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Quantification of microplastics in biowastes including biosolids, compost, and vermicompost destined for land application

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Abstract

The land application of biowastes, including biosolids and compost, is a significant source of microplastics (MP) to terrestrial environments, yet global data on contamination levels are limited. We determined the concentrations of microplastics in biowastes destined for land application in Aotearoa New Zealand. Microplastics were extracted from biosolids, vermicompost, bulk compost, and bagged compost via wet peroxide oxidation digestion and density separations using a modified sediment-microplastics isolation (SMI) unit. The polymer type of each suspected microplastic was confirmed by micro-Fourier-transform infrared (μ-FTIR) spectroscopy, with a minimum detection size of 18 μm. Microplastic concentrations > 0.48 MP/g were identified in every sample, with the highest average abundances in biosolids (2.71 MP/g) and vermicompost (2.69 MP/g), followed by bulk compost (1.94 MP/g) and bagged compost (1.1 MP/g). Fragments (62.7%) were the most frequently detected microplastic morphotype, followed by films (24.7%), fibers (12.2%), and beads (0.4%). Common polymers detected were polypropylene (37.9%), polyethylene (28.6%), and polymethyl methacrylate (PMMA) (11.7%). Identifiable morphotypes included polyurethane foam sponge fragments, polyethylene terephthalate glitter, and PMMA multicolored films. Biodegradable polymers were identified, and their presence in mature compost suggests that compost facilities were unable to provide optimal conditions to support the complete biodegradation of polymers. Annual microplastic contamination in soils from the application of biowaste amendments is projected to be between

1.10 × 10^7 to 2.71 × 10^7 MP/ha. The product origin of most microplastics could not be identified, highlighting the ubiquity of microplastics and the urgent need to reduce plastic at the source to reduce instances of pollution in valuable biowastes.

Keywords: Microplastics, biosolids, compost, vermicompost, biowaste, μ-FTIR

INTRODUCTION

Since the 1950s, the widespread production and use of plastic has led to a global crisis of plastic pollution contaminating all areas of Earth. Microplastics (MP, particles < 5 mm) are smaller plastic particles produced purposefully for a particular function (i.e., microbeads, glitter, resin pellets), or those that arise from the fragmentation of larger plastic items during manufacturing, use, or environmental weathering[1]. The continued fragmentation of plastics in the environment concerns regulators due to microplastics persistence, chemical and physical toxicity, and ubiquity across environmental matrices and biological systems[2].

While attention has been focused on the marine environment, there is a lack of knowledge concerning microplastics in the terrestrial environment, with only 7% of microplastics-related literature from 2006-2021 dedicated to the terrestrial environment[3]. Quantification studies are limited, in part due to the challenges associated with separating microplastics from solid matrices for polymer confirmation analysis, particularly organic-rich samples such as biosolids and composts[4].

Wastewater treatment plants (WWTPs) are a significant pathway for microplastics to enter the terrestrial environment by applying sewage sludge/biosolids and wastewaters to land as a source of irrigation water and nutrients[5,6]. Plastic particles and fibers released from products used in domestic, commercial, and industrial applications are subsequently discharged into sewerage networks and, ultimately, WWTPs[7,8]. The abundance of microplastics can decrease from the incoming influent to the final effluent by > 99%[9,10], but a significant number of microplastics are released into the receiving environment daily via the final effluent[11]. The decrease in microplastic abundance in the aqueous phase during wastewater treatment can be attributed to their retention in the solid phase, with concentrations of microplastics ranging from 2.2 to 1,353 MP/g (dry weight) previously reported for sludge and biosolids [Supplementary Table 1][5,12].

Compost, a significant municipal biowaste, can be a major source of microplastics in terrestrial environments[13,14]; however, limited research has been conducted [Supplementary Table 2]. Compost is produced from a varied range of feedstocks including food waste, green waste (garden and arbor waste), and livestock manures. Feedstocks may also include commercial and industrial animal and food processing wastes and generally exclude human excrement/biosolids, but this depends on the intended land use. An alternative processing method of composting biowastes, including biosolids, involves composting with earthworms (known as vermicomposting), which effectively removes microbial pathogens[15]. The reported abundance of microplastics in composts ranges from 0.012 to 62 MP/g [Supplementary Table 2][16,17]. Sources of microplastics to compost have not been well characterized. The introduction of larger plastics to organic waste may occur due to a lack of education surrounding the separation of wastes and instances of misleading packaging information or "greenwashing" which falsely suggests that the product is compostable[18]. To date, microplastics of biodegradable polymers have not been previously identified in composts or biosolids.

