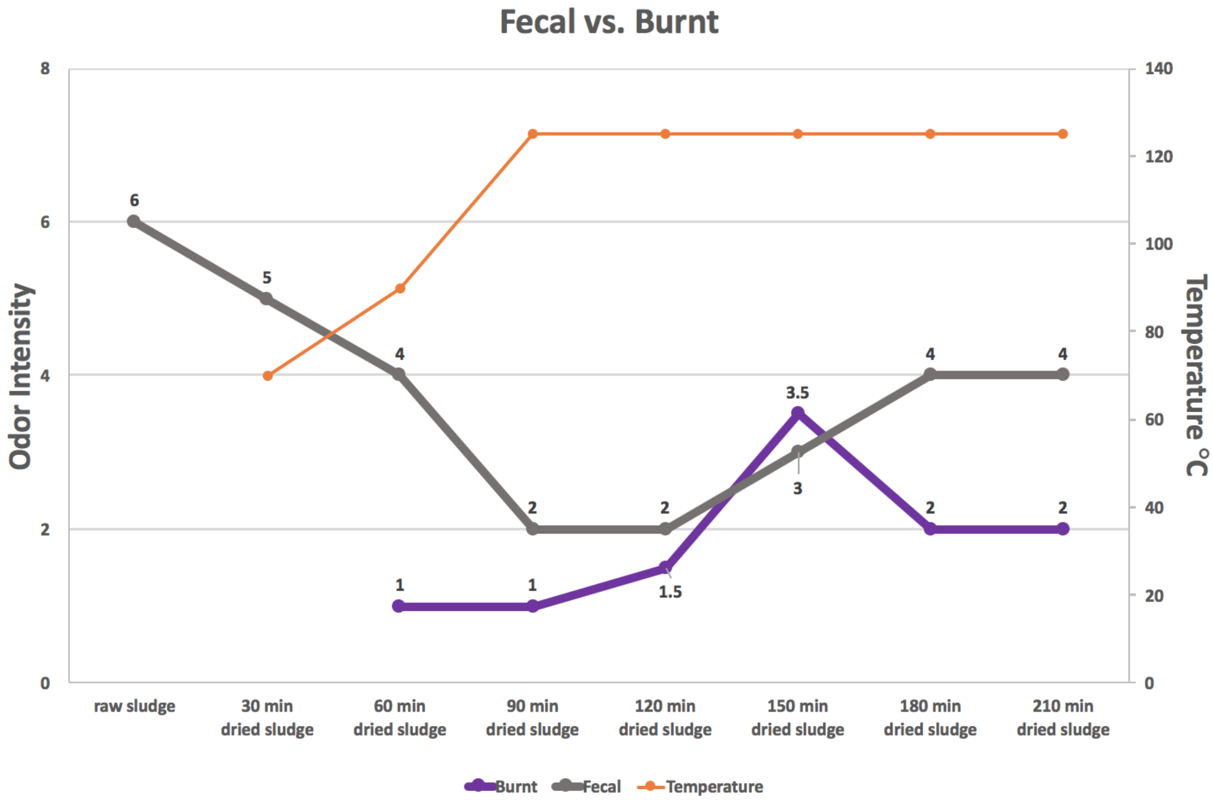


Figure 3.8. Change of Fecal & Burn odor for Dried Sludge at Seven Different Drying Duration for Sludge B

*Note: The number labels are average intensity values for the off gas at corresponding time. The red line represents the temperature change during the sludge drying process.

3.3.7 The Similarities and Differences Between the Sludge Simulation Recipes and Actual Sludge

Table 2 shows that the fecal odor present as a major odor in real sludge was not observed in the mixture recipe for Set IV (5 grams of tannin, 5 grams of lignin and 10 grams of soil) vs. the real sludge samples. The burnt odor was not significant for the mixture recipe either. The caramel odor that is in the burnt category has a high intensity in dried sample. The earthy/musty and sweet odors were present in both the SET IV recipe and sludge drying process. Since Earthy/musty and sweet odors are associated with VOCs, this might imply that VOC compounds are released with the addition of thermal drying at low temperatures. However, no clear pattern was detected between the two samples. The Set IV recipe did not represent sludge drying well but did indicate a possible source of earthy/musty and caramel odor within the sludge drying process.

Odor Type	Raw Sludge	30 min off-gas	60 min off-gas	90 min off-gas	120 min off-gas	150 min off-gas	180 min off-gas	210 min off-gas	coondensate	dried sludge
woody	2	2.6	2	2	3	2	1.5	1		0.5
burnt		0.6	0.5	0.5		0.5	1	1	0.5	
caramel		1.2	1	0.5	1	1	1	1		4
earthy/musty	0.5	1.03	2	1.5	1	1	1	1		

Table 3.2. Odor Profile for Sludge Drying Process of Set IV sludge recipe. *Note: number indicates average odor intensity.

Table 3.3 shows that even though the fecal odor present as a major odor in real sludge, it was not observed in the mixture recipe for Set V (9 grams of whey protein, 9 grams of lard and 9 grams of brown sugar with 33 grams of natural soil). Woody odor in the Set V original mixture recipe appears more frequently in different off-gas samples. This was not observed in sludge A or B. A burnt odor mainly appears after 90 minutes of the drying process for Set V and in the real sludge samples A and B. However, in the sludge sample, the dried sludge has a higher intensity of burnt odor, whereas in mixture recipe, the dried sludge did not show a strong burnt odor. Sweet odor was presented in the Set V mixture recipe and in the drying process for sludge A and sludge B. However, no clear pattern for sweet odor was indicated. This mixture did not represent sludge drying well but did indicate another possible source of the burnt odor.

Odor Type	30 min off-gas	60 min off-gas	90 min off-gas	120 min off-gas	150 min off-gas	180 min off-gas	210 min off-gas	dried sludge
fecal	3.5	2						
earthy/musty	0.5	1						1.5
sweet	1.5	1	2	2	2	2.5	2	1.5
burnt	1	1	5	3	2.5	4.5	3.8	1
woody	1		1.5	3.5	1.5	2	2.5	1

Table 3.3. Odor Profile for Sludge Drying Process of Set V Sludge Recipe *Note: number indicates average odor intensity.

Table 3.4 shows that the fecal odor was present as a major odor in the two real sludge samples and were observed in the mixture recipe for Set VI (protein, carbohydrate, lipid, sludge, and soil mixture with real sludge added and digested anaerobically for 14 days). However, in real sludge, the dried sludge has a higher fecal odor than the mixture recipe. This is because the fecal odorous chemicals are present in the wastewater already as shown by Zhou et al., (2016). The sludge anerobic digestion process does not apparently produce much from the raw materials of protein, carbohydrates and fats that was added in the Set VI mixture. Some of the fecal odor observed may have come from proteins (Zhou et al, 2016). Xue et al. (2022) showed that H2S and ammonia were the main odorants when drying sludge at low temperatures. They concluded that the sulfur-containing gases released during sludge drying were mainly hydrogen sulfide, sulfur dioxide, and methyl mercaptan, accompanied by a small amount of carbon disulfide, dimethyl disulfide, and carbonyl sulfur. Ammonia and organic amines accounted for more than 50% of the released nitrogen compounds. This indicates that protein was hydrolyzed to release nitrogen containing compounds.

A rancid odor was present in the mixture recipe from the fat added to the recipe, but not in the real sludge drying process as that is probably completely digested in the real sludge samples. The rancid odor observed was probably from the decomposition of fats to release Volatile Fatty Acids (VFAs) which are associated with the rancid odor. This is observed in Xue et al., (2022) when they found that the VFAs emission rates markedly increased at the beginning of the drying process. After that, their emission rates gradually decreased with the loss of moisture content. The sweet odor was both high in dried samples for the mixture recipe and occurs in the real sludge A and B. Sweet odors are also associated with VOCs.

Odor Type	raw sludge	30 min off-gas	60 min off-gas	90 min off-gas	120 min off-gas	150 min off-gas	180 min off-gas	210 min off-gas	coondensate	dried sludge
rancid	4.5	4	3.5	4	4.5	5	4.5	4.5	3.5	3.5
fecal	1	2.5	2	1	0.5	1	1.2	1.5	0.5	
sweet	2.5	1	2.5	5	2.5	2.5	2.5	2.5	2	3
rotten veg				1	1					
burnt			1	0.5	0.5	1			3	1

Table 3.4. Odor Profile for Sludge Drying Process of Set VI Sludge Recipe *Note: number indicates average odor intensity.

Figure 3.9 shows that this mixture represented an important fecal odor in sludge drying, though not highly corresponding with real sludge drying; this is an improvement in sludge-drying-odor imitation, compared to Set IV and Set V.

