Alternative Techniques for Characterizing Plastics in Organic Waste Streams

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Executive Summary

The presence of plastics in source-separated organics (SSOs) has aroused increasing concerns in organic waste recycling facilities which generate biogas as a renewable energy source. Plastic contamination in the feedstocks for these facilities can significantly disrupt operations. Plastic contaminants also persist through the digestion process and contaminate the digestate end product that is often destined for agricultural land applications, which is an environmental concern.

A challenge with developing strategies to better manage plastics in these waste streams is the lack of an effective and efficient technique for measuring them in the complex SSO samples. With growing interest in this area of waste management, this white paper reviews alternative characterization techniques for the characterization and quantification of plastics in organic waste. Such techniques could be integrated into quality assurance and quality control protocols for the maintenance of digestate quality.

As plastic characterization in organic wastes is a relatively undeveloped area, techniques used in similar sectors such as food safety, recycling, and composting were investigated, along with the methods that are currently used for this application were reviewed. Non-destructive imaging techniques that were investigated included hyperspectral, ultrasound, terahertz, and X-ray technologies. The use of isolation methods to separate plastics from the waste were examined, and characterization techniques that rely on plastic isolation are discussed. The principles and capabilities of each technique were identified and compared.

Based on a critical analysis, it was determined that hyperspectral imaging stands out as the best candidate for this application, with X-ray-based technologies also showing some potential. Hyperspectral imaging has the ability to classify a wide range of materials based on their spectra, which is unique to each type of material and thus can be used to selectively detect plastics. X-ray imaging was the second-ranked technique due to its superior penetration capability that allows characterization of hidden objects. However, its use may be limited by its inability to selectively detect plastics that have similar density to the organic waste components. Fourier transform infrared (FTIR) spectroscopy and Raman spectroscopy were identified as leading candidates on the basis of their proven abilities in plastic characterization, if the plastics can be separated from the organic matrix.

Although hyperspectral and X-ray imaging techniques show promise for use in organic waste application, pretreatment may be be needed due to sample complexity. A sieving and rinsing method was developed in this study to decrease the complexity of the sample and expose the plastics.

The identified promising techniques will likely require enhancements before they can be implemented in organic waste applications. Enhancements in hardware and software for implementation of hyperspectral imaging, X-ray imaging, FTIR spectroscopy, and Raman spectroscopy have been identified.

Acknowledgements

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Glossary

3D	Three-Dimensional
AAM	Active Appearance Model
AD	Anaerobic Digestion
AI	Artificial Intelligence
ANN	Artificial Neural Networks
$CaCl_2$	Calcium Chloride
CH_4	Methane
DSC	Differential Scanning Calorimetry
FDA	Fisher Discrimination Analysis
$FeSO_4$	Iron(II) Sulfate
FTIR	Fourier-Transform Infrared
HDPE	High-Density Polyethylene
HIU	High-Intensity Ultrasound
HSI	Hyperspectral Imaging
H_2O_2	Hydrogen Peroxide
IR	Infrared
LDA	Linear Discrimination Analysis
LDPE	Low-Density Polyethylene
LIU	Low-Intensity Ultrasound
MCR	Multivariate Curve Resolution
MLR	Multi-Linear Regression
MRF	Markov Random Field
MS	Mass Spectroscopy
MSI	Multispectral Imaging
NaCl	Sodium Chloride
NaI	Sodium Iodide
NIR	Near-Infrared
n.d.	No Date
NMR	Nuclear Magnetic Resonance
PCA	Principle Component Analysis
PETE or PET	Polyethylene Terephthalate
PLSR	Partial Least Squares Regression
PP	Polypropylene
PS	Polystyrene
PVC	Polyvinyl Chloride
Pyr-GC-MS	Pyrolysis Gas Chromatography Mass Spectroscopy
QA/QC	Quality Assurance and Quality Control
SSO	Source-Separated Organics
S-G	Savitzky-Golay Least Squares Method
TGA	Thermal Gravimetric Analysis
THz	Terahertz
Vis	Visible
$ZnCl_2$	Zinc Chloride

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1 Introduction

Plastics are practical materials used in a wide variety of applications in daily life. In recent decades, however, waste plastics have become an environmental challenge. It is estimated that a total of 6.3 billion tonnes of plastic waste have been produced worldwide (Nature Communications, 2018). Plastics can remain in the environment for hundreds of years without decomposing. Even after some degradation, waste plastics remain in the environment in the form of small particles. Of particular significance to this project are the current practices of many food processing facilities, supermarkets, restaurants, and even households that result in packaged foods and other plastic materials being disposed of in source-separated organic (SSO) waste bins. Hence, these organic waste streams that are intended for processing are often contaminated with plastics (Nature Communications, 2018).

Organic wastes can be used to produce renewable energy by anaerobic digestion (AD). AD is a natural process in which microorganisms break down organic materials in an oxygen-free environment and produce a stream rich in methane (CH_4) called "biogas". An AD facility typically consists of the following 4 main stages:

- collection of organic waste in a "pit",
- pre-treatment of the waste,
- the digestion process,
- and generation of biogas and digestate (Figure 1).

Biogas can be employed as an alternative energy source through several pathways, while digestate can be used as a soil conditioner or fertilizer (Jain, et al., 2015).

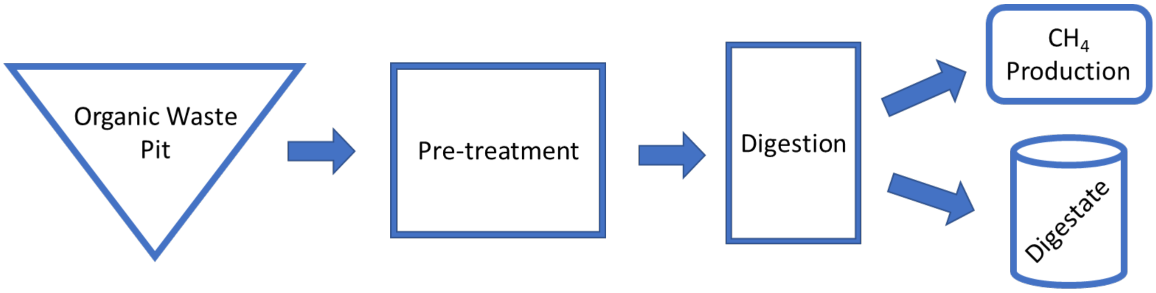

Figure 1. Schematic representation of processes employed in an AD facility

The presence of plastic materials in organic waste has been reported to negatively affect the efficiency of digestion process (Lim, et al., 2018). Plastics can clog pumps and valves and cause damage to the digestion system equipment. Moreover, these materials can pass through the digestion process without decomposing and end up in digestate that could be later used as an organic soil conditioner for agriculture. It is considered best practice to prevent this contamination that can reduce the quality of soil and crops, as well as jeopardize the health of animals in the ecosystem and humans who consume these crops (Bernstad, et al., 2013). However, a challenge with implementing practices for controlling plastics in these streams is the lack of an effective and efficient technique for quantifying plastic contamination in these complex matrices.

The current methods that are used in industry were originally designed for compost applications. The methods, discussed in detail in section 2, are based on sieving and weighing foreign particles, which have been visually detected. The method is time and labour-intensive and subject to human error. It is apparent that there is a need for characterization methodologies that are appropriate for measuring plastics in SSO streams when they are being processed (prior to AD). These streams are typically high in particulate matter content but have often gone through size reduction and slurrying processes.

The purpose of this study is to investigate modern, accurate, time- and labour-efficient techniques for the characterization and quantification of plastics in organic waste streams. Such methods, if integrated into QA/QC protocols, would allow the establishment of a more rigorous baseline of plastic contamination, against which future contamination reduction efforts can be evaluated. The study consisted of a literature review of alternative techniques that have been used in similar fields, an evaluation of their applicability to plastic characterization in organic wastes, and information gathered from commercial vendors of the promising systems.

2 Conventional Methodologies and Implementation in Quality Standards

Two conventional methods for quantifying plastics in compost have been reported with one method having been developed for hard plastics (US Composting Council, 2001b) while the other was developed for film plastics (US Composting Council, 2001a). These methods have been established for compost quality and have been found to be time-consuming and labour-intensive. Hard plastics are separated by sieving and then collected manually using tweezers, prior to being dried in an oven and then weighed (US Composting Council, 2001b). This method is used in Canada and elsewhere, not just the United States (CCME, 2005).

Plastic films are thin, lightweight materials, making it difficult to quantify them by analytical balance. Hence, in the US Composting Council method, plastic films are initially separated by sieving and then coated with an opacifier solution. Images of the films are taken by an imaging technique. The surface area of the films is then calculated using image analysis software (US Composting Council, 2001a). It is possible to quantify the mass of the films by considering density and volume, but many institutions prefer to save time by performing the dry weighing method and accepting errors associated with this approach.

The research team observed an implementation of these methods at a commercial laboratory for the characterization of plastics in samples from SSO streams that were generated at an AD facility. Based on this review, it was concluded that these methodologies would be difficult to employ on an ongoing basis at an AD facility for quality control purposes. They were found to depend heavily on operator experience and attention and human errors could result in major inaccuracies in characterization. In addition, plastic particles smaller than the human eye can detect (approximately 1-5 mm) are neglected by the current method. Hence, it was concluded that enhanced characterization techniques would be desirable.

The literature was reviewed to identify standards that employ measurements of plastics in organic wastes for quality control purposes. Examples of different institutions' specifications for the limits of plastic contamination in compost and waste food slurry are summarized in Table 1. Each institution used a dry mass %w/w (dry plastic per dry compost) method to quantify their plastics as a percentage of the slurry. It is worth noting that the only report of the use of these methods to quantify plastic materials in organic food slurries was by the Los Angeles County Sanitation District (LACSD, 2018). A majority of the specifications have been reported for composts.

As can be seen from Table 1, particles with sizes greater than 2 mm are typically considered contaminants, and maximum of 1.2% w/w for food slurry and 1.0% w/w for compost are the allowable limits for contamination. Hence, it is apparent that the presence of relatively small quantities of plastics in SSOs can result in their being deemed contaminated. Alternative characterization techniques for measuring plastics would need to be sufficiently sensitive in this regard.

Table 1. Compost and food slurry quality specifications of different institutions

	Material	Film Plastics	Film & Hard Plastics	Film & Hard Plastics, Glass and Metals	Particle Size
American Biogas Council (ABC, 2016)	Compost	<0.25%		<1.0% total	-
British Standard Institute (BSI, 2014)	Compost		<0.25%	<0.5% total*	>2mm
Los Angeles Food Waste Slurry (LACSD, 2018)	Food Waste	<0.40%		<1.2% total**	>4mm
Ontario Compost Quality (MOE, 2012)	Compost		<0.50%	<1.0% total	>3mm

*Glass sharps are not included

**Rocks are also included

Relatively few reports of studies that have quantified plastics in organic waste, digestate, or compost streams were identified (Bläsing & Amelung, 2018). A report by TRI Environmental Consulting Inc. described a study conducted in Surrey, British Columbia, from November 2012 to October 2013, where monthly samples of source-separated yard and kitchen waste contained an average of 0.3% plastic contamination (Van Drimmelen, 2013). Gajst (2016) reported average plastic concentrations of 1200 mg/kg (0.12%) in Slovenia. The quantification methods used in both of the previous studies were not specified but are assumed to have been based on weighing methods. Bläsing and Amelung (2018) used a manual method, similar to the standard compost method, to determine plastic concentrations in compost in Germany. Samples of structured compost, compost from green cuttings, and compost from bio-waste were found to contain dry weight plastic concentrations of 2.38 mg/kg (0.00024%), 65 mg/kg (0.007%), and 180 mg/kg (0.02%), respectively. The limited availability of reports on studies that have measured plastics in organics may be indicative of the challenges associated with the current characterization methodologies. With growing interest in the use of SSOs for anaerobic digestion it can be expected that there will be a greater demand for data on plastic contamination of these streams.

3 Properties of Organic Waste

As part of the identification of potential alternative techniques it was deemed important to establish the properties of the organic wastes that are being characterized. In this regard, it was considered important to identify the key types of plastic materials that are likely to be present in SSOs since some plastics may be more readily identified by some methods. Further, it was anticipated that other materials present in SSOs may act to interfere with some characterization methodologies. This section provides an overview of these components.

3.1 Plastics

The six most common types of plastic materials, as well as miscellaneous other plastics, are described in Table 2. Based upon their widespread use, any of these types of plastics could be expected to be found in organic waste samples. The physical and chemical properties of plastic materials, such as density, colour, microstructure, macrostructure, hydrophobicity, etc., should be considered to properly select techniques. In the subsequent review of potential alternative characterization techniques, their ability to distinguish these different types of plastics was considered desirable. From Table 1 it can be seen that plastics are diverse in their uses, morphologies, and physical properties. This diversity can be expected to establish limits on the types of characterization techniques that are viable for specifically detecting plastics because for the technique to be effective, it must be able to characterize and quantify all plastics, not just those in a specific density range or a specific colour or structure (Mertes, 2015) (Sun, et al., 2019).

Table 2. Common household plastic materials (Mertes, 2015)

Abbreviations	Polymer Name	Density	Common Uses
PETE or PET	Polyethylene Terephthalate	1.3 – 1.4 g/cm^3	Soda bottles, water bottles, salad dressing bottles, medicine jars, peanut butter jars, jelly jars, combs, bean bags, rope, tote bags, carpet
HDPE	High-Density Polyethylene	0.94 – 0.97 g/cm^3	Milk jugs, juice containers, grocery bags, trash bags, motor oil containers, shampoo-conditioner, detergent bottles, bleach containers, toys
PVC	Polyvinyl Chloride	1.15 – 1.70 g/cm^3	Some tote bags, plumbing pipes, grocery bags, tile, cling films, shoes, window frames, ducts, sewage pipes
LDPE	Low-Density Polyethylene	0.92 – 0.94 g/cm^3	Cling wrap, sandwich bags, squeezable bottles such as mustard, grocery bags, frozen food bags, flexible container lids
PP	Polypropylene	0.90 – 0.91 g/cm^3	Disposable cups and plates, plastic diapers, Tupperware, kitchenware, margarine tubs, yogurt containers, prescription bottles, stadium cups, bottle caps
PS	Polystyrene	1.04 – 1.05 g/cm^3	Disposable coffee cups, plastic food boxes, plastic cutlery, packing foam, packing peanuts
Other	Miscellaneous Plastics	variable	Plastic CDs and DVDs, baby bottles, large water bottle with high capacity, medical storage containers, eyeglasses

3.2 Matrix Components

The other, non-plastic components of organic waste (the “matrix”) were of interest because they may interfere with plastic characterization. These other components of the matrix include water, lipids, carbohydrates, proteins, minerals, miscellaneous organic compounds, and foreign materials. Examples of sources of these basic components are listed in Table 3 (City of Ottawa, n.d.) (CEC, 2017).