The reuse of biowastes provides a beneficial source of carbon and nutrients to the soil, as well as supporting the circular economy by diverting these wastes from landfills, which in turn reduces greenhouse gas emissions and the reliance on chemical fertilizers[19-21]. Although benefits of soil application of biowastes include improved plant growth and soil quality[22-25], the presence of microplastics in soil can cause deleterious and toxic effects to plants and soil organisms[26-29], as well as changes in microorganism community structure[30,31].

As no standard methods exist for the extraction and analysis of microplastics, a comprehensive comparison of the microplastic content between multiple biowaste types using the same extraction method is absent in the literature. There is also a lack of published data for microplastics in vermicompost, with only one previous study reporting the abundance of microplastics in sewage sludge increased after vermicomposting in a laboratory-based setting[32]. To address this global knowledge gap, this study is the first to analyze solid biowaste samples of biosolids, vermicompost, bulk compost, and bagged compost from five facilities/brands of each sample type around Aotearoa New Zealand to characterize the microplastic content and to estimate the microplastic loading to land from the application of biowaste amendments based on nitrogen requirements.

MATERIALS AND METHODS

Sampling facilities/brands

The final mature product of biowaste samples was collected between June 2021 and September 2022. Biosolids, vermicompost, and bulk compost were collected from five processing facilities per biowaste type (a total of 15 facilities) across Aotearoa New Zealand by a staff member from each facility. Each facility services varying population sizes, feedstocks, and volumes of incoming waste, as determined by a questionnaire (approved by the University of Canterbury Human Ethics Committee, reference number HCE 2021/17/LR-PS). For reasons of confidentiality, limited information about each facility is presented in the Supplementary Materials [Supplementary Section 1]. Bulk compost facilities all processed some degree of food waste. Some vermicompost facilities accepted municipal biosolids from WWTPs as a feedstock. Five brands of bagged composts were purchased in triplicate from local garden centers (a total of 15 bags).

Sample collection

A detailed sampling procedure, along with health and safety measures, are presented in the Supplementary Materials [Supplementary Section 2]. Sample collection kits were sent to each facility, with the contents presented in Supplementary Section 2. Briefly, triplicate samples of each biowaste at each facility/brand were collected by scooping the product with a stainless-steel spoon into individual 1L glass jars, filling to the top of the jar. Biosolids were collected by filling each jar with samples from different sections of the same pile, as generally, only a single pile of biosolids was available. Bulk compost and vermicompost samples were collected by filling each jar with a sample from different sections of the same windrow or pile. Bagged compost samples were collected by filling each jar with a sample from each bag. Once collected, sample jar lids were screwed on tight and the samples were shipped back to the University of Canterbury, where upon arrival, they were stored at 4 °C.

Chemicals and materials

High-performance liquid chromatography (HPLC) grade ethanol and acetone, iron (II) sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$), sulfuric acid, and Decon 90 detergent were purchased from Thermofisher Scientific Inc. (Massachusetts, United States). Hydrogen peroxide (H_2O_2, 30%) was purchased from Jasol New Zealand Ltd. (Auckland, New Zealand). Sodium iodide (NaI) was purchased from Livestock Supplies New Zealand (Gore, New Zealand). Ultra-pure water (ASTM type I water, > 18.2 MΩ) was sourced from a Sartorius Arium® pro filtration system fitted with a 0.45 μm filter (Sartorius AG, Göttingen, Germany). Whatman

grade GF/C glass microfiber filters (1.2 µm pore, 47 mm diameter) were purchased from Merck KGaA (Darmstadt, Germany). Calcium fluoride (CaF_2) windows (13 mm diameter, 1 mm thickness) were purchased from Crystran Ltd. (Poole, United Kingdom). Plastic reference fragments used in the spiked recoveries were 500-1000 µm in length and were purchased from Clariant [polypropylene (PP), high-density polyethylene (HDPE), high impact polystyrene (HIPS), polyamide (PA), polyethylene terephthalate (PET)], Chi Mei Corp. (acrylonitrile-butadiene-styrene, ABS), and Marley (polyvinyl chloride, PVC). Polyethylene (PE) microbeads (between 100-500 µm) were sourced from a facial cleanser; polymethyl methacrylate (PMMA) fibers (approximately 1 mm in length) were created by cutting from a ball of yarn purchased from a craft store. The polymer type of all plastic reference fragments was confirmed by FTIR spectroscopy (Bruker Alpha II).