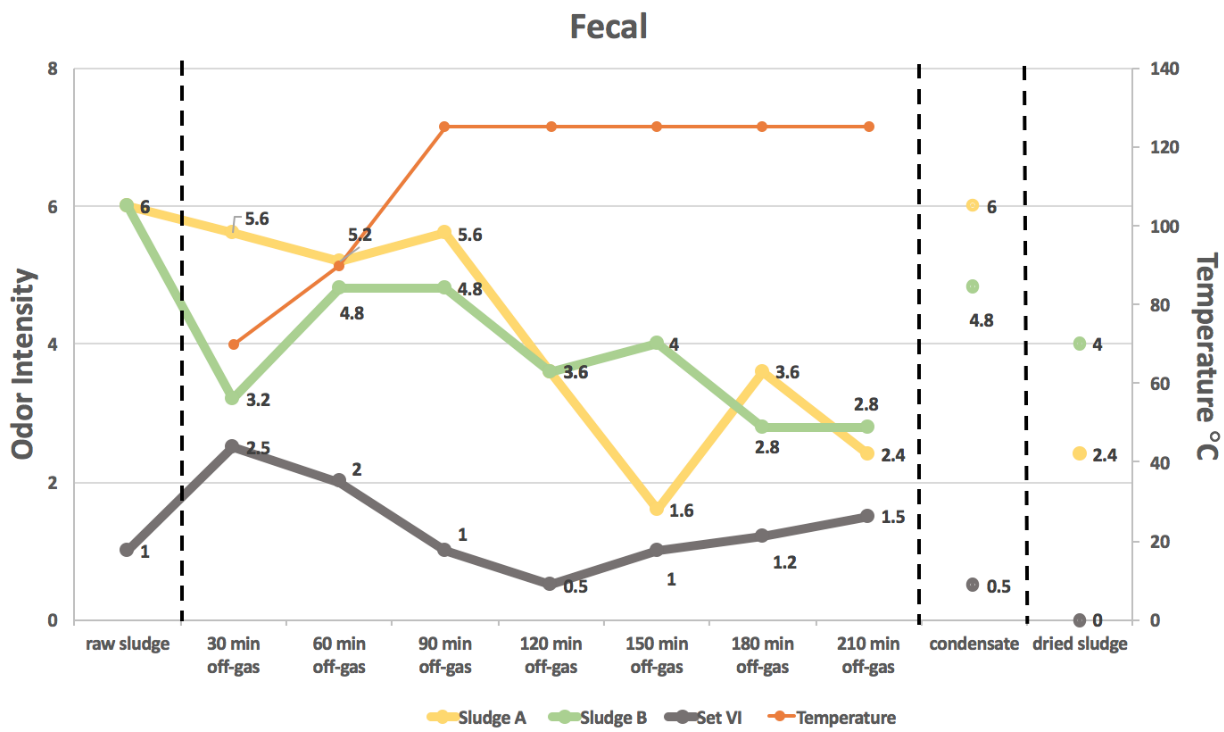


Figure 3.9. Consistency of Fecal Odor Among Sludge A, Sludge B and Set VI Recipe
*Note: The number labels are average intensity values for the off gas at corresponding time.
The red line represents the temperature change during the sludge drying process.

In Set VI, primary odors were consistent. However, other primary odors were found, and different secondary odors were identified. Thus, the pilot procedure would be valuable to complete to determine the particular primary and secondary odors present that would need odor nuisance control at a particular sludge drying facility.

3.3.8 Possible Evolution of Sludge Composition during Sludge Drying

At temperatures ranging within 165–190 ºC, sludge undergoes thermal hydrolysis. The protein in sludge decomposes into organic acids, ammonia, and carbon dioxide. Additionally, carbohydrates were hydrolyzed into small molecules of polysaccharides and monosaccharides at low temperatures. Starch and cellulose were transformed into glucose (Xue et al., 2022). Finally, these polysaccharides and monosaccharides were decomposed into volatile organic acids, resulting in the same end products as proteins. Sulfur in sludge mainly exists in the form of organic sulfur or sulfur oxides, such as thioamino acid, sulfonic acid, and sulfate. Organic sulfur readily decomposes when heated, forming odorous sulfur compounds in drying tail gas and condensate while majority of organic matter is transformed into gases and condensates (Xue et al., 2022).

In their study, Xue et al (2022) showed that potential migration of sludge composition occurs during drying. Thermal drying dissolves TOC, protein, and polysaccharide resulting in the release of VOC. Fatty acids, amino acids, and other organic compounds were generated during protein decomposition and then gets transformed into organic acid, amines, and CO2. Carbohydrates degraded into volatile organic acids. They showed that sulfur compounds decomposed into hydrogen sulfide, thioether, and other sulfur volatiles, which are possibly associated with odor pollution. The by-products were released into the air, while the condensates remained in the dried sludge. The carbon-containing substances degraded into carbon dioxide, VOCs, and carbonates.

3.4 Conclusions

The reuse of sludge largely depends on the odor associated with the dried sludge cakes. Sludge cakes with better odors such as the caramel odor observed in this study improves the social acceptance of sludge as a reuse resource. Through this study and the previous study by Decottiginies et al (2010), it is believed that the OPM along with the sludge drying wheel, Figure 1, is an applicable method to define odor nuisance from the sludge drying process. The OPM method could be used to evaluate changes of sludge composition at a particular sludge drying plant.

All major odors that appeared in Decottignies et al. (2010) study in France are also presented in the experiments performed from Southern California sludge. However, there appears to be variability of odors when different sources of sludge are used. The overall data for real sludge shows that results vary because none of the raw sludge samples can be 100% identical.

The burnt odor as an important new odor group from the sludge drying process was verified in all actual sludge samples. The burnt odor family may come from tannin and lignin material from plants and organic matter. The caramel odor was a combination of burnt odor and sweet odor may result from the degradation of carbohydrate and plant material.

Raw sludge was added into the imitation sludge recipe Number VI and successfully stimulated the anaerobic digestion processes for fats, protein, and carbohydrates that were added. Therefore, the odors produced are much closer to the real sludge drying process than other recipes. Sweet odor mainly came from carbohydrates, and the rancid odor is likely generated from lipid degradation. Lignin and tannin do not contribute to malodors associated with sludge drying process. However, they are highly likely to contribute to the caramel, sweet and woody odors. Odorant type and mass transport for each odor for Sludge A and B was not completely consistent. For future study more repeated runs using the same sludge source need to be completed to develop further scientific validity.

Reference

APHA (American Public Health Association), 2012. Standard methods for the examination of water and wastewater: Method 2170, 22nd ed. APHA, the American Water Works Association (AWWA), and the Water Environment Federation (WEF), Washington, DC, USA

Burlingame, G. A. 1999. Odor profiling of environmental odors. Water Sci. Technol. 40(6), 31-38.

Burlingame, G. A. 2009. A practical framework using odor survey data to prioritize nuisance odors. Water Sci.Technol. 59(3), 95-602.

Curren, J., Snyder, C.L.C., Abraham, S., Suffet, I.H., 2014. Comparison of two standard odor intensity evaluation methods for odor problems in air or water. Water Sci. Technol. 69, 142-146.

Decottignies, V., A. Bruchet, M. Link, M. Aupetitgendre, and I.H. Suffet. 2010. Dried Sludge Odours: Classification and Case Study. Proceedings of the Water Environment Federation. Odors and Air Pollutants 2010, pp. 72-82.

Deng W., Yan J., Li X., Wang F., Zhu X., Lu Z., Cen K. (2009) Emission characteristics of volatile compounds during sludges drying process. Journal of Hazardous Materials, Volume 162, Issue 1, Pages 186-192, ISSN 0304-3894. https://doi.org/10.1016/j.jhazmat.2008.05.022.

Ding, W., L. Li, and J. Liu. 2015. Investigation of the Effects of Temperature and Sludge Characteristics on Odors and VOC Emissions during the Drying Process of Sewage Sludge. Water Science & Technology. 72(4), 543-552.

Fechner, G., 1859. Elemente de Psychophysik, Leipzig University, Germany.

Gao W., Yang X., Zhu X., Zhao X., Yu J., Wang D., Yang M. (2022). The variation of odor characteristics of wastewater sludge treated by advanced anaerobic digestion (AAD) and the contribution pattern of key odorants. Science of The Total Environment, Volume 840, 2022, 156722, ISSN 0048-9697. https://doi.org/10.1016/j.scitotenv.2022.156722.

Greenman, J., El-Maaytah, M., Duffield, J., Spencer, P., Rosenberg, M., Corry, D., Saad, S., Lenton, P., Majerus, G., Nachnani, S., 2005. Assessing the relationship between concentrations of malodor compounds and scores from judges. JADA. 136, 749-757.

Gherghel A., Teodosiu C., De Gisi S. (2019) *A review on wastewater sludge valorisation and its challenges in the context of circular economy.* Journal of Cleaner Production, Volume 228, Pages 244-263, ISSN 0959-6526. https://doi.org/10.1016/j.jclepro.2019.04.240.

Gostelow P., Parsons S.A., Stuetz R.M. (2001) Odor measurements for sewage treatment works Water Res., 35 (2001), pp. 570-597

Guo S., Liu T., Che D., Liu H., Sun B. (2021) Effects of carbohydrates on NOx precursor formation from proteins during sewage sludge pyrolysis. Environmental Technology & Innovation, Volume 23, 101594, ISSN 2352-1864, https://doi.org/10.1016/j.eti.2021.101594.

Hayes, J. E., R. J. Stevenson, and R. M. Stuetz. 2014. The impact of malodour o Odors and Air Pollutants 2010, pp. 72-82(11) n communities: A review of assessment techniques. Science of the Total Environment. 500-501: 395-407

Mallevialle J. and Suffet I.H., 1987. Identification and Treatment of Tastes and Odors in Drinking Water, ISBN 0-89867-392-5. AWWA Research Foundation and Lyonnaise Des Eaux Cooperative Research Report, American Water Works Assoc., Denver, Colo., USA.

Núñez D., Oulego P., Collado S., Riera F. A., Díaz M. (2022) Separation and purification techniques for the recovery of added-value biocompounds from waste activated sludge. A Review, Resources, Conservation and Recycling, Volume 182, 106327, ISSN 0921-3449. https://doi.org/10.1016/j.resconrec.2022.106327.

Raveendravarrier B., Palatel A., Chandrasekharan M. (2020) Physico-chemical Characterization of sewage sludge for thermochemical conversion processes, Energy Sources, Part A: Recovery, Utilization, and Environmental Effects, DOI: 10.1080/15567036.2020.1841858

Reimann D.O. (1989) Thermal conditioning of sludge. Klarschlammentsorgung, VDI-Bildungswerk, Dusseldorf. pp. 12–26 (in German)

Suffet, I.H., Mallevialle, J., Kawczynski, E., 1995. Advances in taste-and-odor treatment and control. American Water Works Association.

Suffet, I. H. and P. Rosenfeld. 2007. The Anatomy of Odour Wheels for Odours of Drinking Water, Wastewater, Compost

Xue S., Ding W., Li L., Ma J., Chai F., Liu J. (2022) Emission, dispersion, and potential risk of volatile organic and odorous compounds in the exhaust gas from two sludge thermal drying processes. Waste Management, Volume 138, Pages 116-124, ISSN 0956-053X. https://doi.org/10.1016/j.wasman.2021.11.040.