Table 3. Examples of sources of matrix components (City of Ottawa, n.d.) (CEC, 2017)

Matrix component	Examples of sources
Water	
Lipids	Fats, oils, grease, nuts, butter, shortening, lard, wax
Carbohydrates	Vegetables, wood, grains, paper, leaves, nutshells, sugars
Proteins	Meats, bones, hair, feathers, eggs, fish, dairy, pet food
Minerals	Sediment, eggshells, misc. shells, ash, litter box filler
Misc. organic compounds	Acids, bases, alcohols
Foreign materials	(Plastics), glass, metal

The wide variety of other materials in the matrix is expected to make characterization of the plastic contamination challenging. Like plastics themselves, organics and foreign materials may span a wide range of physical properties. For instance, the density of some foods may be identical to the density of some plastics. Additionally, many matrix components such as bone, stone, and glass are similar in physical appearance to hard plastics, and fibrous vegetables such as onion may appear similar to film plastics. To minimize the interfering effects of the matrix, cleaning samples to reduce complexity before attempting characterization may be necessary. A cleaning method that was evaluated as part of the current study is described in section 4.

An additional analytical challenge is that the wide variety of matrix materials could result in high levels of heterogeneity within and between samples. Such heterogeneity would impact the number and size of samples that would need to be characterized. These constraints could impact the viability of some alternative characterization methods if they cannot accommodate sufficiently large samples or require inordinately long test methods.

4 Sample Cleaning Assessment

As part of this project, techniques for pre-treating SSO samples prior to characterizing plastics in the samples were reviewed (these are expanded upon in section 5.2.1). On the basis of a critical analysis of reported techniques, a novel sample cleaning method to separate plastics and other particles from the matrix of liquid, paste, and very small particles was tested. The tests had the following three goals:

(1) to gain an understanding of the samples and thus make more accurate hypotheses about which plastic characterization techniques might be effective;
(2) to gain an understanding of the conventional plastics characterization methods and the limitations in their application to slurries; and
(3) to explore an approach that would reduce the complexity of the matrix and expose the plastics, thus potentially making quantification by the methods described in section 5 more accurate.

Samples of organic waste from three different points in the anaerobic digestion pre-treatment process were obtained from an anaerobic digestion facility for testing. Figure 2 shows the consistency of the samples. Sample A1 was taken from a point located between the organics pit and pre-treatment process; thus, it was thick and pasty with pieces of food and other particles large enough and unaltered enough to be easily visible to the naked eye. In the pre-treatment process, a magnet is employed to remove metal particles, and a plastic separator is used to remove some of the plastic. Sample B1 was taken just after the magnet and plastic separator processes. It was less viscous, but still paste-like. Liquids are introduced between the pre-treatment and digester processes, and organic waste and wastewater are mixed to improve viscosity and yield from the digestion process. Sample C1, taken after this mixing process and before the digester, was a viscous liquid with some visible chunks. Testing was conducted on these three samples to identify and quantify plastics manually and thus gain an understanding of the challenges of analyzing these types of samples.

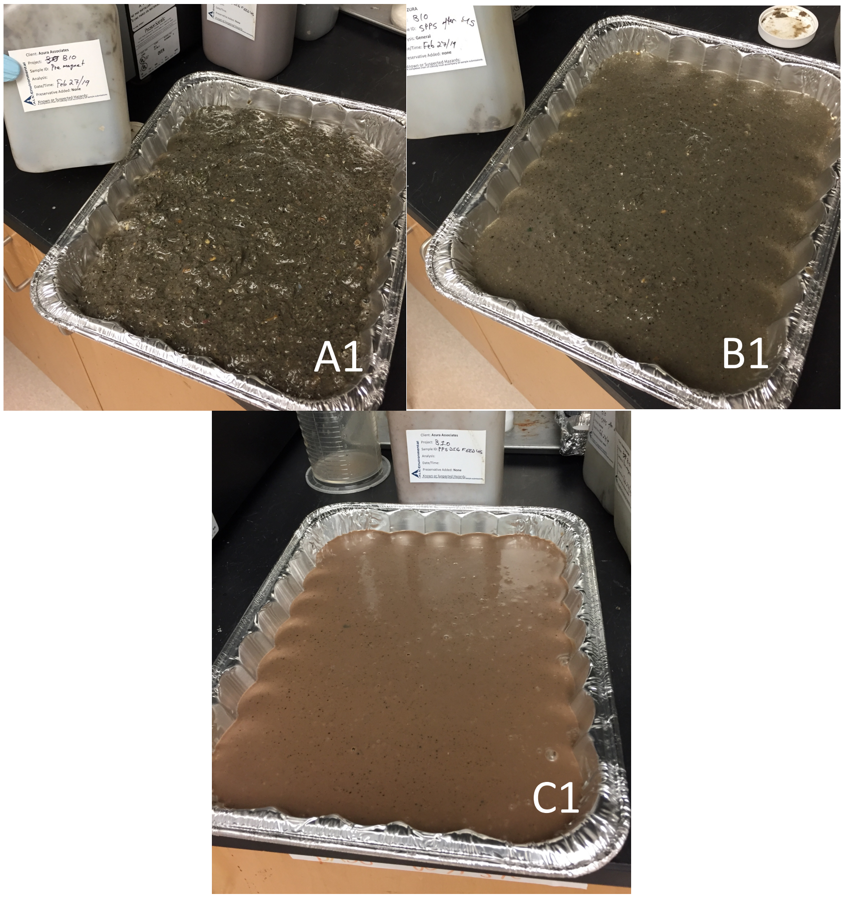

Figure 2. Trial 1. Upper left: Sample A1 – early in the pre-treatment process. Upper right: Sample B1 – intermediate. Bottom: Sample C1 – late in the pre-treatment process.

4.1 Drying Tests

As a preliminary test, samples were dried in trays to assess whether plastics could be identified and separated with no further treatment steps. Samples were dried in an oven at 50 °C over three days. After drying it was found that the samples had hardened into a "cake" with the plastics hidden within the cake. A photo of the dried Sample C1 is shown in Figure 3 to illustrate the problematic morphology of the dried cake. While plastics could be identified in the dried samples, the method was found to be time-consuming and generally ineffective.

Figure 3. Trial 1. Sample C1 dried after three days in an oven at 50 °C and manually cracked.

4.2 Sieving/Rinsing Test

The purpose of this test was to clean the plastic particles to expose them and make them more visible to a potential imaging system. Sieves were employed to remove excess water and micro-scale particles. The metal sieve system consisted of 4.0 mm, 3.35 mm, 2.36 mm, 1.0 mm, and 600 µm sieves. The sieve sizes were selected based on the sizes available in the lab; the specific values were deemed to be less important to the effectiveness of the separation process. After placing one litre of sample in the top of the sieve system, the stack of sieves was placed in a sieve shaker for 15 minutes. The sieve shaker proved mostly ineffective, as all of the samples were too viscous to pass through the sieves. Hence, a rinsing method was introduced. The same sieve system was rinsed using tap water, taking care to ensure flooding did not occur in the lower levels that clogged readily. This approach was found to work well for the most liquid sample (Sample C1) but the clogging proved to be a more serious challenge with Samples A1 and B1, which clogged the top sieve. Thus for Samples A1 and B1, the sieve system was modified to include 6.73 mm, 4.0 mm, and 2.36 mm sieves. A 600 µm sieve was added to the bottom, and its sole purpose was to avoid clogging the drain. The 600 µm sieve was cleaned out regularly and its contents were discarded. Thus, with this setup, all particles smaller than ~2.36 mm were lost, but this was deemed to be acceptable as particles smaller than 2 mm do not negatively affect compost quality.

Figure 4 shows the particles captured on the 6.73 mm and 4.0 mm sieves after rinsing Sample B1 as an example of the results obtained with the developed methodology. This sieving/rinsing method with the 6.73/4.0/2.36 mm sieve setup was determined to be effective for isolating macroplastics from the matrix. A challenge associated with the method was cleaning the sieves after each use, as the particles tended to wrap themselves around the sieve wires.

Figure 4. Trial 1. Left: Sample B1 6.73 mm sieve after rinsing. Right: Sample B1 4.0 mm sieve after rinsing.

After sieving was complete the sieves were inverted into a tray and the particles remaining on the sieves were moved to the tray using tweezers. The tray containing the particles was dried in an oven at 50 °C for 16 hours, and the plastics were then manually extracted using tweezers. The detection of film plastics manually was found to be possible, as they tend to have a large surface area and in a dried matrix, they were the only particles that were flexible and not dehydrated. However, it is more difficult to identify the hard plastics visually, as some of them appeared similar to other components such as bone, shell, seeds, or glass.

A second trial of this method was conducted two months after the first trial. The samples, taken from the same locations of the digester pre-treatment process, were found to be different in composition and color. Figure 5 shows the consistency of the new samples.

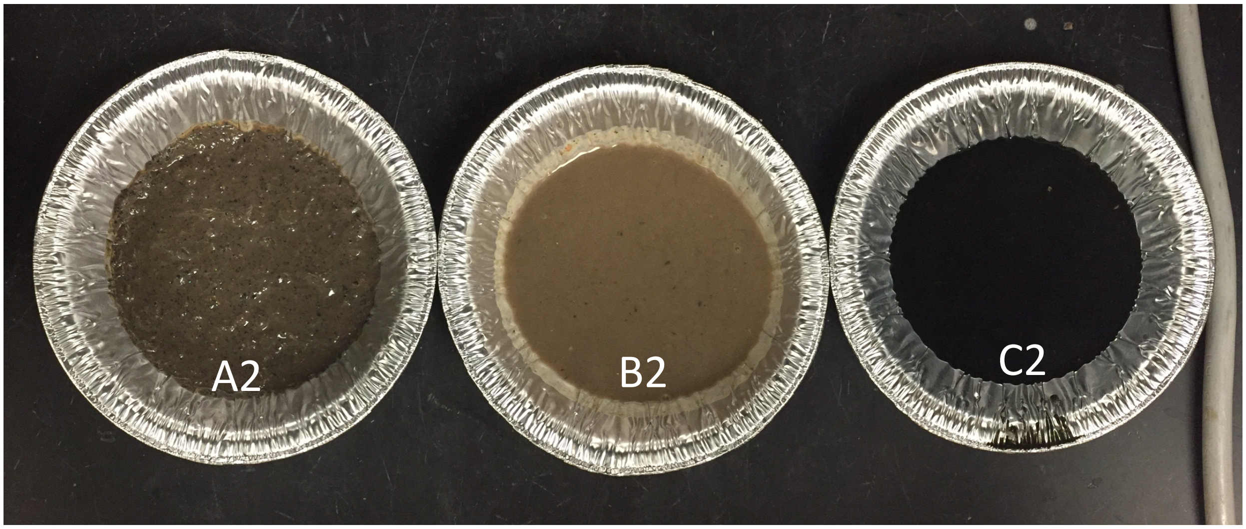

Figure 5. Trial 2. Left to right: Sample A2 (early in the pre-treatment process), Sample B2 (intermediate), and Sample C2 (late in the pre-treatment process).

A comparison between Figure 5 and Figure 2 shows that Sample A2 looked similar to Sample A1, but Sample B2 and Sample C2 were substantially different in composition, colour, and texture. Sample B2 had a lighter consistency more similar to Sample C1 than Sample B1, and Sample C2 appeared dark blue or black.

A solids content test was conducted on the second round samples. Aluminum pans (duplicates of each sample) containing approximately 50 g of each sample (shown above in Figure 5) were placed in an oven at 105 °C until an analytical balance showed no further decrease in mass. The resulting total solids masses are listed below:

- Sample A2 was found to have a solid content of 21.6%.
- Sample B2 was found to have a solid content of 4.8%.
- Sample C2 was found to have a solid content of 3.6%.

It is hypothesized that if a solid scontent test had been performed on the trial 1 samples, Samples B1 and C1 may have had a higher solid content than the trial 2 Samples B2 and C2 due to the observed differences in their consistency.

The trial 2 samples were subjected to the same sieving/rinsing method as trial 1, described in detail in Appendix A. This time, however, 1000 g of each sample was used, and the plastics collected from the sieves were weighed. Only film plastics were identified for this measurement—hard plastics were not found. The dry mass of each sample was determined by multiplying the total sample mass by the solids content for each sample. The masses of plastic in the samples are listed below:

- 0.18 g of plastic (0.8% of dry mass) was collected from Sample A2.
- 0.057 g of plastic (1.2% of dry mass) was collected from Sample B2.

Sample C2 contained plastic as well, but it could not be accurately weighed as the plastics were coated in black organic matter that could not be cleaned off.

On the whole, it was concluded that the sieving/rinsing method was successful at separating most plastics and other large particles from the paste-like matrices, making them easier to analyze further. The method worked well for film plastics, but is not as effective for hard plastics, as hard plastics are more difficult to identify visually.

It is hypothesized that when using imaging techniques, the plastics could be identified and quantified directly on the sieves, with no need to remove the particles from the sieves. However, this method could result in an underestimation of some of the plastics present because plastic films may be wrapped around the sieve wires. This outcome would need to be examined when testing imaging methodologies.

It should be noted that organic waste samples may differ drastically in composition and consistency across sampling events, and any system that is chosen to characterize plastics in organic waste would need to be able to handle this variability. Additionally, it may not always be possible to entirely clean off the plastics by rinsing, so the system would need to be able to handle some amount of obscuring organic matter on the plastics. More information describing the method is provided in Appendix A.

5 Plastic Characterization Techniques

A literature review was conducted to identify alternative plastic characterization techniques which could potentially be applied to organic waste samples. Because of the scarcity of studies on plastic material characterization in organic wastes, related industries such as food, compost, soil, and plastic were also investigated. The advantages, disadvantages, and feasibility of the techniques are discussed in this section. The techniques were divided into two main categories—direct and indirect—depending on how much sample preparation and treatment is required prior to their use.

5.1 Direct Techniques

For the purposes of this study direct techniques were defined as those that could be employed to rapidly characterize the plastic content of organic wastes without using a purification, separation, or extraction step. Such techniques may, however, require a rinsing and sieving step to expose the plastics. These techniques were divided into three subclasses: online, nearline, and offline. Online techniques were defined as those that could perform the analysis without the need to remove sample from the line. Nearline techniques were defined as those that could be conducted in the AD facility but would require samples to be removed from the line. Offline techniques were defined as those that would need to be conducted in a facility or laboratory that would be located at some distance from the AD facility; for example, samples that would need to be analyzed at a commercial laboratory.