Extraction method

The extraction of microplastics from biowastes followed the procedure described by Ruffell *et al.* (2024)[4] and is briefly outlined below. Biowaste samples were dried in an oven at 75 °C for four days to reduce the risk of exposure to *Legionella* species [Supplementary Section 2]. No spectral changes were observed in reference plastics before and after heating [Supplementary Section 3]. Bulk and bagged composts were sieved < 2 mm using a stainless-steel sieve. Biosolid and vermicompost samples were not sieved due to the presence of aggregates in which microplastics may have been encased. Samples were extracted separately in triplicate, where 30 g of dried sample in glass beakers were digested via wet peroxide oxidation (WPO) with Fenton's reagent[33]. Approximately 50 mL each of ultra-pure water, 0.05 M iron (II) sulfate [Fe(II)] solution, and 30% H_2O_2 were added to the sample, and the resulting mixture was left at room temperature for 4-6 h, with the temperature monitored to ensure it remained below 45 °C. The samples were then placed on a hotplate at 35 °C and left overnight. The following morning, 20 mL of H_2O_2 was added, and the temperature increased to 40 °C. Further additions of H_2O_2 were added twice daily until the digest was deemed complete, as indicated when all digested organic material had settled, the solution was opaque and not turbid, and no bubbles evolved after gently moving the solution beaker[4]. The duration of the digestions ranged from 2 days to 14 days, depending on the sample. Additional care was taken with compost samples, which often tended to be more reactive, by lowering the initial volume of H_2O_2 added. The digested samples were transferred to a modified sediment-microplastics isolation (SMI) unit[34], with modifications as described by Ruffell *et al.* (2024)[4]. The SMI unit was topped up with ultra-pure water and stirred with a glass rod to release any trapped microplastics, covered with aluminum foil and left to settle overnight with the valve open. The valve was closed the following morning, and the supernatant was filtered onto a GF/C filter under vacuum, rinsing out the top section thoroughly. The filter was dried under vacuum and stored in a covered petri dish until analysis. A density separation with NaI was then undertaken by opening the valve and adding a corresponding volume (to the bottom half of the SMI unit, approximately 340 mL) of 2.6 g cm^{-3} NaI. The digest solution was stirred and the NaI was diluted by approximately half upon mixing to a density of ~1.8 g cm^{-3}. The SMI unit was covered with aluminum foil and left to settle out overnight, and collection of the supernatant followed the same process as the first density separation, with the supernatant filtered onto a new GF/C membrane.

Microplastic identification and analysis

All filters were first visually analyzed under a stereomicroscope (Nikon SMZ1270, magnification 12.7x) to identify suspected microplastics, following the criteria from Rochman *et al.* (2019)[35] as a guide [Supplementary Section 4]. All suspected microplastics > 18 µm in the longest dimension were transferred with tweezers onto a new GF/C filter inside a glass petri dish (with a glass lid) and photographed, and the longest dimension recorded. In general, more microplastics were present in the NaI density separation. Photographs of example microplastics are presented in the supplementary information [Supplementary Section 4]. Suspected microplastics were then transferred onto a CaF_2 disc (13 mm

diameter, 1 mm thickness) with tweezers, aided by a drop of HPLC-grade ethanol, which was left to evaporate. Particles were spread out on the disc to avoid overlapping or clumping. Larger films and fibers prone to blowing away were held in place on the disc with the aid of adhesive putty on the particle tip, ensuring no interference of the sticky tack with the IR beam. All suspected particles identified under stereomicroscope (100%) were analyzed by micro-Fourier-transform infrared (μ-FTIR) [Bruker Hyperion 2000 (coupled to a Vertex 70 spectrometer), equipped with a mercury cadmium telluride (MCT) detector and potassium bromide (KBr) beamsplitter, OPUS software version 7.8]. All particles were scanned 32 times each in transmission mode with a 15x infrared objective, with a resolution of 4 cm^{-1}, within the spectral range of 4000-1000 cm^{-1}. Background measurements were taken against the CaF_2 disc. Spectra obtained were matched against several commercial polymer reference libraries [Supplementary Section 5] within the Wiley KnowItAll Informatics System spectral library database (Version 20.1.210.0). Polymer assignment criteria included a match greater than 70% with the spectral library, and a thorough assessment of the characteristic peaks of each spectrum to confirm the polymer type.