Yan, Y., Qin, L., Gao, J. *et al.* Protein extraction and sludge dewatering performance of ultrasound-assisted enzymatic hydrolysis of excess sludge. *Environ Sci Pollut Res* **27**, 18317–18328 (2020). https://doi.org/10.1007/s11356-020-08208-2

Zhang, X. Cai, L. Qi, C. Shao, Y. Lin, J. Zhang, Y. Zhang, P. Shen, Y. Wei (2015) Effects of aeration strategy on the evolution of dissolved organic matter (DOM) and microbial community structure during sludge bio-drying. Appl. Microbiol. Biotechnol., 99 (2015), pp. 7321-7331

Zhu X., Yang X., Gao W., Jiao R., Zhao S., Yu J., Wang D. (2022) Effect of low-temperature thermal drying on malodorous volatile organic compounds (MVOCs) emission of wastewater sludge: The relationship with microbial communities. Environmental Pollution, Volume 306, 119423, ISSN 0269-7491. https://doi.org/10.1016/j.envpol.2022.119423.

Chapter 4

A Comparison of Wastewater Sludge Odor Production During Drying from 20^oC – 130^oC Vs Thermal Hydrolysis Directly at 130^oC

4.0 Abstract

Achieving a green and sustainable world requires better sludge management practices and high resource recovery from sludge produced during wastewater treatment globally. The odor from the sludge treatment process can make the sludge less desirable for reuse purposes. This paper examines the odors from two different sludge treatment processes: Thermal Hydrolysis and Sludge Drying. The odor produced from both processes were analyzed using the Odor Profile Method (OPM) and the sludge drying odor wheel. The musty odor was the primary odor in thermal hydrolysis while the fecal odor was the primary odor in sludge drying. Results revealed that thermal hydrolysis is less of an odor nuisance than the sludge drying process.

4.1 Introduction

Wastewater sludge is the residue from treating wastewater in wastewater treatment plants and has been traditionally used as a fertilizer in agriculture or disposed of in landfills. Besides its valuable agronomic properties (e.g., supply of phosphorus and nitrogen), wastewater sludge can contain heavy metals, microorganisms, and a range of hazardous organic substances, which poses a threat to soil, vegetation, animals, and humans. For this reason, there has been increasing restriction applying wastewater sludge to agricultural land (Escala et al., 2012). Achieving a green and sustainable world requires better sludge management practices and high resource recovery from sludge produced during wastewater treatment globally (Mudhoo & Sharma, 2011).

Generally, sludge treatment cost accounts for about 50% of the whole wastewater treatment plant (Chang et al. 2013). Today almost all plants built from 1938 to the 1970's have been closed or upgraded due to technical and odor problems or because of economic reasons (Keep et al., 2000).

Reducing cost and energy associated with sludge treatment is imperative and depends on various technologies including sludge dewatering, drying and thermal hydrolysis. In assessing these technologies, understanding factors that enhance the value of sludges, such as the content of organic matter, nitrogen, phosphorus, as well as the removal of undesirable heavy metals, pathogenic microorganisms, and odors during the process are valuable for economical sludge treatment. (Adamiec., 2006).

Dewatering constitutes an important step to improve waste to useful end products from wastewater sludge. Dewatering and drying reduces the volume of sludge and consequently decreases the cost of handling, transporting, and storing the final product. Additionally, it increases the calorific value of the wastewater sludge permitting its use also as a fuel or a co-fuel in cement kilns, coal-fired power plants, municipal waste incinerators, and mono-incinerators (Bennamoun et al., 2013). The two dominant technologies in wastewater sludge dewatering are belt filter presses and centrifuges. Dewatering increases the dry solids (DS) of sludge from 3-5% to 15-30% (Metcalf and Eddy, 2002). However, these processes have been shown to produce odor nuisances (Vega et al., 2015).

Most sludge drying technologies are energy-intensive and produce unpleasant odors during the drying process (Chang et al., 2013). Direct drying of sludge occurs with direct contact of the sludge with the heat source, which could be hot air or superheated steam, and acts as the conveying medium. Direct drying has the advantage of drying all types of sludge, including primary, secondary, and mixed digested sludge. Unfortunately, the current direct heat drying process for organic sludge has unpleasant odors, contamination of pathogenic microorganisms, expensive treatment costs, and high energy consumption (Chang et al., 2013). Figure 1 shows the occurrence of dewatering and drying in a general sludge treatment process.

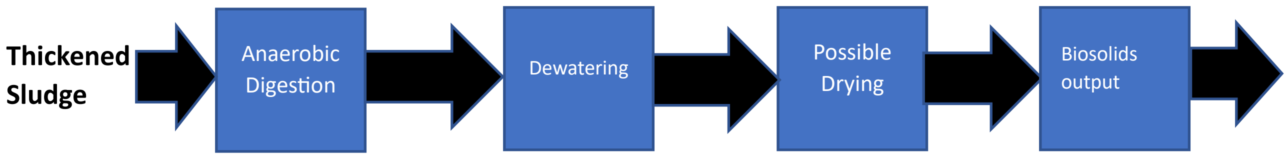


Figure 4.1. Schematic diagram of the Sludge Treatment Process

Due to the high energy demands of thermal drying, many experts consider thermal hydrolysis a successful strategy for sludge treatment (Han et al., 2021). However, primarily in Europe and Asia, studies show that thermal hydrolysis at 120°C to 200ºC, most often with pressures up to 7 bars for one hour could significantly improve the dewaterability of sludge by destroying microbial cells and releasing bound water (Feng et al., 2014). Unfortunately, the pattern of odor production during the Thermal Hydrolysis Process (THP) has not been extensively studied. Also, the relationship between odor production and high temperatures used by THP including sludge content wasn't considered.

The Thermal Hydrolysis Process (THP) involves applying heat to thickened or dewatered sludge produced during wastewater treatment at temperatures between 120°C and 200ºC, for a defined time. (Barber, 2020). Initially, the thermal hydrolysis process was installed before anaerobic digestion but, more recently, it is being used downstream of the anaerobic digestion process as seen in Figure 2 (Barber, 2020). When thermal hydrolysis is used, the dewatered cake produced can achieve 60% dry solids (DS) (Barber, 2016) and therefore needs no further drying and can serve as a fertilizer after treatment.

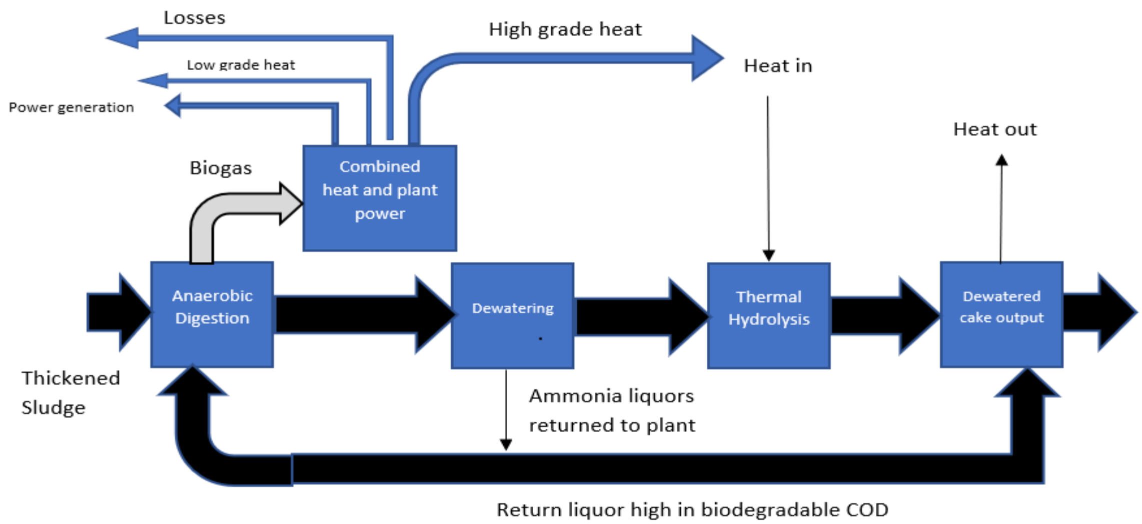


Figure 4.2. Schematic Diagram of the Thermal hydrolysis Process after Dewatering (Barber, 2020)

A significant limitation of THP is the production of hazardous and odorous chemicals, such as NH_3 and volatile sulfur compounds. In addition to causing unpleasant odors, these compounds are harmful to human health when concentrations in the working environment exceed health thresholds (Han et al., 2021). For example, permissible concentration-time weighted average (PC-TWA) of hydrogen sulfide (H_2S) and methyl mercaptan are 1 and 0.5 ppm, respectively. When these concentrations exceed their PC-TWAs, on-site employees may experience upper respiratory tract irritation, central nervous system impairment, or even liver damage (Han et al., 2021).

The heat for thermal hydrolysis is typically supplied by live steam injection at design temperature and concomitant pressure which is then rapidly released (exploded), although some configurations use standard heat exchange (Barber, 2016). Before thermal hydrolysis is applied, the sludge is thickened to about 10 and 30 % (DS) to reduce the heating demands of the thermal hydrolysis plant. The heating of sludge causes solubilization of material within it, destroys materials such as extracellular polymers in activated sludge, and sterilizes the sludge from microorganisms, making the sludge compressible, which makes dewatering of the sludge easier (Barber, 2020). Freeing cell water under hydrolysis changes the viscosity of sludge. Therefore, sludge with about 12% (DS) can be handled in the same way as raw sludge with 5–6% DS. This process allows for a higher sludge concentration in the digester feed, higher buffer capacity, and a stable digestion process (Keep et al., 2000). After processing, if the sludge goes to an anaerobic digester, it is removed from the plant at approximately 100ºC and slightly lower dry solids due to dilution with the added stream. It is then further diluted using clean water or treated effluent to control the loading rate to the downstream anaerobic digestion plant, which reduces the sludge temperature by approximately 20 ºC (Barber, 2020).