5.1.1 Online Techniques

For the purpose of this report, online techniques have been defined as those that have the ability to analyze samples from the processing system. The analysis of materials being conveyed on a conveyor belt is an example of such an approach. The instruments could be installed at the sides or on top of the processing system and have the capability of making real-time measurements. Hyperspectral imaging was the only technology identified in this study that was deemed to have the potential to be employed as an online technique for characterizing plastics in organic wastes.

5.1.1.1 Hyperspectral Imaging

Hyperspectral imaging (HSI) has garnered attention in recent years due to its use in a variety of applications. It is currently used in areas such as agriculture, environment, industry, and medicine (Bouyé, et al., 2018). An HSI system integrates two different but similar techniques: imaging and spectroscopy. The two techniques in combination provides spatially distributed spectral data of the target material (Zheng, et al., 2018). A similar technique, multispectral imaging (MSI), is also discussed in this section. MSI also collects spatial and spectral data, but it only collects information for three to ten spectral bands, whereas HSI may use hundreds to thousands of spectral bands. Spectral resolution is defined as the wavelength bands in the electromagnetic spectrum that an instrument is capable of scanning (Frank, et al., 2015).

A schematic representation of a typical HSI setup is shown in Figure 6. In HSI, the imaging technique is used to collect two-dimensional spatial data from the material. A high-definition camera is used in combination with an illumination apparatus to take high quality pictures with resolutions as fine as 1 nm/pixel (Qin, et al., 2013). Integration of the spectrometer with the camera captures spectral data for each pixel that results from the interactions (transmission, reflection, or absorbance) between the matter and the electromagnetic radiation provided by the light source. Each material will have a characteristic spectral pattern, a “spectral fingerprint”, that can be used to identify it through comparison with spectral libraries. Spectroscopic techniques can collect spectral data from the materials up to 5 nm interval resolutions (Frank, et al., 2015).

Raman spectroscopy and Fourier-transform infrared (FTIR) spectroscopy are the most commonly used spectroscopic techniques in HSI systems. These two techniques are similar as they all gather spectra in the infrared (IR) region of the electromagnetic spectrum. Additional details of Raman and FTIR spectroscopy are provided in Section 5.2.2. In this range of wavelengths, the spectral signals can be used to give information about the vibrational modes of molecules. As plastics show unique properties in the visible (Vis) and near-infrared (NIR) region (700nm-2500nm), Vis-NIR could be considered the best region for plastic characterization.

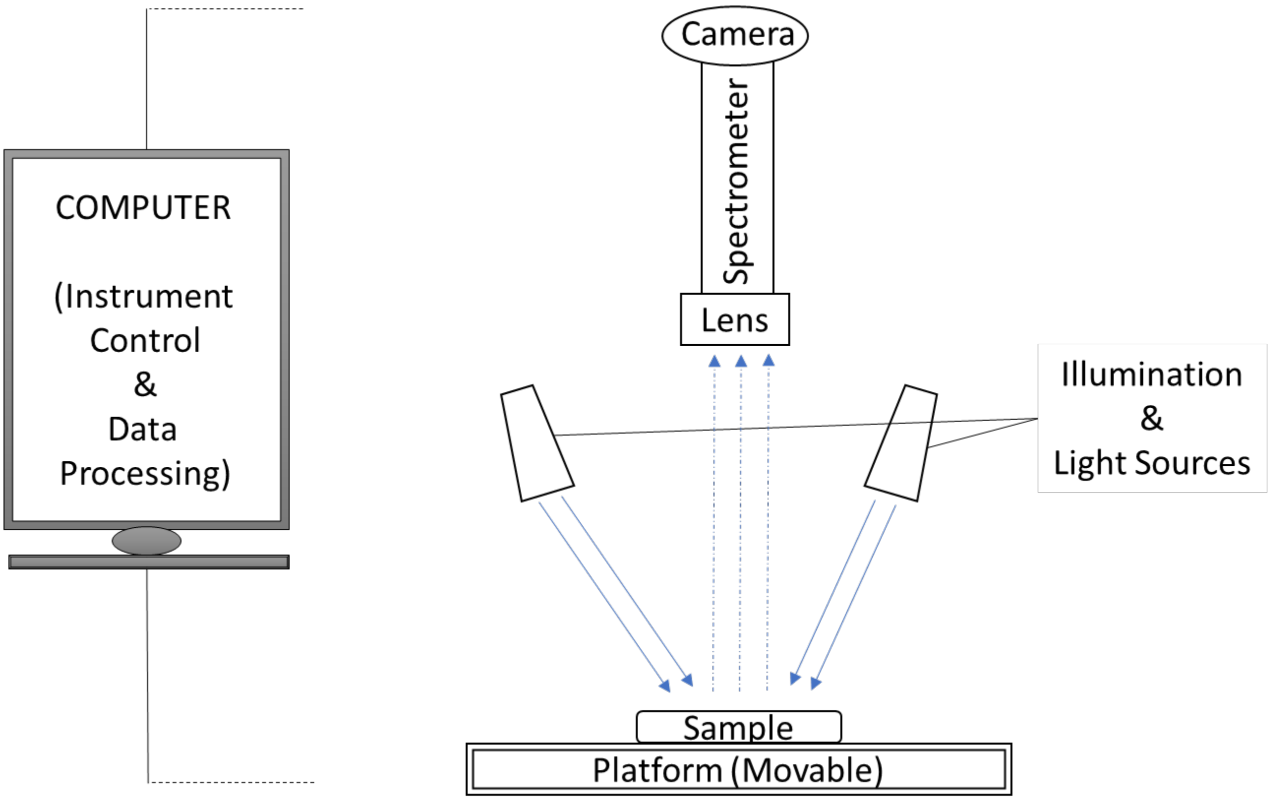

Figure 6. Schematic representation of HSI setup

Three scan options (point, line, and area) are available for HSI imaging depending on the application and the sample area (Figure 7). In the point scan method, a single point is scanned at a time. This approach has also been referred to as the whiskbroom method. It can generate high resolution, but a moving sample holder or detector is required to scan an areal sample. In the line scan method (also known as the pushbroom method), a single narrow line is scanned at one time. Similar to the point scan method, the line scan method can yield high resolution pictures. A less complex moving sample holder or detector is required for this approach. In the area scan method, the entire sample area is scanned all at once. The resolution of the pictures generated by the area scan is typically lower than the other methods, but it is the fastest scanning method and usually does not require a moving sample holder or detector (Qin, et al., 2013). Considering both the analysis rate and resolution together, the line scan method has been determined to be optimal for many applications.

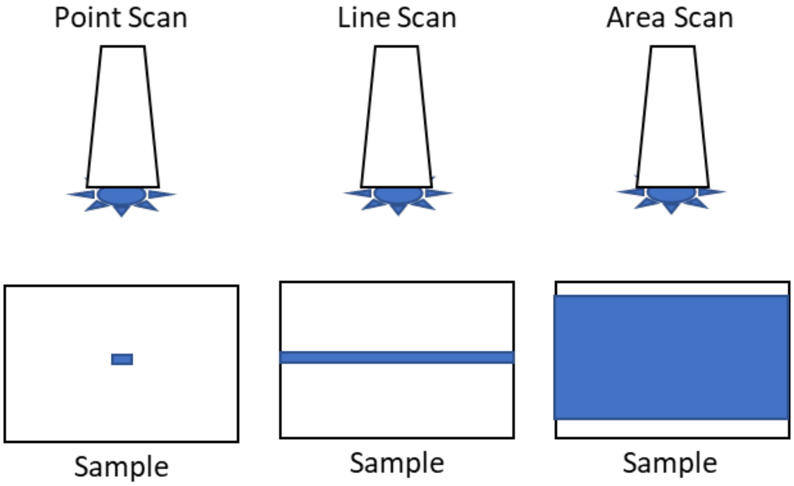

Figure 7. Different scanning options in HSI

Unlike the other imaging techniques, each hyperspectral image contains a large amount of data, as each pixel in the image has spatial and spectral data attached to it. Analysis of the image begins with a preprocessing step used to differentiate background sample and target sample spectral data. Discrimination and wavelet analyses such as principle component analysis (PCA) or Savitzky-Golay least squares method (S-G) have been used for this purpose. After preprocessing, data processing techniques such as PCA, linear discrimination analysis (LDA), Fisher discrimination analysis (FDA), and/or artificial neural networks (ANN) are used to classify each pixel. One way of classifying is using a process called library matching, where spectral information in the image is matched against known spectral libraries to identify unknown materials. If the spectral data resembles a material in the library closely enough to be statistically relevant, the pixel composition can be identified. Machine learning such as ANN can be used to refine matching capabilities over time. At the final step, quantification processes have been performed using either multivariate curve resolution (MCR), partial least squares regression (PLSR), ANN, and/or multi-linear regression (MLR). These methods are referred to as chemometric techniques and can be used to process raw data to generate qualitative and quantitative results. Different chemometric techniques are used for different purposes. For example, for detection studies, PCA, ANN, and MLR have been used; and for quantification purposes, PLS have been employed (Mukherjee & Gowen, 2015) (Calvini, et al., 2018) (Qin, et al., 2013) (Amigo, et al., 2013).

A number of studies have reported the use of HSI methods for detection of plastic contamination in foods. Díaz et al. (2011) used HSI to achieve automated detection of various types of foreign bodies on a pork steak on a conveyor belt. Their system successfully detected plastic materials of different colours, sizes, and types (PET and PE, hard plastic and film plastic). Burns and Nixon (2017) used MSI to differentiate rice-shaped plastics from real rice and to identify the plastic rice grains as foreign bodies. Shan et. al (2019) studied HSI acquisition of different types of plastic materials and organic particles on a filter paper, and were able to detect 8 different types of plastic and separate them from organic particles. Serranti, et al. (2015) used HSI to automate differentiation of plastic particles on a conveyor belt using a line scan camera. Gowen and O'Donnell (2013) applied an HSI system to identify and differentiate foreign bodies in grain products. Plastic shards, glass beads, and rubber fragments were used as foreign bodies. The study showed that HSI could detect and identify all types of foreign bodies among grain samples. Viewed collectively, the prior studies have identified the potential for HSI to identify plastics in variety of matrices.

On the basis of the information gathered HSI has substantial potential for characterizing plastics in wastes. Its ability to scan large areas, identify plastics from unknown substances, and analyze samples without special preparation steps make it a superior technique for identification analysis. It is a non-destructive method with a high signal-to-noise ratio. It can capture high-resolution images and prior knowledge of sample is not needed for characterization. Further, special, time consuming sample preparation steps are not required. However, HSI systems require large data storage capacity and rapid computation with advanced data processing techniques (Amigo, et al., 2013). Furthermore, the methodology has limited penetration capability, meaning that the quantification studies would depend on some assumptions of the thickness or shape of particles, as only the surface area perpendicular to the camera can be calculated from the image. An alternate approach for quantification in HSI is to form a calibration method based on known concentrations of known analytes to facilitate mass predictions of unknown materials after identification (Amigo, et al., 2015).

5.1.2 Nearline Techniques

Nearline techniques were defined as those that can perform sample analysis in the same facility as the digester. Appropriate sample collection is required for these techniques since the sample needs to be removed from the line. The instruments could be installed near the waste stream or in a separate room. It should be noted that the hyperspectral imaging system previously discussed could also be used in a nearline configuration.

5.1.2.1 Ultrasound Imaging

Unlike the other methods discussed in this report, which use electromagnetic radiation, ultrasound techniques use sound waves to characterize objects. Ultrasound refers to sounds with frequencies that are above the upper threshold of human hearing, which is approximately 20 kHz. Ultrasound imaging creates a greyscale image using these high-frequency sound waves and their echoes. Ultrasound systems generally consist of a transducer (source), an amplifier, and a detector (Figure 8). The propagation of the sound wave through a material depends on the acoustic impedance of that material. Hence, this technique creates images that are maps of the acoustic impedances of the materials through which the ultrasound has travelled (Khairi, et al., 2018).

The movement of sound waves can be drastically affected by air or air bubbles in a sample because the acoustic impedance of air is much lower than the acoustic impedance of solids. When ultrasound waves encounter an air bubble, most of the waves are reflected and this can result in loss of data. Several reports have indicated that ongoing enhancements of the technology are being developed to enable the use of ultrasound imaging techniques even in the presence of small air bubbles by using modern transducers (Chandrapala, et al., 2012) (Gallo, et al., 2018).

The applications of ultrasound technology in the food industry can be divided into 2 categories: low-intensity and high-intensity ultrasound. High-intensity ultrasound (HIU) has intensities higher than 1 W/cm^2 (watt per square centimetre) and is used for altering the physical, chemical, biochemical, or mechanical properties of substances (Awad, et al., 2012). HIU is a suitable tool for processing applications such as cleaning, cutting, etc. As HIU can be destructive, it was considered to be not suitable for non-destructive quality control purposes. In contrast, low-intensity ultrasound (LIU) is a commonly used non-destructive technique in the food industry (Gallo, et al., 2018) (Awad, et al., 2012). It has the ability to penetrate soft materials and detect foreign bodies without disturbing the food. Further, real-time measurements can be taken with this technique.

The sensing modes for the ultrasonic sensor are based upon either transmission, reflection, or diffraction modes. The transmission and reflection modes have been most commonly used. The only difference between these modes is the location of the detector. The detector is located across from the source in transmission mode, and in the same location as the source in reflection mode. The change in amplitude of the sound waves received by the detector in transmission or reflection mode allows the measurement of attenuation or acoustic impedance. These parameters could be used for characterization of target analytes. In terms of detection ability, the two modes are similar. Reflection modes are preferred in medicine while transmission modes are used in food industry for foreign body detection (Khairi, et al., 2018).