Quality control/quality assurance

As microplastics are a common contaminant, appropriate quality control/quality assurance measures were incorporated into all aspects of equipment and glassware cleaning, field sampling, method development, sample extraction and analysis to minimize contamination by ambient microplastics. Equipment used in this project was made of glass or metal where possible to avoid contamination by plastic products. All equipment and glassware were thoroughly pre-cleaned with detergent (Decon 90) and were rinsed three times with ultra-pure water and once with solvent-grade acetone to remove any loose plastic fragments. Stainless steel sieves were cleaned in between samples with Decon 90 and were rinsed three times with ultra-pure water and once with solvent-grade acetone. Equipment and glassware openings were sealed with a lid or covered tightly with aluminum foil to prevent contamination by airborne microplastics. During field sampling, the sample collector avoided wearing synthetic shedding clothing, while maintaining appropriate personal protective equipment (PPE) requirements for their facility. During sample processing, cotton laboratory coats were always worn, with clothing underneath chosen to be non-shedding and of natural fibers. All sample processing and extractions occurred in an aluminum foil-lined fume hood. Workspaces and fume hoods were sprayed with 70% ethanol and wiped with a paper towel prior to starting and after completing laboratory work each day.

Appropriate field and laboratory controls were implemented as follows. A pre-cleaned sealed jar was opened during field sampling at each facility to account for ambient microplastics. Once collected, samples were covered with a lid or aluminum foil to reduce exposure to airborne microplastics. Laboratory controls included an empty pre-cleaned beaker with no sample, which was processed using the same method as each sample in the laboratory. Field controls were processed by rinsing the jar out thoroughly with ultra-pure water and vacuum-filtering onto a GF/C membrane. A microscope slide with exposed double-sided tape was placed at the back of the fume hood and workspaces when processing samples to account for ambient microplastic particles. Laboratory, field, and fume hood controls were analyzed by picking off each suspected microplastic under stereomicroscope and analyzing each particle by μ-FTIR following the same method used for the biowaste samples.

All reagent solutions were pre-filtered through a GF/C filter before use, with the exception of NaI (2.6 g cm^{-3}) due to clogging of the GF/C filter. However, 1 L of 1.8 g cm^{-3} NaI from each batch was filtered through a GF/C and checked for contamination under stereomicroscope, and no suspected particles were detected. The use of a method blank also accounted for any possible contamination introduction from any solutions used. This work was undertaken by a single analyst to ensure consistency and reduce bias in results.

Spectral tests of reference fragments (PET, PVC, HIPS, PP, ABS, HDPE, PA) before and after digestion were undertaken with a Bruker Alpha II attenuated total reflectance-Fourier-transform infrared (ATR-FTIR) [Supplementary Section 6]. Spiked recoveries of this method are presented in detail in the Supplementary Materials [Supplementary Section 7], but briefly, spiked recoveries were performed in triplicate for samples of biosolids, compost, and soil. Each sample was spiked with: 1x each reference fragment of PET, PVC, HIPS, PP, ABS, HDPE, and PA, 10x PE microbeads, and 10x PMMA fibers.

Data analysis

All (100%) suspected microplastic particles > 18 μm in the longest dimension were analyzed by μ-FTIR. Only confirmed plastic polymers were included in the results. Spectra that were of a non-plastic or natural origin, unable to be determined, and of a match lower than 70% were excluded. Example spectra of common polymer types are presented in the Supplementary Materials [Supplementary Section 8]. As each replicate underwent two density separations (ultra-pure water and NaI), each separation was filtered onto a new GF/C filter membrane. These filters were analyzed separately, and the results of each filter were combined for each sample replicate. Results are reported as the average of the three replicates for each facility and were standardized to microplastic particles per gram (MP/g, dry weight) by dividing the microplastic number per sample by the mass of the sample (30 g dry weight). The results are reported as mean ± standard error.

Estimation of soil microplastic contamination from amendment application

Estimates of microplastic contamination to soil are based on the following assumptions of 2% nitrogen (N) concentration in biosolids and compost[21,36], and a maximum annual application of N as 200 kg of N per hectare (ha), at an annual application rate of 10 tonnes of amendment per ha[21,36]. Soil loading estimates were based on a soil depth of 0.15 m and soil density of 1.3 kg/m^3. The average abundance of microplastics per sample type (dry weight) was used for calculations. The equations for the estimation of microplastic contamination to soil from amendment application are presented in the Supplementary Materials [Supplementary Section 9].