The ability of thermal hydrolysis to produce high-end biosolids has been observed in an operation plant built in Norway, 1995 to minimize the amount of sludge and maximize biogas production. Today, the plant produces digested and disinfected sludge that is attractive to farmers and is used 100% in agriculture and land reclamation (Keep et al., 2000). Drivers for installation are geographically market driven but typically include increased loading rates (to minimize size of new digestion plants or maximize use of existing facilities); improved sludge cake dewaterability which reduces downstream transport and processing costs; increased production of renewable energy, and sterilization of sludge (Barber, 2016). Table 1 lists summary of global capacity for thermal hydrolysis in order of installed capacity from suppliers.

Table 4.1: Full Scale Suppliers Worldwide of the THP Technology

Name of Manufacturers	Number	% of Total	Installed Capacity	% Installed
Cambi	65	53	6947	77
Shanghai, Pulande	6	5	432	5
Sichuan, Deepblue	11	9	324	4

Tianjin, Yuchuan	4	3	220	2
Biothelys	7	6	206	2
Beijing, Jiankunweihua	5	4	185	2
Beijing, GeeGreen	3	2	180	2
Hube,i Guoxin Tianhui	4	3	146	2
Exelys	6	5	126	1
Shenzhen, Est	3	2	70	1
Turbotech	2	2	55	1
Beijing, Hinergy	1	1	40	0
Lysotherm	2	2	36	0
Haarslev	3	2	32	0
Average Total % of Total	122		8999	

Studies elucidate the advantageous promise of the thermal hydrolysis technology for plants with high cake disposal costs, as cake disposal savings are becoming an increasingly important economic advantage in novel wastewater treatment plants (Svennevik et al., 2019). In addition, significant improvements in dewatering could affect the design and footprint of new plants using thermal hydrolysis. The area needed for storage of dewatered cake can be reduced, fewer trucks will be needed for cake disposal, and alternative cake disposal options could emerge due to the higher dryness of the dewatered cake. However, to implement these factors in the planning of new plants, more information on the expected dry solids concentration of dewatered cake after thermal hydrolysis is needed (Svennevik et al., 2019). Also, the odorous or harmful odor produced during THP remains a significant area not well understood (Han et al., 2021) Thermal hydrolysis treatments such as the Post-Anaerobic Digestion thermal hydrolysis process (Post-AD THP) have been developed to improve sludge dewatering, but the mode of action is not well understood (Svennevik et al., 2019). Thus, to effectively develop strategies to eliminate odors, it is necessary to identify the essential odors and chemical emissions during the thermal hydrolysis process. Deodorization remains a major objectives of sludge treatment and plays an important role as an index to measure the stabilization extent of wastewater sludge. Accordingly, identification of the odor emission characteristics of sludge is important in the evaluation of sludge quality and disposal options (Gao et al., 2022). The measuring of odor includes sensory analysis and instrumental analysis. The sensory analysis is carried out using the Odor Profile Method which is helpful in identifying the odor concentration (OC) or odor intensity (OI) and odor characters subjectively reflecting the degree of odor impact (Gao et al., 2022). High quality of biosolids is a result of higher degradability after THP and more stabilization of its final product which has lower odor levels and is pathogen free reduced the risk of biosolids management which further adds value to the final product (Sahu et al., 2022).

There is little knowledge on the assessment of odors produced during the THP which makes this study unique. The objective of this study is to compare odors produced by THP at 130C using sludge taken before and after centrifugation, identify different malodors produced during sludge drying from room temperature to 130C, to understand the pattern of odor changes and to compare each end-product for reuse in terms of odor.

4.2 Methods

4.2.1 Sludge Sample Collection

Sludge samples were collected from the Orange County Sanitation District on June 7, 2021. Two sets of samples were collected and labeled sample 1 and A while the second set of samples were sample 2 and B. Sample 1 and A were collected before dewatering by centrifugation and sample 2 and B were collected after centrifugation.

Sample 1 and A had 2.14% dry solids content while Sample 2 and B had 40% dry solids. The samples were stored in the refrigerator at a temperature of 4C until the day of experiment.

4.2.1 Experimental Setup

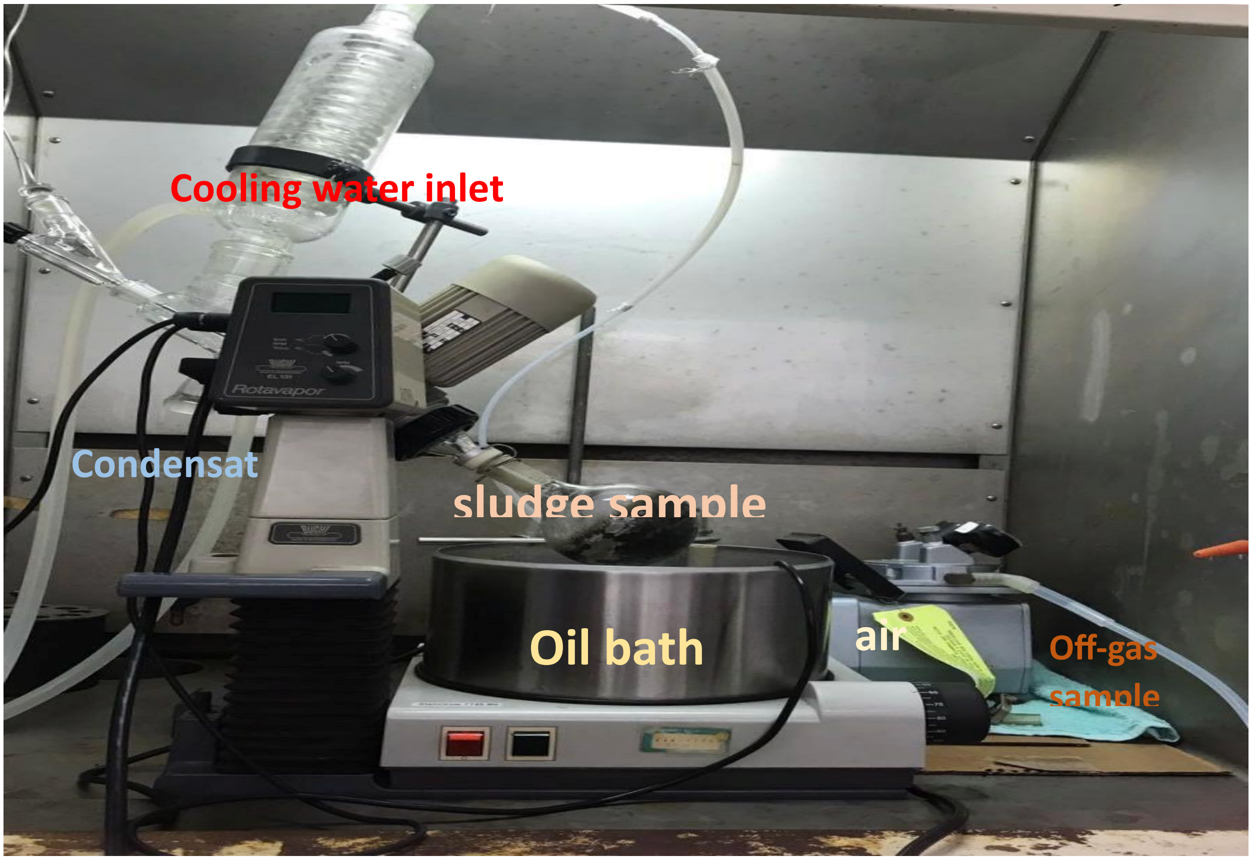


Figure 4.3. Rotary-evaporator system

The laboratory experiment method was constructed following the design of Decottignies et al. (2010) that was designed to be similar to an industrial sludge drying process. Figure 3 shows the rotary-evaporator system that consists of a rotating flask containing the sludge in a temperature adjustable oil bath. The highest temperature reached was 130°C, which is the temperature that falls within 120°C – 200°C required for a thermal hydrolysis facility.

Specific Procedure for Thermal Hydrolysis and Sludging drying:

1. Weighed out 60 g of each sample.
2. Calculated dry solid percentage.
3. Set up equipment, turn on water for condensation, turn on rotary evaporator with the rotating speed as 66 r/min and turn on heater for the oil bath and turn on air pump.
4. Set drying temperature at 130°C for thermal hydrolysis and gradual increase temperature from 22°C to 130°C for sludge drying.
5. Collected 7 three-litter gas sample with Teflon bags after 30 mins interval at 30 mins, 60 mins, 90 mins, 120 mins, 150 mins, 180 mins, and 210 mins.
6. Conducted an odor panel immediately after the samples were collected.

The Odor Profile Method (OPM) was used to categorize typical odors and the intensities of these odors. The odor wheel provided a useful tool to help describe these odorants as well as to help identify the chemical odorants that define those odors characteristics as shown in figure 3 (Burlingame, 2004 and 2009). To describe the intensity of the odor, panelists used table 2 to determine the intensity of each odor perceived.