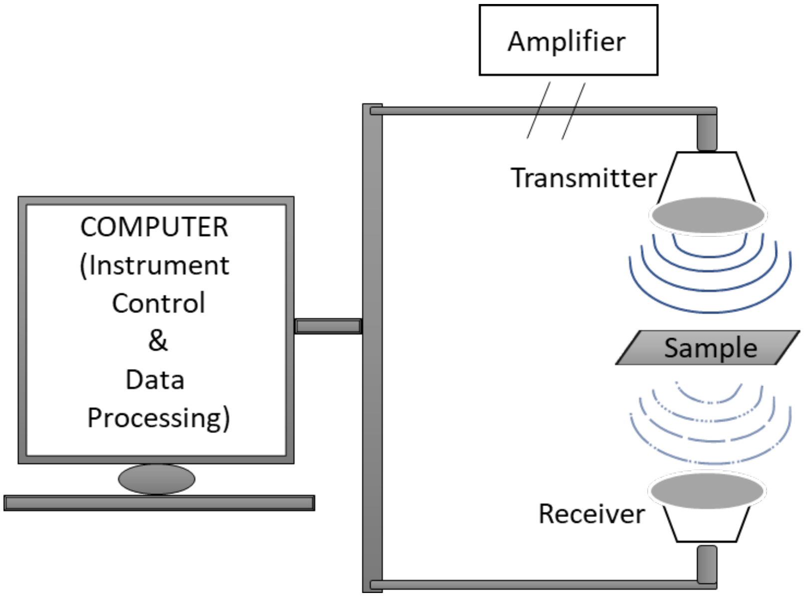

Figure 8. Schematic representation of ultrasound imaging setup

Several reports of the use of ultrasound for detecting foreign matter in foods were reviewed. Ho et al. (2007) successfully detected foreign bodies inside aluminium beverage cans using ultrasound imaging. A 6.6 mm copper rod and a 7.2 mm diameter aluminium rod were inserted into the cans, and these rods were able to be imaged and detected through the aluminium cans. Zhao, et al. (2003) studied detection of glass, metal, and plastic materials with different sizes in bottled beverages using an ultrasonic sensor. Transmission mode was used for this study, and foreign particles could be observed. Pallav, et al. (2009) investigated rubber, wood, and glass particles in cheese. The particles were successfully detected using ultrasound imaging, although the image resolution was low. These studies showed that ultrasound imaging techniques have the ability to detect plastics within a simple organic matrix.

From the reports that were reviewed it was concluded that ultrasound imaging is a rapid and simple technique. LIU is non-ionizing and non-destructive, so it can be used for food quality control and safety applications. The method has excellent penetration ability for detection of foreign bodies in packed or canned foods. Thickness measurements can be gathered using this method as well as the volume of samples. It does not require complicated chemometric techniques or processing tools like other methods. However, its ability to scan large areas at one time is limited. The image resolution is lower than other techniques; thus, the thickness, volume, and mass calculations may be less accurate. Due to the low resolution, it can also be difficult to distinguish which parts of the variation in the image are due to contaminants, and which are simply due to variation in the matrix. This ability to discern contaminants within a diverse sample matrix becomes important in an organic waste application, where there can be substantial background variation (see section 3.2). Additionally, since ultrasound is sensitive to the presence of air bubbles, a specialized sample holder compartment might be needed in some cases.

5.1.2.2 Terahertz Imaging

Terahertz (THz) imaging is a recently developed technology when compared to other techniques described in this report. The THz range of wavelengths lies between the microwave and the far infrared domains in the electromagnetic spectrum, and hence has wavelengths between 3-30 µm (Melo, et al., 2012). The use of the THz domain has received relatively little attention due to a lack of hardware and software that can utilize these frequencies. However, relatively recently, promising and efficient sources, detectors, and associated hardware and software have been developed. THz imaging is now used in applications related to food quality, agriculture, and safety technology (Wang, et al., 2017).

THz imaging is a spectroscopic technique that collects spectral data by measuring the interaction between matter and THz radiation. The THz imaging apparatus generally consists of a laser, a THz emitter (source), a sample chamber, and a THz detector (camera) (Figure 9). The laser is used to generate THz waves and the beam splitter is used to direct half of the laser energy to the detector to make the THz waves discrete signals. Similar to ultrasound imaging, THz instruments can perform analysis in either transmission or reflection mode depending on the detector type. Transmission mode measures how much radiation is transmitted through the substance while reflection mode measures how much radiation is reflected back off of the substance. Transmission mode is typically preferred due to the high penetration capability of THz and problems associated with the reflection mode such as multiple reflections interfering with each other (Wang, et al., 2017). Transmission and reflection modes have also been used at the same time to distinguish different substances.

THz waves can penetrate nonpolar dielectric materials such as fabric, paper, plastic, leather, wood, food, etc. to cause molecules to vibrate in characteristic ways. These attributes make it an extremely attractive tool for agri-food product inspection and low-density materials detection. THz is a lower-energy radiation than X-ray, which allows it to image lower-density materials (Lee, et al., 2012). Like the other spectroscopic methods, the chemometric techniques described in 5.1.1.1 are used to process spectral data. The Savitzky-Golay least squares method has been employed to differentiate background and sample data and smooth signals. LDM, PLS, SVM, and ANN have been used for processing and matching the spectral data generated from samples to identify and quantify unknown materials (Gowen, et al., 2012) (Wang, et al., 2017).

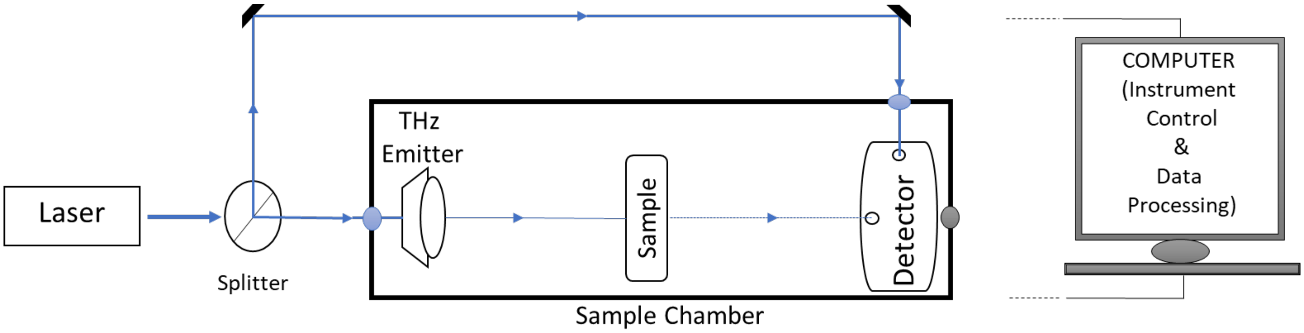

Figure 9. Schematic representation of THz imaging setup

Several reports of the application of THz imaging for measuring contamination of food were reviewed. Ok et al. (2014) used THz imaging to visualize plastics buried in a milk powder matrix. Various types of plastics were placed in a petri dish that was subsequently filled with dried milk powder. Identification of various hard plastic materials was then achieved using THz imaging. The shapes of the particles were detected with good resolution down to about 4 mm. Ok et al. (2019) inserted rubber, pepper seeds, plastic, and metals into wrapped chocolate bars and used THz imaging to identify these materials. A penetration depth of 20-30 mm was achieved, and identification and shape detection were successfully performed. Ikari et al. (2014) used THz to automate detection of foreign bodies in milk powder. Plastic particles with a size of 2 mm were placed on a metal sheet and were covered with 10 mm of milk powder. The THz technique was able to penetrate through the milk powder and detect the particles. When the reports were viewed collectively it appears that THz imaging could be used to identify plastic materials in an organic-rich matrix.

THz imaging is a non-destructive technique capable of performing analyses rapidly. Its penetration capability for low-density dielectric materials can also measure the thickness of materials. However, it is likely that the technique would need to be substantially improved for measurement of plastics in organic waste streams. It has relatively low resolution compared to other techniques. THz signals are highly attenuated in water, meaning samples would need to be fully dried before imaging (Lee, et al., 2012). Further, its application as a quantitative method must be improved as chemometric techniques have not been fully adapted to this technology.

5.1.3 Offline Techniques

In the current study, offline techniques were defined as those that require sample analysis to be conducted in a remote facility or laboratories. Hence, the need for appropriate sampling methods, sample storage, and sample transportation may be important factors that impact on the implementation of these techniques. Some factors that may contribute to the decision to use offline analysis include the size and cost of the instrument, safety requirements, and the need for full-time or specialist staff.

5.1.3.1 X-ray Imaging

X-ray imaging is capable of creating two-dimensional or three-dimensional images through the use of radiation that is in the region of the electromagnetic spectrum with wavelengths between 0.01 nm and 10 nm. It is a powerful (high frequency) form of radiation compared to IR or visible spectrum radiation and can therefore penetrate liquid, gas, and solid materials. Conventional X-ray imaging systems generate images using the X-ray absorption contrast differences of the substances being imaged. They consist of an X-ray generator source and a detector system. These are known as absorption X-ray systems, which can differentiate materials mostly depending on their density differences (Einarsdóttir, et al., 2016) (Haff & Toyofuku, 2008). Historically, X-ray imaging has been used in applications such as medicine, security, and industry.

X-rays have also been used to penetrate food products to visualize internal features of the food (typically within its packaging) and thereby detect physical defects or contaminants without damaging the product. (This method does not leave any residual radiation on the products and is safe for use in food.) However, conventional X-ray imaging methods are ineffective for characterizing non-metallic and/or low-density materials like glass and plastics particles, as the high-energy radiation will pass through these materials without being absorbed. In addition, it is challenging to distinguish plastic materials from food products using conventional X-ray as they have similar densities (Nielsen, et al., 2013).

Grating-based X-ray imaging systems have been developed to overcome some of the problems encountered in foreign body detection in the food industry. Unlike conventional absorption X-rays, grating-based X-ray imaging systems measure reflected and scattered rays in addition to absorption to enhance contrast resolution. Figure 10 presents a schematic of an X-ray imaging setup that uses gratings between the source and detector to distinguish and separately collect attenuated, scattered, and reflected rays (Nielsen, et al., 2013) (Einarsdóttir, et al., 2016). The system has three different modalities—absorption, dark field, and phase contrast—so it can create different images to detect different type of materials. The absorption mode is responsible for measuring attenuation, while dark field measures scattering and phase contrast measures reflection. For the identification of plastic materials, all three modalities may need to be used together as plastic materials have various physical properties such as density and shape.

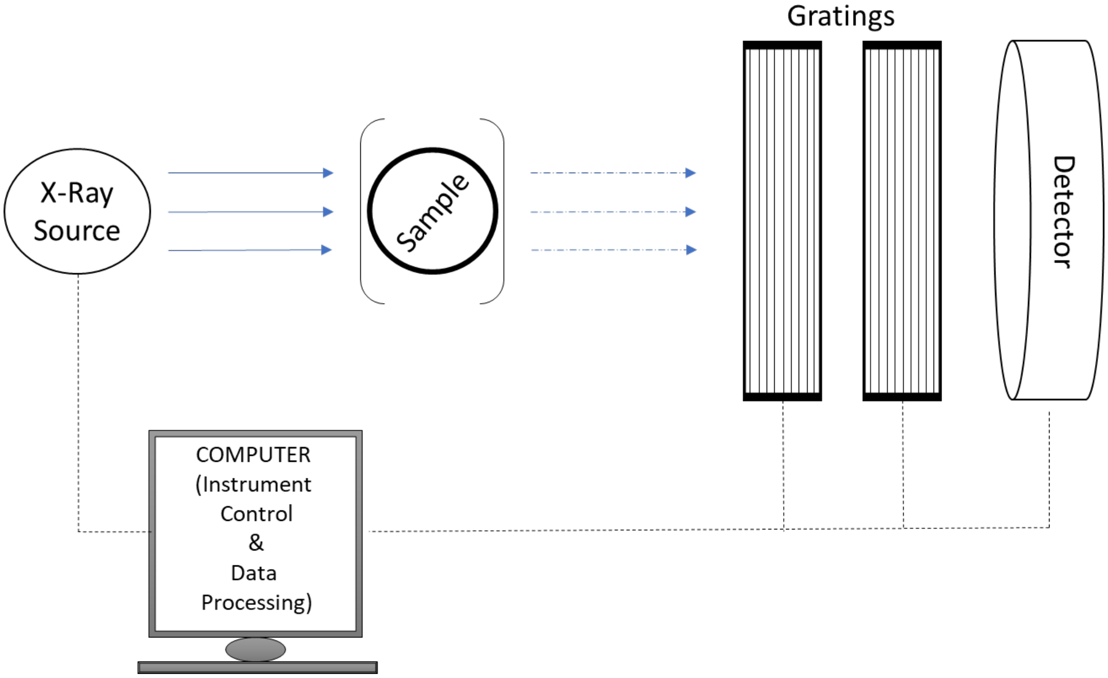

Figure 10. Schematic representation of X-ray imaging setup

The X-ray imaging process creates a high volume of data that needs to be processed. The first step in identification and quantification analysis is to differentiate the background signals from the target sample signals, as in other techniques. Next, image segmentation and processing steps are performed for identification and quantification analysis, respectively. In image segmentation, images are divided into small regions for improved investigation of samples with high resolutions. The Markov random field (MRF) and active appearance model (AAM) algorithms, which can be written in MATLAB, are the two examples of segmentation techniques. In the final step, ImageJ (National Institutes of Health), Octopus (XRE), Mimics (Materialise), or Avizo (FEI) image processing tools have been used to extract useful information (volume fractions, size distributions, shape, etc.) for quantification (Wang, et al., 2018).

Several reports of the use of grating-based X-ray imaging systems were reviewed. Li et al. (2015) studied the detection of low-density materials in food products. A PE plastic sample was inserted into milk powder, and a hollow poly-methacrylate cylinder was buried in minced pork meat. These samples were analyzed to demonstrate that X-ray scatter imaging could be used to detect low-density foreign bodies. Based on these findings, X-ray scatter imaging shows promise as a tool for non-destructive inspection and demonstrates relevance for quality assurance of food products in the food industry. Einarsdóttir, et al. (2016) investigated the advantage of using a grating-based X-ray technique over conventional X-ray imaging for foreign object detection. A novel detection algorithm was developed to compare the results of each X-ray imaging modality and to determine the benefits of applying multivariate and texture analysis. The results gave a clear indication of superior detection results from the grating-based method, and especially show promising detection results of plastic and glass materials. Nielsen, et al. (2013) demonstrated that dark-field X-ray techniques can detect low-density materials like plastics and insects. Viewed collectively it is apparent that current enhanced X-ray imaging methods can provide information on the identity, shape, volume and mass of plastics that are present in matrices that are similar to organic waste streams (Wang, et al., 2018).

On the basis of the reports reviewed, it was concluded that modern grating-based X-ray methods have many advantages. They can provide fast, high-resolution measurements that allow quantification analysis. They have excellent penetration capabilities that are useful in detecting hidden particles or to examine packaged/canned foods. No special sample preparation steps are needed. However, X-ray systems require a high-voltage power supply, which can be expensive. The cost of the instrument and its operation and maintenance are higher than most of the other instruments reviewed in this study. A significant disadvantage of X-ray imaging is that long-term exposure to X-radiation has negative health effects, causing damage to cells and potentially leading to cancer (Wang, et al., 2018). Hence, considerable health and safety protocols associated with this equipment would need to be implemented. However, after the imaging process is completed, there is no residual radiation left on the waste or on the digestate product.