RESULTS AND DISCUSSION

Quality control

Average spiked recoveries were 92%, 95% and 98% for bagged compost, biosolids, and soil, respectively [Supplementary Section 7]. This method was highly effective at recovering a range of polymer types, with all reference fragments recovered in all sample types, which included higher-density polymers such as PVC and PET. Previous recovery trials of microbeads and fibers yielded lower recoveries (50%-90%). This variation was attributed to the beads and fibers being blown off the filter paper by air flow in the fume hood during the transfer of the filter from the filtering apparatus to the petri dish for storage. To minimize this effect, the filter transfer occurred on the laboratory bench instead of within the fume hood. This increased the recovery of microbeads and fibers to rates ranging from 80% to 100%. Possible contamination was accounted for by transferring the fume hood control (double-sided tape) to the laboratory bench during this process. Additionally, residual organic material consisting of undigested plant material (leaves, seeds, sticks) obstructed the identification of fibers under stereomicroscope. The effect of this was minimized by carefully inspecting residual organic material on the filters with tweezers under stereomicroscope to identify any potentially buried or hidden microplastics. Municipal biowastes are complex matrices from which to extract microplastics, and further optimization of extraction methods is required to increase recovery rates and reduce non-target organic material. All suspected microplastics in the laboratory and field controls were analyzed by μ-FTIR. Confirmed plastic particles in the controls were found at a fifth of the facilities, with a total of 8 particles classed as contamination. Microplastics in the controls were of the same polymer type, morphotype, and color as the microplastics present in the samples. All contaminant particles were from the

field controls collected during the sampling of bagged compost and biosolids. Sample results were corrected by subtracting the contaminant microplastic from the total microplastic abundance, morphotype, polymer type, and color for each facility.

Microplastics abundance

All suspected microplastics (n = 4419) were analyzed by μ-FTIR and a total of 3,798 particles were confirmed to be synthetic plastic polymers (equivalent to 86%). Based on these results, polymer confirmation is recommended, as visual analysis alone may result in overestimation. However, residual organic matter, paint coatings, and weathering on the surface of microplastics may also hinder μ-FTIR transmission, leading to inconclusive spectra and, thus, an underestimation of plastics detected. Microplastics > 0.48 MP/g were detected in all samples. The average abundance of microplastics at each sample type and facility/brand is presented in the Supplementary Materials [Supplementary Figures 1 and 2, Supplementary Table 3]. Microplastics were typically highest in biosolids (2.71 ± 0.08 MP/g, range of 0.90 ± 0.06 to 4.94 ± 0.33 MP/g) and vermicompost (2.69 ± 1.12 MP/g, range of 0.52 ± 0.05 to 6.92 ± 1.70 MP/g), followed by bulk compost (1.94 ± 0.33 MP/g, range of 1.06 ± 0.04 to 2.99 ± 0.28 MP/g) and bagged compost (1.1 ± 0.06 MP/g, range of 0.48 ± 0.10 to 2.61 ± 0.29 MP/g).

Microplastic abundance within biosolids in this study is within the lower range of those previously reported [Supplementary Table 1][5,37,38]. Generally, the abundance of microplastics in biosolids was under 100 MP/g; however, a study from Canada found microplastics at much higher abundances, with a median abundance of 636 MP/g, ranging from 228-1,353 MP/g[12]. In this study, the biosolids from larger WWTPs (facilities 2, 4, 5, which service populations of >100,000 people) had higher average abundances of microplastics from 2.81 ± 0.24 to 4.94 ± 0.33 MP/g, compared to the lower abundances detected in facilities 1 and 3, which service populations of < 10,000 people (1.73 ± 0.18 and 0.90 ± 0.06 MP/g, respectively). Further work is needed to determine if there is a correlation between the abundance of microplastics in biosolids and population.

This study is the first to quantify microplastics in vermicompost produced from a diverse range of feedstocks, including food scraps, green waste, biosolids, and industry processing wastes. The highest average abundance of microplastics in vermicompost was from facility 4, with 6.92 ± 1.70 MP/g, and this was also the facility with the highest average abundance of microplastics across all sample types in this study. The other four vermicompost facilities ranged in abundance from 0.52 ± 0.05 to 2.43 ± 0.29 MP/g. The vermicompost in this study are from commercial facilities that accept different feedstocks; however, there is some degree of overlap. The particle number and size of microplastics in vermicompost may be influenced by earthworms fragmenting macro- and microplastics into smaller micro- and nanoplastics during ingestion and excretion. Two laboratory studies have shown that fragmented microplastics present in the gut and feces of earthworms were smaller than the particles initially introduced in the trial[39,40]. Further, suspected depolymerization of polylactic acid (PLA) and polybutylene adipate terephthalate (PBAT) microplastics was observed in the gut of earthworms during a laboratory-based study, but not in the test soil[39].