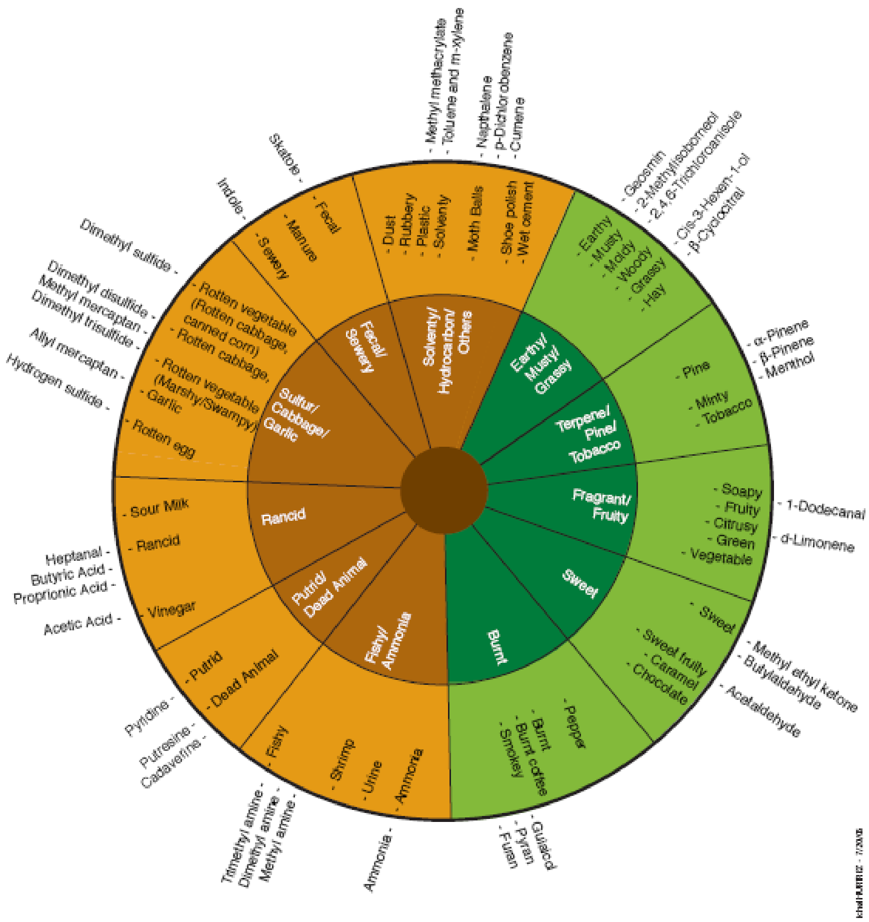


Figure 4.4. Sludge drying odor wheel.

Table 4.2: Flavor Profile Analysis Odor Intensity Strength Scale

Intensity Ratings	Designation	Intensity Description
Below 1	No odor	No odor
1	Threshold	Odor detected, but cannot describe the odor ie Odor Threshold Concentration (OTC)
2	Very weak	Odor barely perceptible
4	Weak	Odor recognized, and can be described (ORC) Odor Recognition Concentration
6	Moderate	Odor readily perceived and identified
8	Medium	Odor is uncomfortable to smell for extended periods
10	Strong	Odor is very uncomfortable to smell for extended periods
12	Very Strong	Odor is unbearable to smell even for short periods

In relating odor concentration and odor intensity, the Weber-Fechner law is the most used law to describe the relationship as; $I = k \, Log(C/C_o)$ (Huang & Guo, 2018). One of the many advantages of the Weber-Fechner equation is that it can be used to predict odor concentration since it states that the relationship of psychological perceived intensity and physical feature like odor concentration could be derived as a linear function. Therefore, odor intensity can potentially be used to predict odor concentration (Pawnuk et al., 2023).

The intensity (I) is proportional to the logarithm (base 10) of the concentration (C) relative to a previous concentration (C_o). To further demonstrate this relationship, the Weber-Fechner curve relates the OTC in air to the odor intensity of a chemical concentration in air. The sludge drying odor wheel is a useful tool for the odor panel to describe odor characters and odor intensity scale for each odorant. The OPM scale is based upon the Flavor Profile Analysis in Standard Method

#2170, (APHA, 2012) that is used as sensory panel methods in drinking water odor control studies (Suffet et al., 1987)

Figure 5 shows the Weber–Fechner Curve. For each odor character, the OPM method develops an odor intensity that is related to log concentration of the odorous chemical. The odor intensity scale has 7 points from 1 to 12; 1 (threshold), 2, 4 (recognition), 6, 8, 10, and 12. At the odor threshold intensity of 1, an odor panelist can state that the odor is different than clean air. Only at an odor intensity of 4 can the panelist recognize odor characteristics on the odor wheel. The OTC is formally described as the point where 50% of a population (minimum of 4 panelist) can define a difference between pure air and air containing an odorant. The odor recognition threshold is where 50% of an odor panel can define the type of odor from the odor wheel.

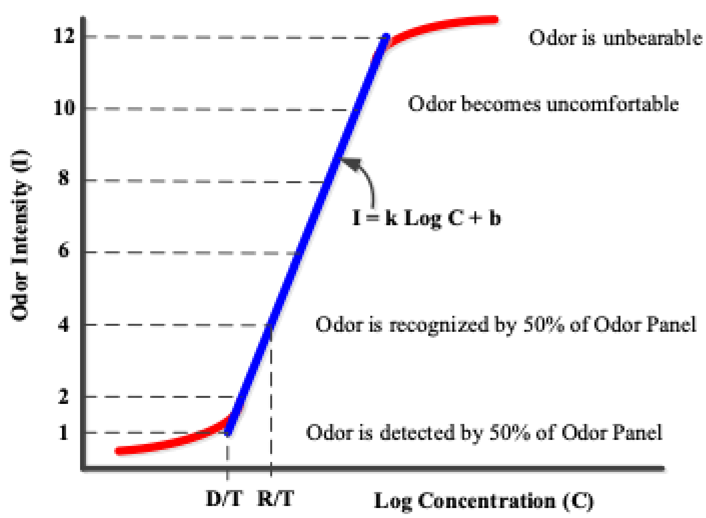


Figure 4.5. Weber-Fechner Curve

4.2.2 Thermal Hydrolysis and Sludge Drying

Sludge used for thermal hydrolysis were labeled Sample 1 and 2. Sample 1 represents the sludge collected before centrifugation while Sample 2 was the sludge after centrifugation. A 210-minute simulation of thermal hydrolysis at a temperature of 130°C without pressure was done for Sludge Sample 1 on July 20, 2021. Since, the formation of condensation in the bag can affect the odor panel results, the condensate was separated from the air before collecting the air. The aggregate for the intensity of each odor detected by the panel was recorded and analyzed. The same procedure was repeated for sludge sample 2. The results from each experiment were compared.

Sludge sample for sludging drying process were labeled sample A and B. Sample A represents the sludge collected before centrifugation while Sample B was the sludge after centrifugation. The sludge drying process was like the thermal hydrolysis method except for the gradual increase of temperature. A 210-minute simulation of the sludge drying process at rising temperature from 22°C to 130C°C was done for Sludge Sample A which was completed on August 10, 2021. Since, the formation of condensation in the bag can affect the odor panel results, the condensate was separated from the air before collecting the air. After the panels recorded the observed odor characteristics and intensity, the results were recorded and analyzed. The same procedure was repeated for sludge sample B. The results from each expertimet were compared.

4.3 Results and Discussion

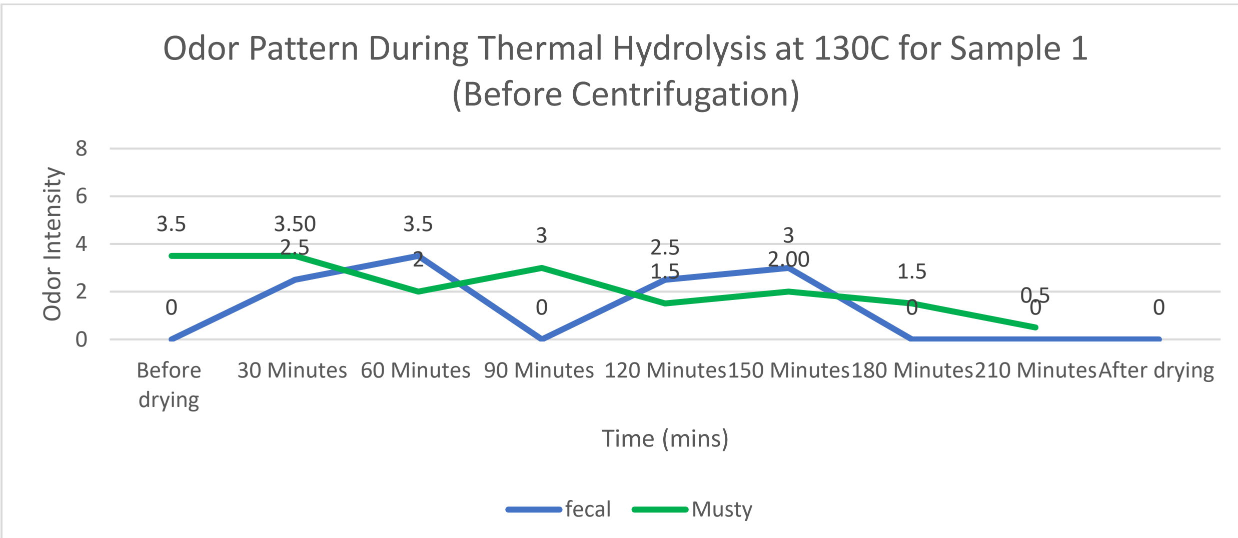
4.3.1 Thermal Hydrolysis

Table 3 shows the primary odor for sludge sample 1 was the musty odor. The odor was detected by atleast half of the odor panel and was present throughout the thermal hydrolysis process. The musty odor was detected in the off gases collected at time before drying, 30 mins, 90 mins, 180 mins and 210 mins. The musty odor tended to decrease with time being most intense before drying. Another primary odor was the fecal odor which was detected at time 30, 60, 120, and 150 mins but disapeared after 150 mins leaving the musty odor as a final residue off gas odor. For the sludge after drying, the rootten vegetable odor was detected. An odor note of rotten vegetable persisted as background odor throughout the thermal hydrolysis process. Other odor notes detected from less than half the panelist include, burnt and manure odor notes at the beginning of the process and burnt at the end of the process. Table 3 shows the odors detected by the OPM.