X-ray imaging was deemed to be among the best candidates in this study on the basis of its penetration capability and the fast and high-resolution analysis. It is believed that quantification could be successfully achieved using existing image processing tools, and volume-based quantification is possible since X-ray imaging can produce three-dimensional images. For the purposes of this report, X-ray imaging has been classified as an offline technology due to the cost of the infrastructure and procedures required for its safe use. However, X-ray imaging systems have been used online and nearline in the food industry as the level of radiation emitted by the X-ray instruments used in food industry is low enough not to damage the food. Therefore, they have the potential to be employed safely for waste characterization. The main disadvantage of this technique is its limited ability to detect low-density materials, including plastics.

5.2 Indirect Techniques

The techniques discussed in this section have been defined as “indirect” because an isolation step is required before using these techniques. These instruments can only provide measurements for a single point with high precision as shown in Figure 7. Therefore, in addition to an isolation method such as purification, separation, or extraction, a special sample preparation step might also be needed for some instruments. Indirect methods were not subcategorized as they could all be used in a nearline mode. A discussion of plastics isolation techniques is provided prior to reviewing the indirect characterisation methods.

5.2.1 Plastic Isolation Methods

It is anticipated that in the intended application the quantity of plastics in samples will contribute only a small percentage of the organic waste composition. Hence, isolation of the plastics from the matrix is necessary to facilitate some characterization methods. A number of methods that could be used to separate plastic materials from organic-rich matrices have been reported in the literature. These methods have mostly been used for extracting plastics (especially microplastics) from wastewater and ocean water samples. At the end of this section, the applicability of these methods to the analysis of organic waste samples is further discussed. Although none of these methods work with 100% efficiency, efficiencies of up to 95% have been reported (Zhang, et al., 2019) (Sun, et al., 2019). Commonly reported plastics separation methods include sample digestion with hydrogen peroxide (H_2O_2), digestion with Fenton's reagent, enzymatic digestion, density fractionation, and Nile red staining (Prata, et al., 2019) (Bläsing & Amelung, 2018).

Several isolation methods based upon digestion of background organics have been reported. H_2O_2 digestion is a well-established method that typically uses a 30-35% H_2O_2 solution to oxidize the organic materials at temperatures around 50 °C. The reaction lasts 24-36 hours depending on the organic content (Hurley, et al., 2018) (Qiu, et al., 2016). Fenton's reagent is a mixture of 30-35% H_2O_2 solution and iron(II) sulfate ($FeSO_4$). The $FeSO_4$ catalyzes the H_2O_2 oxidation reaction, resulting in faster digestion (Prata, et al., 2019) (Tagg, et al., 2017) (Zhang, et al., 2019). Enzymatic digestion methods use enzymes such as Proteinase K or trypsin to digest organic materials. Enzymatic digestion can potentially yield high efficiencies, but enzymes are expensive and challenging to store when compared to H_2O_2 and Fenton's reagent (Cole, et al., 2014) (Courtene-Jones, et al., 2017).

Density fractionation is a common method used to separate plastic materials from soil matrices. The general method uses salt solutions such as NaCl, $CaCl_2$ NaI, or $ZnCl_2$, which can be prepared to have a density of 1.5-1.6 g/cm^3. These methods work well for plastics in soil matrices, which generally consist of background materials denser than plastic. Density fractionation becomes less effective as the density of the matrix becomes similar to that of the plastics. The densities of food materials and some plastics are similar to each other, so this method is not as effective at separating plastics from organic waste. Density fractionation has, however, been used as an additional separation step after pre-treatment by a digestion (Prata, et al., 2019) (Herrera, et al., 2018) (He, et al., 2018) (Imhof, et al., 2012).

Nile red is a lipophilic fluorescent dye that can be used to mark plastic materials as it adsorbs to hydrophobic materials such as plastics. In this method, the Nile red dye is initially dissolved in methanol to form a solution for the plastic staining. After staining, the plastic materials can be quantified using fluorescence techniques and image analysis software. However, this method was developed for ocean samples and may not work as well in an organic-rich matrix because naturally hydrophobic materials such as lipids and natural polymers present in the matrix may fluoresce as well (Maes, et al., 2017) (Shim, et al., 2016) (Erni-Cassola, et al., 2017).

Of all the methods reviewed, Fenton's reagent was deemed to be the most efficient and rapid. It is likely the most practical for separating plastics from organic waste because of the aforementioned challenges with the other methods. As organic waste contains much higher organic content as well as larger plastics and other foreign bodies than wastewater or ocean water, it might not be convenient to apply digestion reagents directly to the sample. A potential strategy to reduce the consumption of reagents would be to integrate it with the method described in section 4. Plastics and organic materials larger than 4 mm could initially be separated by hand. The digestion method could then be used on samples retained on the 2 mm sieve, as plastics become difficult to identify with the naked eye below 4 mm. This approach would limit the amount of organic waste digested and thus also limit the reagent required.

It is unknown how effective digestion methods will be for an organic-rich application, as few previous studies on the subject were identified. Karami, et al. (2017) used 60 mL of H_2O_2 (35%) to digest 6 g of fish muscle and skin spiked with microplastic particles. They achieved 98.3% digestion efficiency at 50 °C and 100% digestion efficiency at 60 °C, with plastic recovery rates between 84-105% for different types of plastics. Other studies (Hurley, et al., 2018) (Tagg, et al., 2017) have been conducted on samples with less concentrated organics, such as wastewater and soil samples, and confirmed the efficiency of Fenton's reagent for dilute organics.

5.2.2 Indirect Plastic Characterization Techniques

Several characterization techniques for identification and quantification of plastics once they have been separated from the background matrix have been reported. Particles larger than 5 mm could be detected by the naked eye and collected manually as in the conventional method described in Section 2. Once collected the particles can be weighed for quantification. However, in some cases, it may be difficult to differentiate hard plastics from glass, stone, and organic materials. Additionally, for particles smaller than 5 mm, detection and collection can prove difficult. For these reasons, a suitable and precise analytical instrument would be preferable. Raman, FTIR, Mass Spectroscopy (MS), thermal gravimetric analysis (TGA), or differential scanning calorimetry (DSC) and Nuclear Magnetic Resonance (NMR) have been employed for these purposes. In this section, the principles, properties, advantages and disadvantages of these instruments are discussed (Silva, et al., 2018) (Sun, et al., 2019).

FTIR spectroscopy (Figure 11-a) and Raman spectroscopy (Figure 11-b) are similar techniques that employ radiation with wavelengths located around the IR region of electromagnetic spectrum. Both can be used to provide information about the vibrational modes of molecules. The main difference between the two techniques is the type of molecular vibrations used to determine the structure of the molecule. FTIR measures how much light is absorbed, transmitted, or reflected by the bonds of a vibrating molecule, while Raman measures the energy that is scattered after being excited by a laser (Araujo, et al., 2018) (Alassali, et al., 2018) (Mecozzi, et al., 2016). Both techniques can be used to characterize plastic materials because of the unique behaviors of the plastics in and around the IR region of the electromagnetic spectrum. Furthermore, substantial libraries of plastic materials exist for both techniques.

The key advantage of Raman spectroscopy is that it requires little to no sample preparation, while the FTIR method has constraints on sample thickness, uniformity, and dilution to avoid saturation. Moreover, Raman can detect particles with smaller sizes and deals with water absorption peaks better than FTIR. However, FTIR is considered to better address interferences coming from the background and overlapping signals due to the use of an interferometer and the Fourier Transform algorithm employed in signal processing. Chemometric techniques such as ANN, PCA, LDA, FDA, and PLS can be used to analyze the spectral data obtained in Raman and FTIR. These techniques are used to provide identification, concentration, and mass determination analysis (Caponigro, et al., 2019) (Brinton, 2005) (Elert, et al., 2017).

FTIR and Raman are well established in plastic processing facilities for identification, sorting, quality control, and quantification purposes. The superior response of these devices to plastic materials makes them suitable techniques for plastic characterization analysis. After separating plastic materials from wastewater, ocean water, or soil samples, these two techniques can be used for characterization purposes. Bay, et al. (2016), Harrison, et al. (2012) and Simon, et al. (2018) used FTIR to quantify plastics in wastewater. Scheurer, et al. (2018) and Piehl, et al. (2018) studied identification and quantification of microplastics and macroplastics in soil by using Raman and FTIR spectroscopy. These studies show that if plastic materials can be separated from the matrix, these techniques could be able to be used for identification and quantification analysis.

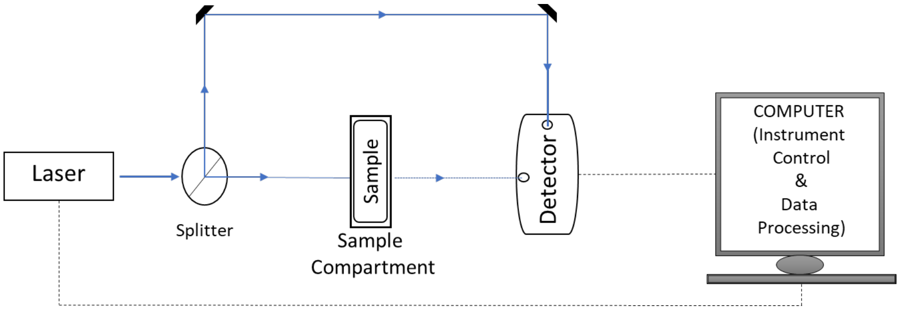

Figure 11-a. Schematic representation of FTIR Spectroscopy setup

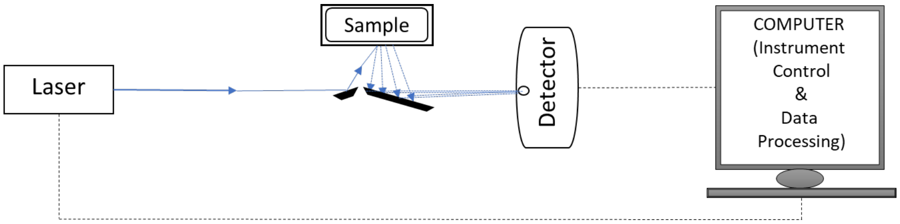

Figure 11-b. Schematic representation of Raman Spectroscopy setup

Mass spectroscopy (MS) or tandem MS (MS/MS) is a technique based on accurate mass measurement analysis, and it can generate rich chemical information from a substance. It can be used to quantify known materials, to identify unknown compounds within a sample, and to elucidate the structure and chemical properties of different molecules. It is a destructive technique which studies the effect of ionizing energy on molecules. Multiple ions are generated from the sample, and then separated according to their specific mass-to-charge ratio and the relative abundance of each ion type is recorded (Sun, et al., 2019) (Elert, et al., 2017). The most commonly used MS type for the polymer/plastic characterization is Pyrolysis Gas Chromatography Mass Spectroscopy (Pyr-GC-MS). In this technique, mass spectroscopy is combined with gas chromatography (GC). GC is a chromatographic technique used to separate substances that can be vaporized without decomposition. Pyr-GC-MS is a well-established tool for polymer/plastic analysis and has been widely used to study polymer structure, composition, molecular weight, molecular weight distribution, bulk and surface properties, and impurity content. Pyr-GC-MS is a highly sensitive technique. Rapid analysis can be done for many polymer samples, and prior sample preparation specific to this instrument is not needed. However, MS can only analyze samples on a mg level; therefore, this technique is not ideally suited for analysis of complex samples such as organic waste (Dumichen, et al., 2017) (Dekiff, et al., 2014) (Fries, et al., 2013). However, it remains a good fit for the characterization of microplastics (Nuelle, et al., 2014).

Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) are examples of thermal analysis techniques. These techniques measure physical properties as a function of temperature, time, and other variables. TGA measures the physical change of a substance by heating it gradually in a closed vessel. The change in mass is measured with changing temperature. DSC measures the substance by heating it gradually along with a reference substance in a separate vessel. The change in mass and the change in physical state (e.g. melting) can be observed with this technique. Identification of plastic materials with these techniques is possible, but it is complicated when the plastics are present in a mixture of unknown or complex components. TGA and DSC generate a single signal curve at the end of the analysis, and consequently it is difficult to differentiate the signals coming from different components (Menczel, et al., 2009) (Schindler, et al., 2017). Studies of the identification of microplastics using TGA and DSC techniques were found in the literature. Using known plastics as reference materials, the identification of less-complex plastics was achieved. Quantification studies have been carried out by matching peak areas of the DSC curves of unknown samples with those of reference materials. However, as the number of unknown substances increased, the peaks from each substance interfered with each other more, making quantification analysis impossible (Majewsky, et al., 2016).

Nuclear Magnetic Resonance (NMR) spectroscopy is one of the most efficient characterization techniques in chemistry. It has become popular because of its ability to provide structural information down to atomic level. It makes use of superconducting magnets which generate a magnetic field to measure the resonant frequencies of molecules (Elipe, 2003) (Dykstra, 2006). Most commonly used NMR methods are proton (^{1}H) and carbon (^{13}C) analysis, which respectively determine the types and number of hydrogen atoms or carbon atoms present in a molecule. NMR techniques could be used for polymer characterization since all plastic materials contain carbon and hydrogen atoms. It can provide physical, chemical, and structural information from organic compounds in liquid or solid form. It has been primarily used for the identification purposes, but it has also been reported to be a valuable tool for absolute and relative quantitative measurements such as concentration and mass (Brandolini & Hills, 2000) (Adams, 2019). NMR instruments traditionally require a large-scale magnet system which is costly and must be laboratory-based. Recently, with developing magnet technology, lower cost, portable NMR devices have become widespread. Although portable devices cannot generate various types of results simultaneously like laboratory-based devices, different types of portable devices can be developed for different measurement applications (Blumich & Singh, 2018) (Adams, 2019). This technique may have problems with signal overlapping and interference with more complex samples. NMR stands as a good technique for polymeric material characterization, but it may not be suitable for complex organic samples.

With regard to simplicity, cost, practicability, identification, and quantification abilities, the FTIR and Raman spectroscopic techniques appear to be the most suited for indirect organic waste applications.

6 Critical Analysis

In this section, the direct and indirect techniques were separately scored with respect to selected criteria, which were weighted based on their importance to a plastic quantification system. The scores of each technique are compared and discussed for each criterion. The current state of technology development and information gathered from commercial vendors are also discussed.