The abundance of microplastic in composts in this study is within the lower range of previous studies, which range from 0.012 to 62 MP/g (dry weight) [Supplementary Table 2][41-43]. It is likely that the number of microplastics present in compost depends on the feedstocks. The feedstocks in this study varied [Supplementary Section 1] and the contribution of microplastics from each feedstock is unknown. Bulk compost samples from facilities 1 and 3 contained similar concentrations of microplastics (1.64 ± 0.08 MP/g and 1.77 ± 0.09 MP/g, respectively) and were collected from large centralized commercial facilities

compared to facilities 2, 4, and 5 that varied in the abundance of microplastics (1.06 ± 0.04, 2.27 ± 0.08, and 2.99 ± 0.28 MP/g, respectively) which may be due to their decentralized nature. It is otherwise unknown if there are any differences in microplastic abundance between centralized and decentralized composting facilities. Of the bagged composts in this study, brand 3 contained the highest average abundance of microplastics (2.61 ± 0.29 MP/g) compared to the other bagged composts (range from 0.48 ± 0.10 to 1.14 ± 0.16 MP/g). This brand is made solely from green waste from municipal drop-off points, which may result in more plastic contamination compared to the other brands that contain varying types of livestock manure and bark mulch as feedstocks. There is a lack of knowledge surrounding microplastic contamination of single feedstocks in the literature. Similar to this study, compost produced from food waste was higher in microplastic abundance than that made solely of green waste in a previous study[17]. In addition, the composting process can fragment microplastics, increasing particle number and subsequently decreasing particle sizes, making it difficult to identify the sources of microplastics in composts[44].

Morphotype of microplastics

Microplastics were categorized into their morphotypes, namely fragments, fibers, films, and beads [Figure 1]. The average morphotype proportion by sample type is presented in Figure 2, and the average morphotype proportion between facilities in the Supplementary Materials [Supplementary Figure 3, Supplementary Table 3]. On average, fragments were the most frequently detected morphotype (62.7%), followed by films (24.7%), fibers (12.2%), and beads (0.4%). This was consistent across sample types, except for biosolids, where fibers were the second-most abundant (22.1%) on average, followed by films (16.4%). Fragments were present in a similar proportion, with around 60% majority in biosolids, vermicompost, and bulk compost, compared to a higher proportion in bagged composts of just over 70%. Fragments were the most abundant morphotype in biowaste at all facilities/bagged compost brands apart from a vermicompost and bulk compost facility where films dominated (59.4% and 52.9%, respectively). Bulk compost samples from one processing facility contained a near-equal split of fragments (49.5%) and films (47.4%). The higher proportion of fragments in this study contrasts with previous studies of biosolids where fibers were highly abundant, ranging from over 60%-90% [Supplementary Table 1]. Differences in extraction methods and compost feedstocks may explain the higher recovery of fragments in this study compared to previous studies. On average, films were highly abundant in bulk compost (34.5%), vermicompost (28.9%), and bagged compost (19.0%) in this study, but the sources of these films are unknown. In this study, fibers were the second most abundant morphotype in the biosolids from two processing facilities, comprising 34.6%-39.8% of morphotypes on average within those facilities. Fibers in biosolids arise from the shedding of synthetic clothing, blankets, and other textiles during laundering[45]. Films, the predominant morphotype in two previous compost studies, were attributed to food scrap disposal bags and food packaging mistakenly placed in compost[17] and packaging from fertilizers and woven bags[41].

For the majority of microplastics detected, it was not possible to determine their product origin due to a lack of distinctive identifiable features and small size. Primary microplastics, however, may be recognizable, such as microbeads and glitter, but the actual origin and exact use remain speculative as they are variable in application. Beads, microbeads or 'spheres' were detected in all sample types in this study except in bulk compost. Microbeads are often reported as their own morphotype category in microplastics analysis as they gained attention worldwide as a source of unnecessary plastic litter as abrasives in cleaning products, industrial applications, and rinse-off personal care products[46,47]. Since 2017, several countries have banned the sale of microbead-containing products[46], and in June 2018, Aotearoa New Zealand also prohibited the manufacture and sale of wash-off products, including facial cleansers[48]. Despite the ban, microbeads were detected in this study, indicating the continued use of microbead-containing rinse-off personal care cleansers, or their presence from other sources unrelated to municipal wastewater where they could enter bagged compost. Beads have been previously found in the literature in higher proportions in biosolids from