Table 4.3: Summary of Odor Production From Off Gasses During the Thermal Hydrolysis

Sample Type	Sample 1 – Gases produced from sludge before centrifugation	
Date Analyzed	7/20/2021	
Panel Method	Odor Profile Method (OPM)	
Odor Panel	4 People	
Sample ID	**Date Analyzed**	**Odor Characteristics and Intensities**
Before drying	7/20/2021	**Musty**, 3.5 ±3.6 Odor Note: Fecal, Manure
30 Minutes	7/20/2021	**Musty** 3.50 ±0.87; **Fecal** 2.5 ±2.6
60 Minutes	7/21/2021	**Musty** 2.0±2.0; **Fecal** 3.5 ±3.6 Odor Note: Rotten vegetable
90 Minutes	7/22/2021	**Musty** 3.0±2.2 Odor Note: Fecal, Rotten vegetable
120 Minutes	7/23/2021	**Musty** 1.5 ±1.7; **Fecal** 2.5 ±2.6
150 Minutes	7/24/2021	**Musty** 2.0±1.4; **Fecal** 3 .0±3.0
180 Minutes	7/25/2021	**Musty** 3.0±1.0 Odor Note: Fecal, Rotten vegetable

210 Minutes	7/26/2021	**Musty** 0.5 ±0.9 Odor Note: Fecal, Burnt and rotten vegetable
After drying	7/27/2021	**Rotten Vegetable** 3.5 ±2.1 Odor Note: Burnt, Sweet, Fecal, Musty, sour, fruity



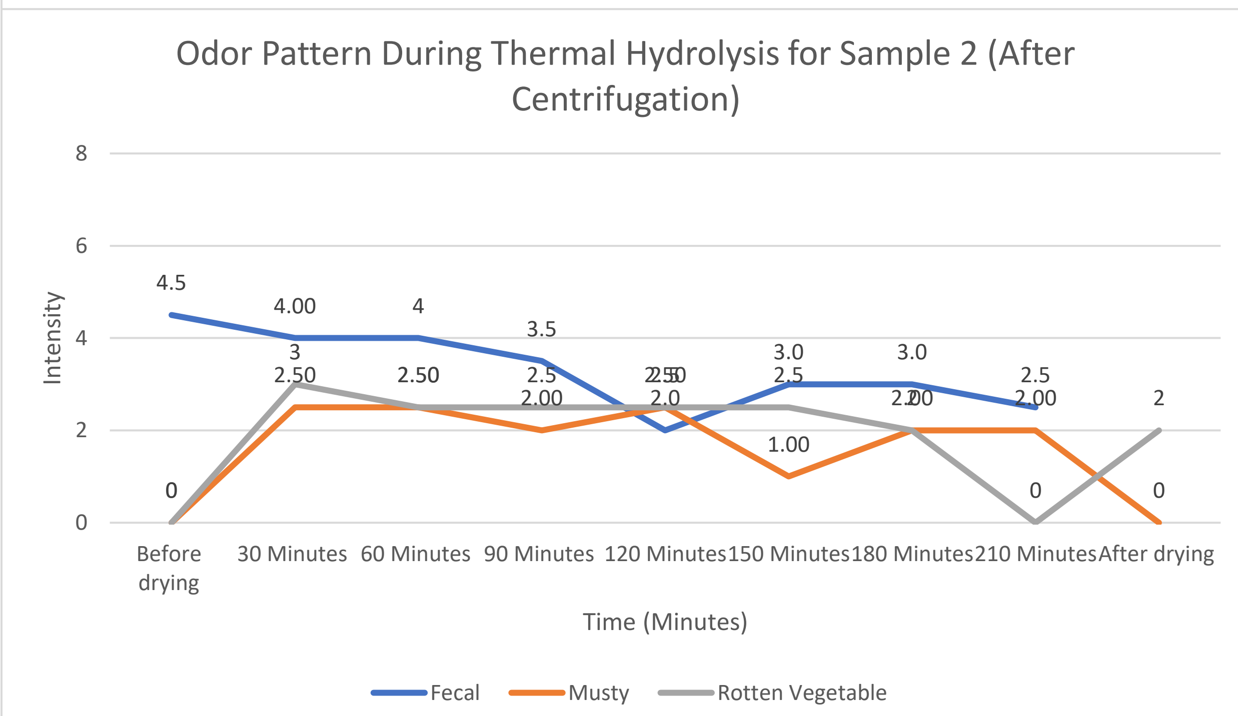


Figure 4.6. Primary odors produced during the thermal hydrolysis process for sludge before and after centrifugation.

Sludge Sample 2: Fecal odor was the primary odor for the sludge after centrifugation. The fecal odor was detected before, during and throughout drying times. The fecal odor was only absent in the dried sludge. Rotten vegetable and musty odor were also a prominent odor that was detected in the off gases from 30 mins – 210 mins. Figure 6 and 7 show the pattern of odor emission during the thermal hydrolysis process. Fecal odor was intense throughout the drying process with intensity range 4-6. Musty odor had constant low intensities of 2-4. Rotten vegetable odor was significant throughout and gradual decreased with time.

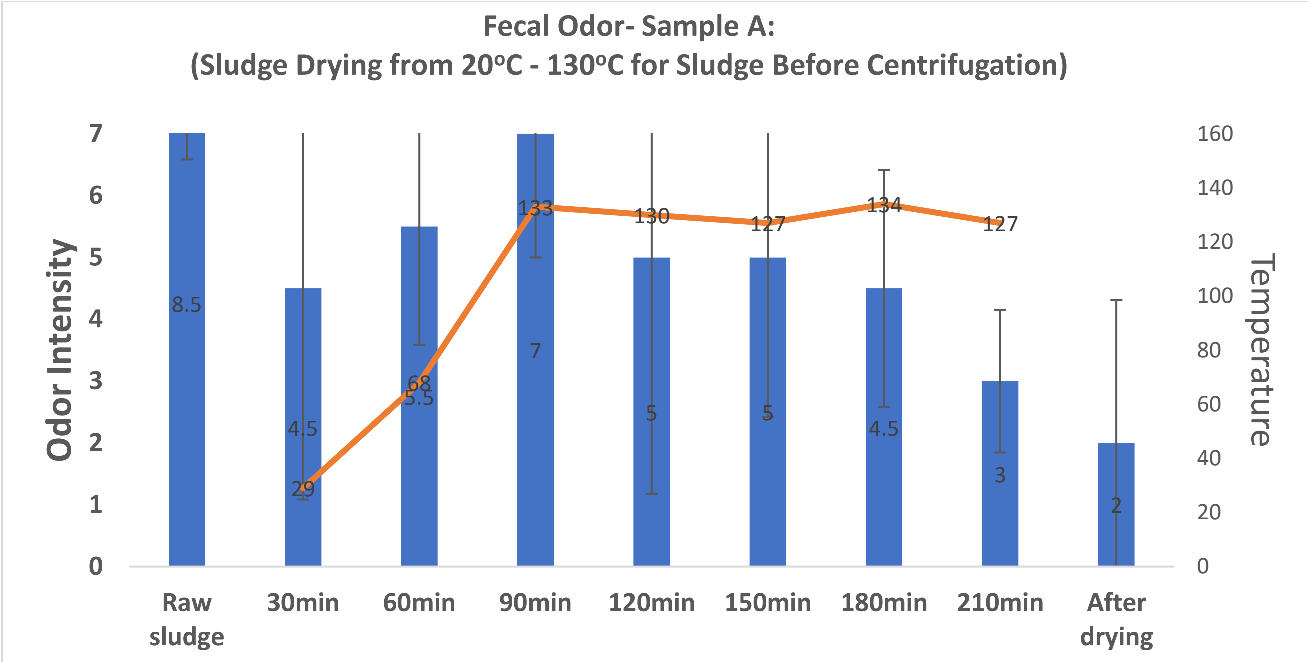
The dried sludge in both sample 1 and 2 retained the rotten vegetable odor after drying which can be explained by the volatilization of chemicals emitting the musty and fecal odor thereby unmasking the rotten vegetable odor. However, comparing both sludge 1 and 2, thermal hydrolysis before dewatering by centrifugation is less of an odor nuisance since the musty odor is less intense than the fecal odors.

4.3.2 Sludge Drying

For both sludge A and B, fecal odor was the prominent odor, detected before drying and throughout the drying process and in the dried sludge. The fecal odor started at a high intensity of 8 for the sludge A sample before drying and reduced to an intensity of 2 after the drying process. Musty odor was also detected at 30, 60 and 120 mins of collected off- gases. Other odor notes include fishy, sweet, burnt, garlic, ammonia, chlorinous and rancid. Table 4 shows a comprehensive analysis of odors detected while figure 4.8 shows the pattern of the fecal odor through the sludge drying process.