6.1 Direct Techniques

Direct techniques were scored with respect to six criteria: plastic detection, quantification, resolution, area scan, penetration, and cost. These categories are defined as follows:

- Plastic detection: The ability to detect and identify plastics in an organic waste matrix. Plastics have different chemical structures and physical properties from both each other and other substances in the matrix. Characterization techniques should have the ability to both detect plastics and differentiate them from other substances.
- Quantification: The ability to quantify plastics by mass or volume. Quantification can be accomplished by measuring surface area and calculating volume of the samples, or by directly measuring the mass of the material using signal peak intensity differences.
- Resolution: The potential resolution of the images produced. Resolution is important in detecting and differentiating small particles.
- Area scan: The ability to scan large areas of sample at a time or in a short time.
- Penetration: The depth of signal penetration in organic waste samples. The penetration capability of various techniques depends on some physical properties of the substance to be imaged, such as density and color. Penetration in low density materials like food and plastic are considered a reference.
- Cost: The combined cost of initial purchase, including all parts of instrument and software, and operation/maintenance costs.

The main aim of the project is mass-based quantification of plastic materials in an organic waste matrix. In the light of this purpose and by evaluating the physical and chemical conditions of samples, sample area, thickness, and feasibility, each category was weighed as follows:

Plastic detection	3x
Quantification	3x
Resolution	2.5x
Area scan	2x
Penetration	2x
Cost	1x

Two score tables were created for direct techniques. In Table 4, the techniques were scored on a scale of 1-5 on the basis of the findings of the literature review (Section 5). The justification of the scores is subsequently presented. Table 5 then provides the scores after multiplication by the weight constants listed above to calculate the final scores.

Table 4. Scores assigned to direct techniques

	Plastic Detection	Quantification	Resolution	Area Scan	Penetration	Cost
HSI	5	4	5	5	2	3
X-Ray	2	4	3	5	5	2
THz	3	3	2	3	4	3
Ultrasound	3	3	2	2	4	4

Table 5. Weighted scores for direct techniques

	Plastic Detection	Quantification	Resolution	Area Scan	Penetration	Cost	Row Sum
HSI	15	12	12.5	10	4	3	**56.5**
X-Ray	6	12	7.5	10	10	2	**47.5**
THz	9	9	5	6	8	3	**40**
Ultrasound	9	9	5	4	8	4	**39**

The ability to detect plastics was a highly weighted criterion. HSI using the Vis and IR regions of the electromagnetic spectrum received the highest score since plastic materials show unique properties in those regions that make the detection easier. THz and ultrasound received intermediate scores as they are proven techniques that have been used in the food industry for low-density material (plastic) detection. X-ray technologies received the lowest score in this category as they employ a high-energy radiation that is less likely to detect plastic materials.

The quantification ability of the techniques was another highly weighted criterion for the intended application with HSI and X-ray receiving high scores in this regard. HSI has good surface area-based quantification, and mass/volume-based quantification can be achieved using peak intensities in the spectra to calculate relative mass of the particle. X-ray imaging can generate 3D images and directly quantify mass. THz and ultrasound received lower scores. They can provide thickness and surface area measurements for volume calculations, but due to low resolution and lack of ability to selectively detect plastics, the quantification process cannot currently be automated.

The resolution of these techniques was a moderately weighted criterion in the analysis. HSI received a high score because it uses a high-definition camera to generate high resolution images. X-ray received an intermediate score as it can generate 3D images with high resolution; however, it may not be effective at imaging smaller low-density particles. THz and ultrasound received low scores in this criterion. The resolution of THz images would need to be improved to compete with HSI and X-ray. Ultrasound is sensitive to variability in the matrix and would not be able to distinguish plastics from noise.

The area scan ability of the techniques was a moderately weighted criterion in the analysis. HSI and X-ray received high scores because they can be set up to scan large areas. THz and ultrasound received intermediate and low scores, respectively, because they have limited source and detector mounts, so the scanned sample area is limited.

The penetration ability of the techniques was a moderately weighted criterion in the analysis. X-ray received the highest score because its high-energy signal provides excellent penetration through low-density materials. HSI received a low score because it uses Vis and IR beams, and their penetration through low-density materials is limited. Although THz has a lower frequency than IR radiation, it has a good penetration capability in low-density materials, so it received an intermediate score. Ultrasound also received an intermediate score as its penetration capabilities were reported to be sufficient to examine canned or packed food.

Cost was the lowest-weighted criterion in the analysis. X-ray is the most expensive system, as it has high capital and operating costs, so it received a low score. In contrast, ultrasound is a low-power device with a relatively low capital cost, so it received a high score. HSI and THz were assigned intermediate scores in this criterion.

On the basis of the final scores (Table 5), HSI and X-ray were identified as the technologies that have the highest potential of being adapted for the intended application. They are both good candidates for plastic characterization in organic waste. While HSI stands out with its ability to detect plastic materials and generate high resolution images, X-ray stands out with its penetration ability. However, the X-ray techniques may have challenges with respect to detection of plastic materials in some cases.

6.2 Indirect Techniques

The indirect techniques were scored on the basis that they would be employed after a suitable separation technique (discussed in section 5.2). These techniques were scored with respect to five criteria: plastic detection, quantification, sample size, sample complexity, and cost. Plastic detection, quantification, and cost were defined in section 6.1. The remaining categories are defined as follows:

- Sample Size Limit: The limit of sample mass or volume that the instrument can analyze at one time.
- Sample Complexity: The ability of the instrument to analyze complex samples that contain a variety of different substances. The complexity of the sample could cause interferences or overlapping of the peaks in a spectrum that makes differentiation difficult, as discussed in section 5.

The categories were weighted as follows:

• Plastic detection	3x
• Quantification	3x
• Sample size limit	2x
• Sample complexity	2x
• Cost	1x

Two score tables were created to compare the indirect techniques. In Table 6, the techniques were scored on a scale of 1-5 on the basis of the findings of the literature review (Section 5). The justification of the scores is subsequently presented. Table 7 provides the scores after multiplication by the weight listed above to calculate the final scores.

Table 6. Score table for indirect techniques

	Plastic Detection	Quantification	Sample Size Limit	Sample Complexity	Cost
FTIR	5	5	4	4	4
Raman	5	5	5	3	4
Pyr-GC-MS	5	5	1	3	2
TGA	3	1	2	2	4
DSC	3	3	2	2	4
NMR	4	4	2	3	2

Table 7. Weighted score table for indirect techniques

	Plastic Detection	Quantification	Sample Size Limit	Sample Complexity	Cost	Row Sum
FTIR	15	15	8	8	4	**48**
Raman	15	15	10	6	4	**48**
Pyr-GC-MS	15	15	2	6	2	**40**
TGA	9	3	4	4	4	**24**
DSC	9	9	4	4	4	**30**
NMR	12	12	4	6	2	**34**

The plastic detection and quantification capabilities of these techniques were highly weighted criteria in the analysis. As discussed in section 5.2.2, FTIR, Raman, Pyr-GC-MS, and NMR are favorable instruments for detection, identification, and quantification analysis as long as the samples are clean and homogenous. Hence they received the higher scores in these criteria. TGA and DSC cannot quantify effectively; hence they received relatively lower scores.

The sample size limit of the techniques was a moderately weighted criterion. Sample size is important to this study because in order to achieve more accurate and precise results, a sample large enough to represent the whole batch should be analyzed. FTIR and Raman spectroscopies received high scores because they can analyze a higher sample mass at a time than Pyr-GC-MS, NMR, TGA, and DSC. However, the sample mass of all indirect techniques is limited to the milligram level.

The sample complexity of the techniques was a moderately weighted criterion. Organic waste is a complex material that contains various substances, as discussed in section 3. FTIR, Raman, Pyr-GC-MS, and NMR received high or intermediate scores because they have high resolution and accuracy as well as spectrum processing methods discussed in section 5.2.2, so they could deal with most of the interference and overlapping peaks. TGA and DSC received low scores because they generate only one broad curve, and hence it is difficult to distinguish peaks from different sources within the sample.

Cost was the lowest-weighted criterion in the analysis. NMR is the most expensive system, as it has a high capital cost due to the high cost magnets. Pyr-GC-MS is also an expensive instrument as it has a tandem structure and contains different techniques. These techniques both received low scores. By contrast, FTIR, Raman, TGA, and DSC are relatively simple devices with low capital cost, so they received high scores.

According to the final scores of the indirect techniques, FTIR and Raman spectroscopies are considered to be most feasible for adapting to plastic characterization in organic waste if samples can first be isolated and cleaned properly. As the sample size limits of these techniques are low, it is suggested that these methods are more suitable for microplastic characterization, rather than macroplastics such as this study seeks to characterize in organic waste.

7 Current State of Technology

7.1 Commercial Vendors

An environmental scan of technology vendors was performed to gauge the extent of existing plastic identification and sorting technology currently being used in full-scale operating facilities. A number of potentially viable technologies were first identified. These initial investigations identified technologies such as hyperspectral, multispectral, and X-ray imaging systems that are being employed in the municipal solid waste and material recycling facility sectors. Then, an internet search was conducted to identify existing commercial vendors that provide these technologies (listed in Appendix B). Lastly, attempts were made to contact as many of these vendors as possible.

Through this desktop study, it was determined that there are currently applications in place that achieve similar goals to the plastic identification objective of this study. For example, the Norwegian multispectral/hyperspectral imaging company Tomra, which is the largest waste sorting company of its kind in the world, has more than 15,000 installations worldwide. These Tomra sorting systems are used by customers in various fields such as recycling, mining, and bulk food wastes. Other vendors, such as MachineX, also have established hyperspectral imaging sorting installations that primarily separate materials collected from municipal recycling programs. Therefore, a significant body of knowledge and experience exists in the marketplace already that is relatable to the overall goals of this project.

However, distinctions must be made between the existing technologies and installations, and some of the technical requirements of this project. For example, nearly all of the current sorting installations in the world are based on dry material, not wet SSO materials and not wet slurries. The presence of wet materials poses a number of challenges, such as equipment handling characteristics, sensor characteristics, separation mechanisms, etc. Another challenge is the size and variability of the plastic contaminant. Therefore, even though a related industry exists, additional development is required to apply the lessons learned in the recycling industry and to customize the existing technologies to fit the goals and objectives of this current project.

7.2 Enhancements Required for Current State of Technologies

A number of enhancements have been identified that may be required to put selected techniques into practice. As mentioned previously, organic waste slurries have different properties than those prevalent in recycling, municipal solid waste and hence it is expected that modifications of the techniques will be needed.

The suggested enhancements would allow the techniques to provide information that would be useful for optimization of plastics removal systems. In this regard, a plastic characterization system would output separate mass information for hard plastics and film plastics. For hard plastics, it would be desirable to provide information on the distribution of the mass of plastics as a function of particle size. That is, particle size would be binned, and a mass quantity determined for each bin, so that the size of particles that are contributing the most to the total plastic mass could be discerned. For film plastics, only a single mass output would be necessary since film plastics cannot be effectively characterized by volume or other “size” quantities. In addition, techniques that can discriminate the various types of plastic present and thus output this nformation may be preferred as it may provide insight into the source s of materials that are problematic for plastics removal equipment. The collective information could be used for depacking optimization, classification, source analysis, etc.

The necessary and optimal enhancements have been hypothesized for HSI, X-ray imaging, and FTIR/Raman spectroscopy techniques in the following sections. Enhancements were organized into two main categories: software and hardware.

7.2.1 Hyperspectral Imaging

The primary limitations of HSI are that it has low penetration and it cannot identify particles underneath a layer of organic grime. HSI could be used online if enhancements are employed to overcome the aformentioned problems. Alternatively, it could be used nearline, enabling samples to be rinsed and spread out before the hyperspectral image is captured and thus reducing the issues associated with the ability of HSI to characterize plastics through a layer.

The enhancements that have been identified are as follows:

HSI as an online technique:

- Software enhancements
    - Artificial intelligence (AI) and machine learning techniques could be employed to allow the system to discern and characterize plastics that are partially masked by organic materials
    - Algorithms that provide separate outputs for hard and film plastics, as well as a mass/volume distribution for hard plastics are needed
- Hardware enhancements
    - A sampling channel that diverts a portion of the sampled stream from the main stream which is shallow enough for penetration of NIR signals would be required for on-line applications.

HSI as a nearline technique:

- Software enhancements
    - Less sophisticated AI and machine learning techniques could be employed to allow the system to discern and characterize plastics that have a reduced cover of organic materials
    - Algorithms that provide separate outputs for hard and film plastics, as well as a mass/volume distribution for hard plastics are needed
- Hardware enhancements
    - A sample holder that provides a shallow layer of material to ensure adequate signal penetration would be needed

    - A rinsing mechanism that is integrated with the sample holder would be desirable

7.2.2 X-ray Imaging

The primary limitation of X-ray imaging is that its measurements depend on density differences between target materials and background materials. In addition, discrimination of different types of plastics cannot be achieved accurately. Since the densities of organic waste samples are very similar to the densities of plastics, and the densities of plastics are very similar to each other, the capability of the existing technology to characterize plastics in organic waste needs to be evaluated.

The enhancements that have been identified are as follows:

- Software enhancements
    - Algorithms that report mass/volume distribution information and separate hard/film information are required
- Hardware enhancements
    - Hardware enhancements that improve similar-density discrimination capabilities may be needed

7.2.3 FTIR Spectroscopy and Raman Spectroscopy

The primary limitations of FTIR and Raman spectroscopy is the small sample sizes that can be accomodated (as they are point-scan techniques) and, in addition, the plastics need to be isolated from the matrix before characterization. Fenton's reagent or H_2O_2 digestion may be employed for isolation of plastics however, the sample scan area would need to be expanded to provide high volume sample analysis, as a relatively high volume of sample must be analyzed to represent the whole batch. FTIR and Raman imaging techniques should be investigated as a possible solution to the area problem. FTIR and Raman imaging techniques have lower resolution than HSI however, this limitation may be addressed through the use of isolation and rinsing methods to make the sample less complex (Su & Sun, 2018).

8 Conclusions and Recommendations

This report presents a review of alternatives to conventional techniques for characterizing plastics in organic waste. Hyperspectral imaging, ultrasound imaging, terahertz imaging, X-ray imaging, FTIR spectroscopy, Raman spectroscopy, MS, TGA, DSC, and NMR were reviewed and assessed for their applicability in this context. The techniques were categorized as direct and indirect techniques according to their ability to achieve characterization without the need for an isolation method. Direct techniques were further subcategorized as online, nearline, and offline, while all indirect techniques were considered nearline.

As a direct and potentially online technique, HSI was determined to be the most promising technique reviewed. It is the only technique that can classify a wide area of materials based on their spectra, which is unique to each type of material, rather than on properties such as density or acoustic impedance, which may be similar between the plastics and the matrix.