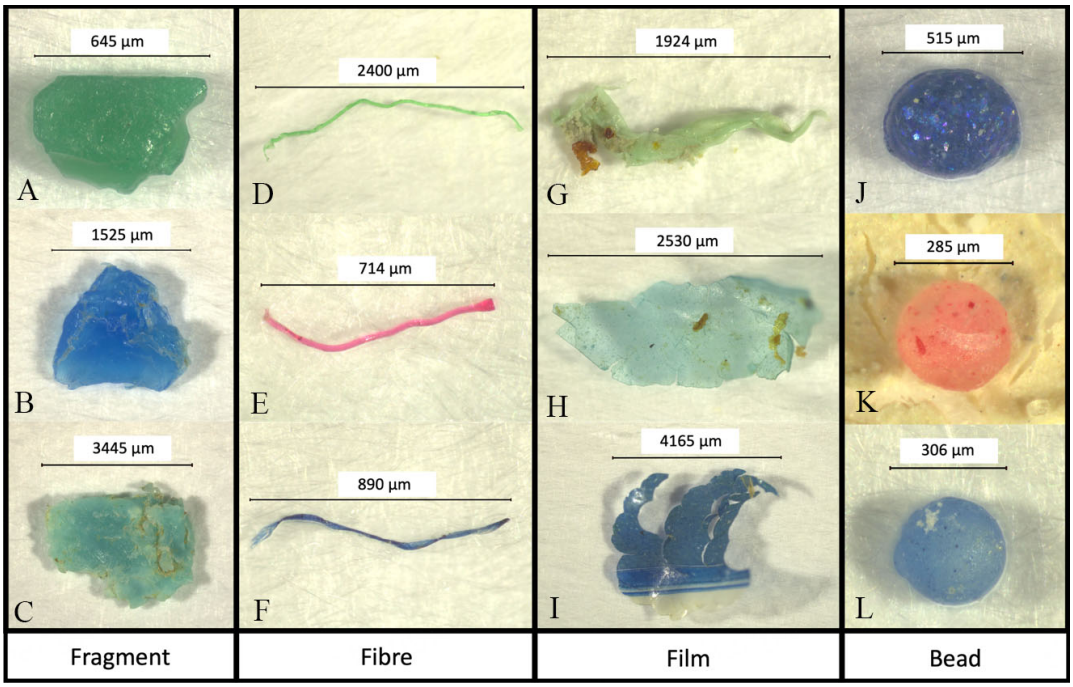

Figure 1. Examples of microplastic morphotype classifications of microplastics in biowaste samples. Fragments (A-C), fibers (D-F), films (G-I), and beads (J-L).

1.3%-17.1%[37,49] and in compost at 6.6%[17] of the total morphotypes in those studies.

Fragments of polyurethane (PU) foam sponge used as packing insulation and cleaning sponges are a distinctive morphotype [Figure 3A-C] and were present in every biowaste sample type in this study, comprising 1.8% of all microplastics, with 82% of that present in biosolids samples, suggesting origin from household cleaning sponges. Polyurethane foam sponge fragments have been reported only once before in the literature in coastal soil from China[50], and this study is the first to report it in biowastes.

Polyethylene terephthalate glitter [Figure 3D-F] was also present in every sample type and comprised 2.3% of total morphotypes, with 51% in vermicompost and 37% in biosolids samples. Suspected glitter particles have previously been observed in biosolids samples from Canada[12] and waste-activated sludge from Australia[51]. To the best of our knowledge, glitter has not been previously detected in composts. Glitter is widely reported in the literature as also being a primary microplastic, which is an unnecessary source of litter present in personal care products, cosmetics, homewares (art, crafts, toys, festive décor, kitchenware), and clothing and should receive comparable attention in terms of product bans as microbeads have[52,53]. Due to the prevalence of glitter in this study, it is necessary to consider it as an official morphotype.