Table 4.4: Summary of Odor Production from Off-gases for Sludge Drying Process

Sample Type	Sludge A (Before centrifugation)	
Date Analyzed	8/10/21	
Panel Method	Odor Profile Method	
Analysis Temp.	Room Temperature	
Odor Panel	Total odor panel of 4	
Sample ID	**Date Analyzed**	**Odor Characteristics and Intensities**
Before drying	8/10/21	**Fecal** 8.5±1.9 Odor Note; ammonia, musty, chlorinous
30 minutes	8/10/21	**Fecal** 4.5±3.4, musty 2.5±1.9 Odor Note; sulfide
60 minutes	8/10/21	**Fecal** 5.5±1.9, musty 3.0±3.8 Odor Note; garlic, fishy
90 minutes	8/10/21	**Fecal** 7.0±2.0 Odor Note; musty, garlic, fishy
120 minutes	8/10/21	**Fecal** 5.0±3.8, musty 2.5±3.0 Odor Note; fishy, rancid, garlic
150 minutes	8/10/21	**Fecal** 5.0±2.6 Odor Note; fishy, musty, garlic
180 minutes	8/10/21	**Fecal** 4.5±1.9 odor note; sweet, burnt, garlic, musty
210 minutes	8/10/21	**Fecal** 3.0±1.2, garlic 2.5±3.0 Odor Note; burnt, musty
After drying	8/10/21	**Fecal** 2.0±2.3, rotten veg 5.5±3.8 Odor Note; sweet, musty



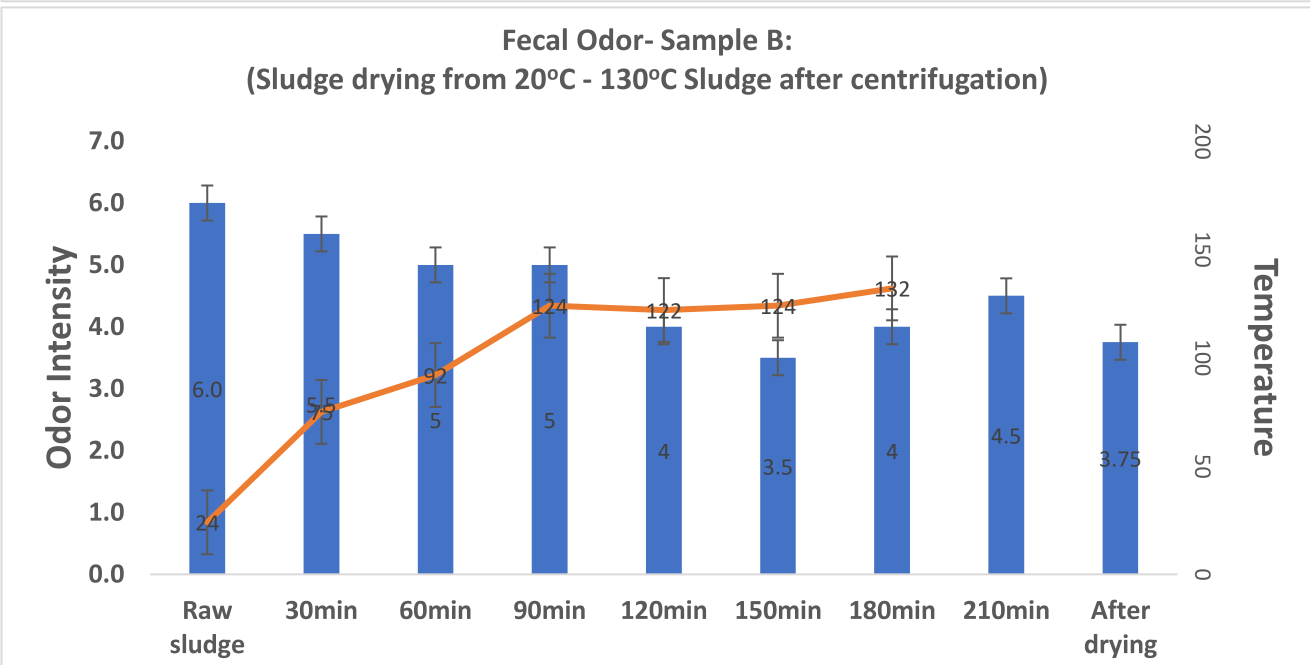


Figure 4.8. Primary odor produced during sludge drying for sludge before and after centrifugation.

Sludge Sample B followed the same odor emission pattern as sludge sample A. The fecal odor was the most prominent odor present before drying and even in the dried sludge. The highest intensity was at 8 in the sludge before drying. Musty odor was also detected before drying and at 30 mins, 60 mins, and 120 mins. Other odor notes include earthy which is similar to musty smell, ammonia, sweet, pine rotten egg and rotten vegetables.

Table 4.5: Summary of Odor Production from Off-gases for Sludge Drying Process

Sample Type	Sludge B (After centrifugation)	
Date Analyzed	8/13/21	
Panel Method	Odor Profile Method	
Analysis Temp	Room Temperature	
Odor Panel	Total odor panel of 4	
Sample ID	**Date Analyzed**	**Odor Characteristics and Intensities**
Before drying	8/13/21	**Fecal** 8.0±3.5, musty 2.0±2.3, ammonia 3.0±3.5 Odor Note; pine
30 minutes	8/13/21	**Fecal** 7.3±2.3, musty 4.5±3.4 Odor Note; ammonia, sweet
60 minutes	8/13/21	**Fecal** 5.0±3.5, musty 3.0±2.0 Odor Note; sulfur, sweet
90 minutes	8/13/21	**Fecal** 5.0±4.2 Odor Note; sweet, rotten egg, musty
120 minutes	8/13/21	**Fecal** 4.0±4.9, musty 2.5±3.0 Odor Note: ammonia, burnt, sweet
150 minutes	8/13/21	**Fecal** 3.5±4.7 Odor Note; ammonia, burnt, sweet, musty, pine
180 minutes	8/13/21	**Fecal** 4.0±4.9 Odor Note; rancid, ammonia, rotten veg, sweet, musty
210 minutes	8/13/21	**Fecal** 4.5±3.4 Odor Note: sweet, musty, burnt

After drying	8/13/21	**Fecal** 3.75±4.3, earthy 3.75±2.9 Odor Note; sweet, rotten veg

The odors emitted in the sludge drying process were similar to the odors observed by Bian et al 2018. Fecal odor was a primary odor emitted in the off gases of the laboratory 210 mins simulation. Although in the Bian et al study, the fecal odor disappeared after drying, for this study the dried sludge retained the fecal odor. For both sludge drying samples, A and B, the fecal odor was observed throughout the drying times and in the dried sludge. In a similar fashion, Gao et al (2022) conducted a study using sludge samples from five wastewater plants with different sludge treatment processes including the use of sludge drying and thermal hydrolysis. To obtain a quantitative and distinguishable characterization of odor, a modified flavor profile analysis was used to provide the odor intensity of the sample and the category of odor as well (Gao et al., 2022). The odor wheel was also used to identify possible chemicals causing these odors. Raw sludge possessed fecal/sewery and sulfide/cabbage/garlic odor characters, while sludge cakes were characteristic of earthy/musty/moldy and grassy/woody odor. The result from their study were closely related to the odors observed in our study. Other odor notes were fishy, rancid, garlic, sulfide and burnt. In an odor point of view, The result from our study suggests that sludge drying before centrifugation is less of an odor nuisance than sludge after centrifugation.

4.3.3 Comparing Thermal Hydrolysis and Sludge Drying Odor Production

Although the fecal odor was a primary odor for both treatment processes, the fecal odor in the thermal hydrolysis decreased over time while in the sludge drying process the odor was observed throughout the drying process and was present after drying. Additionally, the musty odor which was the other primary odor in the thermal hydrolysis process is less of an odor nuisance when compared to the fecal odor. Therefore, from an odor production point of view, thermal hydrolysis before and after centrifugation is less of an odor nuisance than sludge drying process. The emission of volatile organic compounds (VOCs) from dewatered anaerobic digested sludge included different categories of malodorous substances such as volatile organic sulfur compounds, volatile organic aromatic compounds, aldehydes, ketones, and alcohols which were associated with microbial degradation of proteins. Therefore, different odor characters were reported for dewatered anaerobically digested sludge, such as putrid, sewer, earthy, and garbage odor, and the range of odor concentration or odor intensity varied largely (Gao et al., 2022).

4.3.4 **Chemicals Causing Malodors.**

Malodors are a result of unmasking volatile chemicals through the application of heat. Each odor unmasked during the drying process has been linked to a chemical causing the malodor according to the sludge drying wheel. For this study, the chemicals possible causing the odors are presented in Table 7. A GC-MS procedure is needed to further confirm the present of these chemicals in the off-gases.

Table 4.7: Major Odors and Likely Chemicals Suggested by The Odor Wheel that Needs Further Study

Odors Observed	Chemicals to Study
Fecal	Indole & Skatole
Rotten Vegetable	Methyl Mercaptan
Rotten Vegetable	Dimethyl Trisulfide
Rotten Vegetable	Dimethyl Sulfide
Musty	Methyl Isoborneol

4.4 Conclusion

Thermal hydrolysis is becoming increasingly popular as a viable option to treat sludge more efficiently and economically. The thermal hydrolysis process was compared to the sludge drying process for odor production. The pattern of prominent odors was:

Thermal Hydrolysis- sample 1 –before centrifugation: Musty was the primary odor, then Fecal and Rotten Vegetable respectively.

Thermal Hydrolysis – sample 2- after dewatering by centrifugation: the primary odors were Fecal, Rotten Vegetable, Musty respectively.

From an odor production point of view, thermal hydrolysis before dewatering is less of an odor nuisance. For the sludge drying process before and after centrifugation: Fecal odor was the only prominent odor and decreased with time, but the dried sludge retained the fecal odor. Thermal hydrolysis before and after centrifugation also had fecal odor as one of the major odors but it decreased with time and the dried sludge had no fecal odor indicating from an odor production point of view, thermal hydrolysis before and after dewatering is less of an odor nuisance than sludge drying. In both thermal hydrolysis samples, all other odors were eliminated except rotten vegetable which was present in dried sludge sample 2. Thermal hydrolysis for sludge before centrifugation produces less odorous biosolids. Future studies will investigate the chemicals associated with the odors observed at a temperature higher than 130 and with applied pressure.

Reference

APHA (American Public Health Association), (2012). Standard methods for the examination of water and wastewater: Method 2170, 22nd ed. APHA, the American Water Works Association (AWWA), and the Water Environment Federation (WEF), Washington, DC, USA

Barber W.P.F. (2016). Thermal hydrolysis for sewage treatment: A critical review. Water Research, Volume 104, Pages 53-71, ISSN 0043-1354. https://doi.org/10.1016/j.watres.2016.07.069.

Barber W. (2020). Sludge Thermal Hydrolysis: Application and Potential. IWA Publishing. ISBN: 9781789060270

Bian, Yuge & Liu, Fuqiang & Decottignies, Virginie & Bruchet, Auguste & Suffet, I.. (2018). Characteristics of Nuisance Odor Emissions from the Sludge Drying Process. Proceedings of the Water Environment Federation. 2018. 547-562. 10.2175/193864718824828713.