A rinsing and sieving technique described in this report could potentially be used in combination with HSI and the samples could be imaged directly from the sieves. The combined methods have not been tested; further experiments using an HSI system would be required to confirm the effectiveness of this method.

X-ray imaging as an offline technique was the second-ranked technique among the direct methods due to its superior penetration capability. It did not surpass HSI due to its inability to detect low-density plastics and its inability to discriminate plastics from other similar-density materials.

FTIR or Raman spectroscopy were identied as the top choices for indirect methods. FTIR and Raman are proven techniques in the field of plastic characterization. The main disadvantage of these techniques is their inability to analyze large areas or quantities. If Fenton's reagent or H_2O_2 digestion methods were successfully implemented and plastic materials and other impurities were successfully collected, FTIR or Raman spectroscopy could subsequently be used to identify and quantify plastics in the intended application.

Technology vendors were surveyed to identify plastic characterization techniques that are currently being employed in waste management and similar industries. The survey revealed that HSI and X-ray imaging technologies are being used in somewhat similar applications but are not being employed in a manner that is directly transferrable to quantification of plastics is organic slurries. Enhancements of the highest ranking techniques that would fill the gaps between the current technologies and the preferred applications were identified. The enhancements typically involved a combination of hardware and software modification. Furthermore, the integration of HSI and X-ray imaging systems should be investigated as it could provide simultaneous identification, discrimination, volume, and quantification abilities. On the basis of the information gathered and analysis conducted in this study, it is recommended that the selected techniques be rigorously tested with real waste matrices. Based on the results of these tests, these methods may be integrated into QA/QC protocols for ensuring that plastics removal equipment if functioning appropriately.

References

ABC, 2016. *Digestate Standard Testing & Certification Program.* s.l.:s.n.

Adams, A., 2019. Non-destructive analysis of polymers and polymer-based materials by. *MAgnetic Resonance Imaging compact NMR,* Volume 56, pp. 119-125.

Alassali, A., Fiore, S. & Kuchta, K., 2018. Assessment of plastic waste materials degradation through near infrared spectroscopy. *Waste Management,* Volume 82, pp. 71-81.

Amigo, J., Marti, I. & Gowen, A., 2013. Hyperspectral imaging and chemometrics: A perfect combination for the analysis of food structure, composition and quality. *Data Handling in Science and Technology,* Volume 28, pp. 343-370.

Amigo, J. M., Babamoradi, H. & Elcoroaristizabal, S., 2015. Hyperspectral image analysis: A tutorial. *Analytical Chimica Acta,* Volume 896, pp. 34-51.

Araujo, C., Nolasco, M., Ribeiro, A. & Ribeiro-Claro, P., 2018. Identification of microplastics using Raman spectroscopy: Latest developments and future prospects. *Water Research,* Volume 142, pp. 426-440.

Awad, T. et al., 2012. Applications of ultrasound in analysis, processing and quality control of food: A review. *Food Research International,* Volume 48, pp. 410-427.

Bayo, J., Olmos, S., Lopez-Castellanos, J. & Alcolea, A., 2016. Microplastics and microfibers in the sludge of a municipal wastewater treatment plant. *International Journal Sustaniable Development Planning,* Volume 11, pp. 812-821.

Bernstad, A., Malmquist, L., Truedsson, C. & Cour Jansen, J., 2013. Need for improvements in physical pretreatment of source-separated household food waste. *Waste Management,* 33(3), pp. 746-754.

Bläsing, M. & Amelung, W., 2018. Plastics in soil: Analytical methods and possible sources. *Science of the Total Environment,* Volume 612, pp. 422-435.

Blumich, B. & Singh, K., 2018. Desktop NMR and its applications from materials science to organic chemistry. *Angewandte Chemie,* Volume 57, pp. 6996-7010.

Bouyé, C., Robin, T. & Humières, B., 2018. *Spectral imaging spreads into new industrial and on-field applications.* s.l., s.n., pp. 1-9.

Brandolini, A. & Hills, D., 2000. *NMR Spectra of Polymers and Polymer Additives.* New York: Marcel Dekker, Inc.

Brinton, W., 2005. Characterization of man-made foreign matter and its presence in multiple size fractions from mixed waste composting. *Compost Science & Utilization,* Volume 13, pp. 274-280.

BSI, 2014. *Specification for whole digestate, separated liquor and separated fibre derived from the anaerobic digestion of source-segregated biodegradable materials,* s.l.: s.n.

Burns, M. & Nixon, G., 2017. *Final report: Application of multispectral imaging (MSI) to food and feed sampling and analysis,* s.l.: LGC Group.

Calvini, R. O. G., Foca, G. & Ulrici, A., 2018. Development of a classification algorithm for efficient handling of multiple classes in sorting systems based on hyperspectral imaging. *Journal of Spectral Imaging,* Volume 7, pp. 1-15.

Caponigro, V. et al., 2019. Raman and Fourier transform infrared hyperspectral imaging to study dairy residues on different surfaces. *Journal of Spectral Imaging,* Volume 8, pp. 1-20.

CEC, 2017. *Characterization and Management of Organic Waste in North America—Foundational Report,* Montreal, Canada: Commission for Environmental Cooperation.

Chandrapala, J., Oliver, C., Kentish, S. & Ashokkumar, M., 2012. Ultrasonic in food processing - Food quality assurance and food safety. *Trends in Food Science & Technology,* Volume 26, pp. 88-98.

City of Ottawa, n.d. *Green bin and leaf and yard waste.* [Online]
Available at: https://ottawa.ca/en/residents/garbage-and-recycling/green-bin-and-leaf-and-yard-waste
[Accessed 26 March 2019].

Cole, M. et al., 2014. Isolation of microplastics in biota-rich seawater samples and marine organisms. *Scientific Reports,* Volume 4, pp. 1-8.

Courtene-Jones, W. et al., 2017. Optimisation of enzymatic digestion and validation of specimen preservation methods for the analysis of ingested microplastics. *Analytical Methods,* Volume 9, pp. 1437-1445.

Dekiff, J., Remy, D., Klasmeier, J. & Fries, E., 2014. Occurrence and spatial distribution of microplastics in sediments from Norderney. *Environmental Pollution,* Volume 186, pp. 248-256.

Díaz, R. et al., 2011. Hyperspectral system for the detection of foreign bodies in meat products. *Procedia Engineering,* Volume 25, pp. 313-316.

Dumichen, E. et al., 2017. Fast identification of microplastics in complex environmental samples by a thermal degradation method. *Chemosphere,* Volume 174, pp. 572-584.

Dykstra, R., 2006. *The Development of a Spectrometer for Portable NMR Systmes.* s.l.:Physics at Massey University.

Einarsdóttir, H. et al., 2016. Novelty detection of foreign objects in food using multi-modal X-ray imaging. *Food Control,* Volume 67, pp. 39-47.

Einarsdóttir, H., Ersbøll, B. K. & Larsen, R., 2016. *Image Analysis for X-ray Imaging of Food.* s.l.:Technical University of Denmark (DTU).

Elert, A. et al., 2017. Comparison of different methods for MP detection: What can we learn from them, and why asking the right question before measurements matters?. *Environmental Pollution,* Volume 231, pp. 1256-1264.

Elipe, M., 2003. Advantages and disadvantages of nuclear magnetic resonance spectroscopy as a hyphenated technique. *Analytica Chimica Acta,* Volume 497, pp. 1-25.

Erni-Cassola, G., Gibson, M., Thompson, R. & Christie-Oleza, J., 2017. Lost, but found with Nile red: A novel method for detecting and quantifying small microplastics (1 mm to 20 µm) in environmental samples. *Environmental Science & Technology,* Volume 51, pp. 13641-13648.

Frank, H., Markus, W. & Sixto, A., 2015. *Identification and sorting of plastics film waste by NIR-Hyperspectral Imaging.* s.l., s.n., pp. 9-14.

Fries, E. et al., 2013. Identification of polymer types and additives in marine microplastic particles using pyrolysis-GC/MS and scanning electron microscopy. *Environmental Science Processes & Impacts,* Volume 15, pp. 1949-1956.

Gajst, T., 2016. Analysis of Plastic Residues in Commercial Compost - Bachelor Thesis.

Gallo, M., Ferrara, L. & Naviglio, D., 2018. Application of Ultrasound in Food Science and Technology: A Perspective. *Foods,* Volume 7, pp. 1-18.

Gowen, A. A. & O'Donnell, C. P., 2013. Near infrared hyperspectral imaging for foreign body detection and identification in food processing. *Spectroscopy Europe,* Volume 25, pp. 6-11.

Gowen, A., O'Sullivan, C. & O'Donnell, C., 2012. Terahertz time domain spectroscopy and imaging: Emerging techniques for food process monitoring and quality control. *Trends in Food Science & Technology,* Volume 25, pp. 40-46.

Haff, R. P. & Toyofuku, N., 2008. X-ray detection of defects and contaminants in the food industry. *Sensing and Instrumentation for Food Quality and Safety,* 2(4), pp. 262-273.

Harrison, J., Ojeda, J. & Romero-Gonzales, M., 2012. The applicability of reflectance micro-Fourier-transform infrared spectroscopy for the detection of synthetic microplastics in marine sediments. *Science of the Total Environment,* Volume 416, pp. 455-463.

He, D. et al., 2018. Microplastics in soils: Analytical methods, pollution characteristics and ecological risks. *Trends in Analytical Chemistry,* Volume 109, pp. 163-172.

Herrera, A. et al., 2018. Novel methodology to isolate microplastics from vegetal-rich samples. *Marine Pollution Bulletin,* 129(1), pp. 61-69.

Ho, K. S., Billson, D. R. & Hutchins, D. A., 2007. Inspection of drinks cans using non-contact electromagnetic acoustic transducers. *Journal of Food Engineering,* Volume 80, pp. 431-444.

Hurley, R. R., Lusher, A. L., Olsen, M. & Nizzetto, L., 2018. Validation of a Method for Extracting Microplastics from Complex, Organic-Rich, Environmental Matrices. *Environmental Science & Technology,* 52(13), pp. 7409-7417.

Ikari, T., Takahashi, N., Fukasawa, R. & Duling, I., 2014. *Non-metallic foreign matter detection in powder using Terahertz pulse,* Otawara, Tochigi, Japan: Spectra Design.

Imhof, H. et al., 2012. A novel, highly efficient method for the separation and quantification of plastic particles in sediments of aquatic environments. *Limnology and Oceanography: Methods,* Volume 10, pp. 524-537.

Jain, S. et al., 2015. A comprehensive review on operating parameters and different pretreatment methodologies for anaerobic digestion of municipal solid waste. *Renewable and Sustainable Energy Reviews,* Volume 52, pp. 142-154.

Karami, A. et al., 2017. A high-performance protocol for extraction of microplastics in fish. *Science of the Total Environment,* Volume 578, pp. 485-494.

Khairi, M., Ibrahim, S., Yunus, M. & Faramarzi, M., 2018. Noninvasive techniques for detection of foreign bodies in food: A review. *Journal of Food Process Engineering,* 41(6), p. e12808.

LACSD, B., 2018. *DOC #4511874.* s.l.:s.n.

Lee, Y. et al., 2012. Detection of foreign bodies in foods using continuous wave terahertz imaging. *Journal of Food Protection,* Volume 75, pp. 179-183.

Li, F. et al., 2015. Confocal three-dimensional micro X-ray scatter imaging for nondestructive detecting foreign bodies with low density and low-Z materials in food products. *Food Control,* Volume 54, pp. 120-125.

Lim, J. W. et al., 2018. Effects of disposable plastics and wooden chopsticks on the anaerobic digestion of food waste. *Waste Management,* Volume 79, pp. 607-614.

Maes, T. et al., 2017. A rapid-screening approach to detect and quantify microplastics based on fluorescent tagging with Nile red. *Scientific Reports,* Volume 7, pp. 1-10.

Majewsky, M., Bitter, H., Eiche, E. & Horn, H., 2016. Determination ofmicroplastic polyethylene (PE) and polypropylene (PP) in environmental samples using thermal analysis (TGA-DSC). *Science of the Total Environment,* Volume 568, pp. 507-511.

Mecozzi, M., Pietroletti, M. & Monakhova, Y., 2016. FTIR spectroscopy supported by statistical techniques for the structural characterization of plastic debris in the marine environment: Application to monitoring studies. *Marine Pollution Bulletin,* Volume 106, pp. 155-161.

Melo, A. M., Gobbi, A. L., Piazzetta, M. H. O. & de Silva, A. A. M. P., 2012. Cross-shaped terahertz metal mesh filters: Historical review and results. *Advances in Optical Technologies,* Volume 2012.

Menczel, J., Prime, R. & Gallagher, P., 2009. Thermal Analysis of Polymers: Fundamentals and Applications. *John Wiley & Sons, Inc.*

Mertes, A., 2015. *Types of Plastic and their Recycle Codes.* [Online]
Available at: https://www.qualitylogoproducts.com/promo-university/different-types-of-plastic.htm

MOE, O., 2012. *Guideline for Composting Facilities and Compost Use in Ontario,* s.l.: s.n.

Mukherjee, S. & Gowen, A., 2015. A review of recent trends in polymer characterization using non-destructive vibrational spectroscopic modalities and chemical imaging. *Analytica Chimica Acta,* Volume 895, pp. 12-34.

Nature Communications, 2018. The future of plastic. *Nature Communications,* 9(1), p. 2157.

Nielsen, M. S., Lauridsen, T., Christensen, L. B. & Feidenhans'l, R., 2013. X-Ray dark field imaging for detection of foreign bodies in food. *Food Control,* Volume 30, pp. 531-535.

Nuelle, M., Dekiff, J., Remy, D. & Fried, E., 2014. A new analytical approach for monitoring microplastics in marine sediments. *Environmental Pollution,* Volume 184, pp. 161-169.

Ok, G., Kim, H. J., Chun, H. S. & Choi, S., 2014. Foreign-body detection in dry food using continuous sub-terahertz wave imaging. *Food Control,* Volume 42, pp. 284-289.

Ok, G., Shin, H. J., Lim, M. & Choi, S., 2019. Large-scan-area sub-terahertz imaging system for nondestructive food quality inspection. *Food Control,* Volume 96, pp. 383-389.

Omnexus, n.d. *Density of Plastics: Technical Properties.* [Online]
Available at: https://omnexus.specialchem.com/polymer-properties/properties/density
[Accessed 27 March 2019].

Pallav, P., Hutchins, D. A. & Gan, T. H., 2009. Air-coupled ultrasonic evaluation of food materials. *Ultrasonics,* Volume 49, pp. 244-253.

Piehl, S. et al., 2018. Identification and quantification of macro- and microplastics on an agricultural farmland. *Scientific Reports,* Volume 8, pp. 1-9.

Prata, J., Costa, J., Duarte, A. & Rocha-Santos, T., 2019. Methods for sampling and detection of microplastics in water and sediment: A critical review. *Trends in Analytical Chemistry,* Volume 110, pp. 150-159.

Qin, J. et al., 2013. Hyperspectral and multispectral imaging for evaluating food safety and quality. *Journal of Food Engineering,* Volume 118, pp. 157-171.

Qiu, Q. et al., 2016. Extraction, enumeration and identification methods for monitoring microplastics in the environment. *Estuarine, Coastal and Shelf Science,* Volume 176, pp. 102-109.

Scheurer, M. & Bigalke, M., 2018. Microplastics in Swiss Floodplain Soils. *Environmental Science & Technology,* Volume 52, pp. 3591-3598.

Schindler, A. et al., 2017. Identification of polymers by means of DSC, TG, STA and computer-assisted database search. *Journal of Thermal Analysis and Calorimetry,* 129(2), pp. 833-842.

Serranti, S. et al., 2015. An innovative recycling process to obtain pure polyethylene and polypropylene from household waste. *Waste Management,* Volume 35, pp. 12-20.

Shan, J. et al., 2019. Simple and rapid detection of microplastics in seawater using hyperspectral imaging technology. *Analytica Chimica Acta,* Volume 1050, pp. 161-168.

Shim, W., Song, Y., Hong, S. & Jang, M., 2016. Identification and quantification of microplastics using Nile red staining. *Marine Pollution Bulletin,* Volume 113, pp. 469-476.

Silva, A. et al., 2018. Microplastics in the environment: Challenges in analytical chemistry - A review. *Analytica Chimica Acta,* Volume 1017, pp. 1-19.

Simon, M., Alst, N. & Vollertsen, J., 2018. Quantification of microplastic mass and removal rates at wastewater treatment plants applying Focal Plane Array (FPA)-based Fourier Transform Infrared (FT-IR) imaging. *Water Research,* Volume 142, pp. 1-9.

Sun, J. et al., 2019. Microplastics in wastewater treatment plants: Detection, occurrence and removal. *Water Research,* Volume 152, pp. 21-37.

Tagg, A. et al., 2017. Fenton’s reagent for the rapid and efficient isolation of microplastics from wastewater. *Chemical Communications,* Volume 53, pp. 372-375.

US Composting Council, 2001a. *TMECC 03.05 Film Plastics.* s.l.:s.n.

US Composting Council, 2001b. *TMECC 03.06 Glass, Shards, Metal Fragments and Hard Plastics.* s.l.:s.n.

Van Drimmelen, B., 2013. *Organic Waste Composition Audit - Project No. C83.208,* Surrey, British Columbia: TRI Environmental Consulting Inc.

Wang, K., Sun, D. & Pu, H., 2017. Emerging non-destructive terahertz spectroscopic imaging technique: Principle and applications in the agri-food industry. *Trends in Food Science & Technology,* Volume 67, pp. 93-105.

Wang, Z. et al., 2018. Visulizing 3D food microstructure using tomographic methods: Advantages and disadvantages. *Annual Reviews,* Volume 9, pp. 323-343.

Zhang, S. et al., 2019. Microplastics in the environment: A review of analytical methods, distribution, and biological effects. *Trends in Analytical Chemistry,* Volume 111, pp. 62-72.

Zhao, B., Basir, O. A. & Mittal, G. S., 2003. Detection of metal, glass and plastic pieces in bottled beverages using ultrasound. *Food Research International,* Volume 36, pp. 513-521.

Zheng, Y. et al., 2018. A discrimination model in waste plastics sorting using NIR hyperspectral imaging system. *Waste Management,* Volume 72, pp. 87-98.

Appendix A – Rinsing/Sieving Experiment Details

Procedure

Equipment

- 1 kg of sample
- Sieves: 6.73 mm, 4.0 mm, 2.36 mm, 600 µm (sieve sizes can be adjusted to suit various applications if, for example, collection of smaller particles is desired)
- Deep sink
- Oven
- Tweezers
- Wash bottle
- Tray

PPE

- Nitrile gloves
- Laboratory coat
- Recommended: face mask
- Recommended: fume hood or exhaust snorkel fume hood

Method

1. Stack the sieves from the smallest openings on the bottom to the largest openings on the top.
2. With the sieve stack in sink, add a small portion of the sample to the top.
3. Rinse until all or most of the sludge and small particles are gone from the top sieve. To avoid flooding, it is best to keep rinsing concentrated in one spot and move this spot as needed, as opposed to attempting to broadly rinse the whole sieve at once.
4. Repeat steps 2-3 until all of the sample has been used. During this process, regularly check the 600 µm and 2.36 mm sieves to make sure they are not clogged. If one is beginning to clog, remove the clogged sieve from the stack and rinse it separately until it drains, then put it back into its place in the stack and continue rinsing the sample.
5. Once the top sieve has been fully rinsed, remove it from the stack and ensure that the next sieve down has been fully rinsed. Repeat until all sieves have been fully rinsed.
6. Sieves/samples can now be prepared for imaging or H_2O_2 digestion, if desired.
7. If manually collecting plastics, use tweezers to remove visible plastics from the 6.73 mm and 4.0 mm sieves. (Plastics cannot be accurately detected by naked eye on the smaller sieves.)
8. When all visible and easily accessible plastics have been removed from the sieves, invert sieves onto a tray and use a wash bottle to get the particles off the sieve and into the tray.

9. Use tweezers to remove visible plastics from the tray. If desired, dry excess water in an oven at 50 °C for approx. 8 hours beforehand.
10. If desired, clean plastics and weigh on an analytical balance.

Original Observations

The following are the original observations that were taken during the experiments described in section 4. “Pre magnet” is Sample A1, “SPPS” is Sample B1, and “PPS” is Sample C1.

General observations

- Samples are odourous – face masks help and fume hood/snorkel helps tremendously
- Samples are in the process of being digested and cannot be left for long periods of time (more than two days) even in the fridge – the gases produced in the digestion process will pressurize the bottles
- The further away from the pit the samples are taken, the more dilute the samples become; therefore, a total suspended solids count should be taken to normalize the samples relative to each other

Raw feeds

- PPS: viscous liquid (not paste) with suspended solids
- SPPS: viscous liquid, somewhat pasty
- Pre magnet: paste

Methods

Drying samples directly at 50 °C for three days

- Ineffective for all samples
- Samples “bake” even at low temperature, with the result that it is impossible to find the plastics within the “cake”
- Samples become crispy and hard to work with

Shaker 15 minutes with sieve setup: 4.0 mm, 3.35 mm, 2.36 mm, 1.0 mm, 600 µm

- Somewhat effective for liquidy PPS sample
    - Viscous liquid formed film over sieve holes, resulting in mild clogging and only the thinnest liquid falling to the bottom
    - Shaker produced froth which contributed to clogging in smaller sieves
    - Sieves needed to be rinsed – shaker alone was not enough to separate solids from viscous liquid
- Ineffective for pasty pre magnet sample
    - Very little sample penetrated past 4.0 mm sieve
    - Extensive rinsing required

Rinsing setup: 6.73 mm, 4.0 mm, 1.0 mm

- 1.0 mm sieve gets clogged very quickly and should be individually rinsed regularly to avoid flooding and loss of sample
- Particles between 4.0 mm and 1.0 mm will be caught on 1.0 mm sieve but 1.0 mm sieve gets very full – better off with 2.36 mm sieve as the last sieve for plastic collection
- Sample should be added in small amounts and each sieve should be individually rinsed between sample additions to avoid clogging

Rinsing setup: 6.73 mm, 4.0 mm, 2.36 mm, (600 µm)

- 2.36 mm optimal size for particle collection – however, 600 µm sieve should be added to collect particles smaller than 2.36 mm for disposal, to avoid clogging drains
- 600 µm sieve gets clogged very quickly and should be individually rinsed regularly to avoid flooding and loss of sample

Removing solids from sieves: flip sieve over and rinse solids into tray using wash bottle

- Effective, although some solids (fibers, films) remain wrapped around sieve wires and must be manually extracted using tweezers
- Best case scenario sieves can be imaged directly without needing to transfer solids to a secondary container
- Rinsing results in tray being wet – samples should be oven-dried or filtered

Drying setup: 50 °C for ~16 hours (used for pre magnet, SPPS)

- 16 hours is probably too long – all water evaporated but samples were crispy and baked, making them hard to work with
- Advantage: plastic films are very easy to find because they are the only things that aren't crispy and dehydrated – they remain flexible and stretchy

Filtering setup: funnel with filter paper (used for PPS)

- 20-25 µm filter paper (Whatman #41) is slow but usable
- 11 µm filter paper (Whatman #1) is unacceptably slow

Extracting plastics using tweezers

- Time consuming, tiring, subject to human error
- Films are relatively easy to find in dried samples because they retain flexibility while organic materials dry out
- Films also have a characteristic appearance that is very different from most organics; however, films may resemble onions and other fibrous vegetables
- Films may be flat and wide (large surface area) or rolled up and long (small surface area) – flat and wide plastics are easier to recognize
- Hard plastics are more difficult to find because they don't necessarily look unique – they may look identical to rock or bone or shell or glass fragments
- Hard plastics also are destroyed in the grinder more efficiently than films, so they have a smaller surface area and are therefore more difficult to detect using the naked eye

Appendix B – Commercial Vendors

The following are a list of commercial vendors selling HSI, X-ray, and ultrasound equipment that could be used for future testing in this area. Vendors that have been successfully contacted have additional information listed.

Company name	Website	Location	Technology	Suggested applications	Additional information
Allied Vision Technologies GmbH	https://www.alliedvision.com/	USA: Exton, PA	HSI	Industrial inspection, medical imaging, traffic, remote sensing, food sorting, pharmacy, mining	
Condi Food B.V.	https://condifood.com/	The Netherlands: Warmond	HSI		
Cubert GmbH	https://cubert-gmbh.com/	USA: Newton, MA (distributor: Bodkin Design)	HSI	Remote sensing, agriculture, skin analysis, medical screening, sorting	
Delta Optical Thin Film A/S	https://www.deltaopticalthinfilm.com/	USA: San Jose, CA (distributor: Silvaco)	HSI	Agriculture, satellite imaging, medical screening, food inspection	
DV s.r.l.	http://www.dvoptic.com/	Italy: Padova	HSI	Remote sensing, sorting, food inspection, forensics, agriculture, medical	

Llewellyn Data Processing LLC	https://maxmax.com/	USA: Carlstadt, NJ	HSI	Art, remote sensing, forensics, surveillance, skin analysis	
Norsk Elektro Optikk AS	https://www.hyspex.no/	Norway: Skedsmokorset	HSI	GPS, materials characterization	
P&P Optica, Inc.	https://ppo.ca/	Canada: Waterloo, ON	HSI	Food inspection	
Perception Park GmbH	https://www.perception-park.com/	Germany: Berlin	HSI		• Software only
PSI Technics GmbH	https://www.psi-technics.com/EN/index.php	Germany: Urmitz	HSI	Materials classification, food inspection, automotive, pharmacy, sorting, mining	
SOLPI, S.L.	http://solpi.es/	Spain: Barcelona	HSI		
Specim, Spectral Imaging Ltd.	http://www.specim.fi/	Canada: Pinawa, MB (distributor: Channel Systems)	HSI	Food inspection, forensics, sorting	
Telops, Inc.	https://www.telops.com/	Canada: Quebec City, QC	HSI	Surveillance, NDT, combustion analysis, geoscience, ballistics, manufacturing	
Thorlabs, Inc.	https://www.thorlabs.com/	USA: Sterling, VA	HSI		• CM401 Cerna HSI system - $51,500

Eigenvector Research	http://www.eigenvector.com/	USA: Manson, WA	HSI, NMR	Industrial inspection, pharmacy, semiconductors, materials characterization	
Headwall Photonics, Inc.	http://www.headwallphotonics.com/	USA: Boston, MA	HSI, Raman	Recycling sorting, remote sensing, food inspection, agriculture, medical screening, surveillance, satellite imaging	• Cost of hyperspectral system: $50,000 - $200,000 (incl. lighting) • Will analyze samples with 2-3 week turnaround - $2000/day, or free for simple tests
inno-spec GmbH	https://inno-spec.de/en/	Germany: Nuremburg	HSI, Raman		
BaySpec, Inc.	https://www.bayspec.com/	USA: San Jose, CA	HSI, Raman, MS	Biology, materials characterization, pharmacy, forensics, remote sensing, agriculture, geoscience	• Will analyze 8 samples for $2500-$3000 with 2 weeks' notice • Hyperspectral system can scan on a conveyor (100 fps)
LLA Instruments GmbH & Co. KG	https://www.lla-instruments.com/	Germany: Berlin	HSI, X-ray	Sorting, mining, agriculture, food inspection, medical screening, pharmacy	

Teledyne DALSA	https://www.teledynedalsa.com/en/home/	Canada: Waterloo, ON	HSI, X-ray	NDT, dentistry, medical screening, aerospace, traffic, surveillance	
XIMEA corp.	https://www.ximea.com/	USA: Lakewood, CO	HSI, X-ray		
Teledyne Princeton Instruments	https://www.princetoninstruments.com/	USA: Trenton, NJ	HSI, X-ray, Raman		
Polytec GmbH	https://www.polytec.com/us/	Germany: Waldbronn	Raman		
Spectra Research Corp.	https://www.spectraresearch.com/	Canada: Mississauga, ON	Raman	Pharmacy, pulp & paper, mining, food, semiconductors	
Eagle Product Inspection LLC	https://www.eaglepi.com/	USA: Lutz, FL	X-ray	Food inspection, pharmacy, manufacturing	
Mettler Toledo, Inc.	https://www.mt.com/ca/en/home.html	Canada: Mississauga, ON	X-ray	Food inspection, pharmacy, manufacturing	
Teknologisk Institut	https://www.dti.dk/	Denmark: Taastrup	X-ray		

Tomra Systems ASA	https://www.tomra.com/en	USA: West Sacramento, CA	X-ray	Sorting	• Work with organic waste • Wet sorting in Europe • Combine techniques such as NIR spectroscopy, LIBS, LED spectrometry, X-ray, … to achieve best results • R&D based in Frankfurt - samples would be sent there
Sesotec GmbH	https://www.sesotec.com/na/en-US	Canada: Cambridge ON	X-ray, HSI	Food inspection, plastics processing, sorting	• Might to sample analysis free of charge in Germany