Bart Peeters (2010) Mechanical Dewatering and Thermal Drying of Sludge in a Single Apparatus, Drying Technology, 28:4, 454-459, DOI: 10.1080/07373930903155614

Bennamoun L., Arlabosse P, Léonard A. (2013). Review on Fundamental Aspect of Application of Drying Process to Wastewater Sludge, Renewable and Sustainable Energy Reviews, Volume 28,Pages 29-43, ISSN 1364-0321, https://doi.org/10.1016/j.rser.2013.07.043.

Burlingame, G.A., Odor profiling of environmental odors. Water Sci. Technol. 1999; 40(6), 31-38.

Chen G., Yue P. L., Mujumdar A. S. (2002) Sludge Dewatering and Drying, Drying Technology, 20:4-5, 883-916, DOI: 10.1081/DRT-120003768

Chang F., Ko C., Wu J., Wang H. P., Chen W. (2013) Resource recovery of organic sludge as refuse derived fuel by fry-drying process. https://doi.org/10.1016/j.biortech.2013.03.112.

Curren, J., Snyder, C.L.C., Abraham, S., Suffet, I.H., 2014. Comparison of two standard odor intensity evaluation methods for odor problems in air or water. Water Sci. Technol. 69, 142-146.

Dai Z., Su M., Ma X., Wang G., Li D., Liu C., Weng H. (2018) Direct thermal drying of sludge using flue gas and its environmental benefits, Drying Technology, 36:8, 1006-1016, DOI: 10.1080/07373937.2017.1368541

Decottignies V., Bruchet, A., Suffet, M. (2010). Dried Sludge Odours: Classification and Case Study. Proceedings of the Water Environment Federation. 2010. 72-82. 10.2175/193864710802768299.

Escala M., Zumbühl T., Koller C., Junge R., Krebs R. (2013). Hydrothermal Carbonization as an Energy-Efficient Alternative to Established Drying Technologies for Sewage Sludge: A Feasibility Study on a Laboratory Scale. *Energy & Fuels* 2013 *27* (1), 454-460 https://pubs.acs.org/doi/pdf/10.1021/ef3015266

Euroby Limited. (2021). Centridry – Enhanced Sludge Dewatering Process. Centridry - Sludge Drying - Enhanced Sludge Dewatering ... (environmental-expert.com)

Fechner, G., 1859. Elemente de Psychophysik, Leipzig University, Germany.

Feng G., Tan W., Zhong N., Liu L. (2014). Effects of thermal treatment on physical and expression dewatering characteristics of municipal sludge. Chemical Engineering Journal, Volume 247, Pages 223-230, ISSN 1385-8947. https://doi.org/10.1016/j.cej.2014.03.005.

Gao W., Yang X., Zhu X., Zhao S., Yu J., Wang D., Yang M. (2022). The variation of odor characteristics of wastewater sludge treated by advanced anaerobic digestion (AAD) and the contribution pattern of key odorants, Science of The Total Environment, Volume 840, 156722, ISSN 0048-9697, https://doi.org/10.1016/j.scitotenv.2022.156722. (https://www.sciencedirect.com/science/article/pii/S0048969722038190)

Greenman, J., El-Maaytah, M., Duffield, J., Spencer, P., Rosenberg, M., Corry, D., Saad, S., Lenton, P., Majerus, G., Nachnani, S., (2005). Assessing the relationship between concentrations of malodor compounds and scores from judges. JADA. 136, 749-757.

Gross T. S. C. (1993). Thermal Drying of Sewage Sludge. **https://doi.org/10.1111/j.1747-6593.1993.tb00843.x**

Han Z., Li R., Shen H., Qi F., Liu B., Shen X., Zhang L., Wang X., Sun D. (2021). Emission characteristics and assessment of odors from sludge anaerobic digestion with thermal hydrolysis pretreatment in a wastewater treatment plant. Environmental Pollution, Volume 274, ISSN 0269-7491. https://doi.org/10.1016/j.envpol.2021.116516.

Janusz Adamiec (2002) Drying of Waste Sludges in A Fluidized Bed Dryer With A Mixer, Drying Technology, 20:4-5, 839-853, DOI: 10.1081/DRT-120003766

Keep U., Machenbach I., Weisz N., Solheim O. E. (2000). Enhanced stabilization of sewage sludge through thermal hydrolysis - three years of experience with full scale plant. *Water Sci Technol* 1, 42 (9): 89–96. doi: https://doi.org/10.2166/wst.2000.0178

Liu X., Lee D. (2015). Some Recent Research and Development in Drying Technologies: Product Perspective, Drying Technology, 33:11, 13391349, DOI: 10.1080/07373937.2015.1026986

Mamais D., Tzimas A., Efthimiadou A., Kissandrakis J., Andreadakis A. (2009). Evaluation of Different Sludge Mechanical Dewatering Technologies. https://www.destechpub.com/wp-content/uploads/2019/01/JRST-Volume-6-Number-1-January-2009.pdf?x82929#page=30

Mudhoo A., Sharma S. K. (2011). Microwave Irradiation Technology In Waste Sludge And Wastewater Treatment Research, Critical Reviews in Environmental Science and Technology, 41:11, 999-1066, DOI: 10.1080/10643380903392767

Niessen W. R. (2012). Thermal Processing of Biosolids. https://pdhonline.com/courses/c449/c449content.pdf

Sahu A. K., Mitra I., Kleiven H., Holte H. R., Svensson K.(2022). Chapter 24 - Cambi Thermal Hydrolysis Process (CambiTHP) for sewage sludge treatment, Clean Energy and Resource Recovery, Elsevier, Pages 405-422, ISBN 9780323901789. https://doi.org/10.1016/B978-0-323-90178-9.00020-2. https://www.sciencedirect.com/science/article/pii/B9780323901789000202)

Svennevik O. K., Solheim O. E., Beck G., Sørland G. H., Jonassen K. R., Rus E., Westereng B., Horn S. J., Higgins M. J., Nilsen P. J. (2019). Effects of post anaerobic digestion thermal hydrolysis on dewaterability and moisture distribution in digestates. *Water Sci Technol* 1; 80 (7): 1338–1346. doi: https://doi.org/10.2166/wst.2019.379

Suffet, I.H. (Mel), S. Braithwaite, Y. Zhou. Bruchet, A. (1987) "The Drinking Water Taste-and-Odor Wheel After 30 Years, Chapter 2. In "Taste and Odour in Source and Drinking Water: Causes, Controls, and Consequences" Editors: T-F. Lin, S. Watson, and I.H. (Mel) Suffet, Publisher, International Water Association.

Suffet, I.H., Mallevialle, J., Kawczynski, E., 1995. Advances in taste-and-odor treatment and control.

American Water Works Association.

Tolga Tunçal (2010). Evaluating Drying Potential of Different Sludge Types: Effect of Sludge Organic Content and Commonly Used Chemical Additives, Drying Technology, 28:12, 1344-1349, DOI: 10.1080/07373937.2010.482704

Wakeman R. I. (2007) Separation technologies for sludge dewatering. Journal of Hazardous Materials,Volume 144, Issue 3, Pages 614-619, ISSN 0304-3894, https://doi.org/10.1016/j.jhazmat.2007.01.084.

Vega E., Monclús H., Gonzalez-Olmos R., Martin M. J. (2015). Optimizing chemical conditioning for odor removal of undigested sewage sludge in drying processes. https://doi.org/10.1016/j.jenvman.2014.11.012.

Chapter 5
Conclusions and Future Research

5.1 Conclusion

Odor evaluation studies are necessary steps to creating an effective odor management strategy. Odor wheels have been successfully created and used in various fields, such as drinking water and composting, to identify and reduce odors. Although the development of the sludge drying odor wheel was underway when these studies began, the result from this dissertation validates the odor wheel. It helps develop a more comprehensive odor wheel that can be used in wastewater treatment facilities and sludge drying facilities to correctly identify odorants during the sludge drying process.

To validate the sludge drying odor wheel, the results from the first study demonstrated that sludge drying, both in Limay, France, and at UCLA, US, produced similar odors, and even their difference could be explained by the sludge sources. The results from the second study produced similar odors compared to the odor wheel, demonstrating that sludge composition affects the type of odorants produced since different sludge and artificial sludge produce various odorants. The results from the last study showed that the pattern of odors produced during sludge drying changes as temperatures change. Using Thermal hydrolysis, which employs high temperature to dry sludge, produced a musty odor, which is less of an odor nuisance when compared to direct drying of sludge at low temperature, which produces a fecal odor.

Although there are no policies or regulations on odor, previous methods used in odor evaluation rely only on olfactometry. This study proves that sensory analysis (OPM) and chemical analysis can effectively predict an odor's character and intensity. Therefore, this study provides a better strategy for reducing odors at a wastewater treatment plant. At a WWTP, this strategy can be applied at:

- Fence line of WWTP and sludge drying facilities.
- During the transportation of sludge from a facility.
- End products (Dried sludge) for land application.

This dissertation successfully used a combination of OPM, Weber-Fechner curves and GC-MS for odor activity values to determine odors produced during the sludge drying process. The overall goal of the dissertation which was to develop knowledge to control odors produced during sludge drying at a WWTP and the results showed that the goals and objectives were met.

5.2 Future Research

- The burnt and caramel odor produced during this study requires further investigation. Future studies can replicate these odors, and chemical analysis can be done to determine the chemicals responsible for them.
- Future studies could investigate synergistic and antagonistic reactions occurring during the drying process that could produce more desirable odors, such as caramel and earth/musty odors.
- Further chemical analysis using more sensitive instruments can be done to confirm more odors on the sludge drying odor wheel.
- Future studies should explore various temperatures to determine an optimal temperature for sludge drying that will produce fewer odors and better-dried sludge that can be socially acceptable for land application.
- Furthermore, the evolution and disappearance of certain odors based on other pretreatment before drying should be investigated to understand if a pretreatment (dewatering) could produce a better odor.