Polymethyl methacrylate multicolored films [Figure 3G-I] comprised 8.2% of total microplastics in this study and were present in biosolids, vermicompost, and bulk compost. While these multicolored films are used in a variety of applications, they are most commonly used in packaging[54]. Multicolored films were highly abundant in a single vermicompost facility (facility 4), with a total of 220 items detected from the three replicate jars of that facility, comprising 70% of all multicolored films detected across all sample types. Multicolored microplastics in environmental samples are not as widely reported in the literature, but were detected in table salt from Italy[55], in 0.2% of surface water samples from a river in Taiwan[56], and in 22% of samples of European intertidal snails[57]. To the best of our knowledge, multicolored films have not been

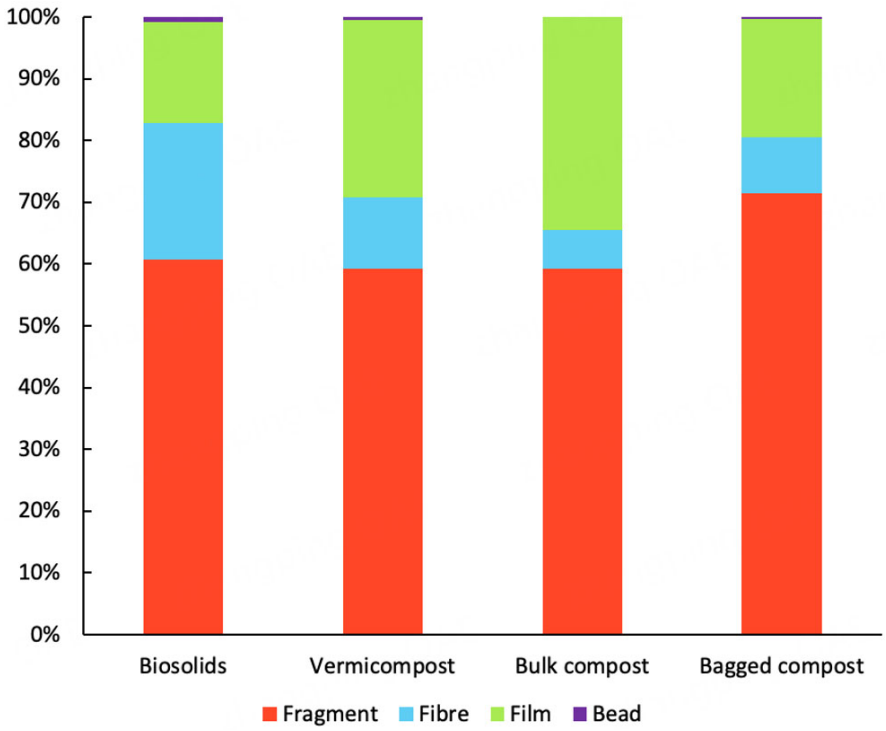

Figure 2. The average proportion of microplastic morphotypes by sample type.

detected previously in biosolid or compost samples. These films tended to be brittle, probably due to the processing methods, and fragmented easily when touched with tweezers.

Microplastics polymer type

Overall, PP (37.9%) and PE (28.6%) were the most frequently detected polymer types, followed by PMMA (11.7%), PET (6.8%), Polystyrene (PS) (4.1%), PU (2.7%), PA (1.2%), and PVC (1.0%). The remaining 6.0% (grouped as "other") comprised Polycarbonate (PC), Polytetrafluoroethylene (PTFE), silicone, epoxy resin, co-polymers, and polymers considered to be biodegradable. Co-polymers were identified in 90% of biowastes and made up 3.4% of all polymer types. Co-polymers detected included ABS, styrene-acrylonitrile (SAN), ethylene-propylene (EPM), ethylene-vinyl alcohol (EVOH), styrene-butadiene rubber (SBR), acrylonitrile-butadiene (NBR).

Polymers regarded as readily biodegradable were detected in 7/20 biowastes, with PLA detected in one vermicompost facility and two bulk compost facilities, PBAT detected in three bulk compost facilities, polycaprolactone (PCL) detected in one biosolid facility, and poly(methyl vinyl ether-co-maleic anhydride) (PVM/MA) in one biosolid facility. Polybutylene adipate terephthalate comprised 2.7% of polymers in bulk compost. Biodegradable polymers were not detected in bagged compost. To the best of our knowledge, this is the first study to have identified biodegradable polymers in mature biosolids and compost destined for land application. The presence of biodegradable plastics (particularly PLA and PBAT in vermicompost and bulk compost facilities) of the mature finished product in this study suggests that the composting processes employed at the commercial facilities and decentralized community initiatives investigated were not sufficient to support the complete biodegradation of these compostable polymers. There is little evidence of complete biodegradation of biodegradable polymers in soil, and therefore, it is likely they will have similar environmental fates to conventional polymers and accumulate[58,59]. More research into the suitability and

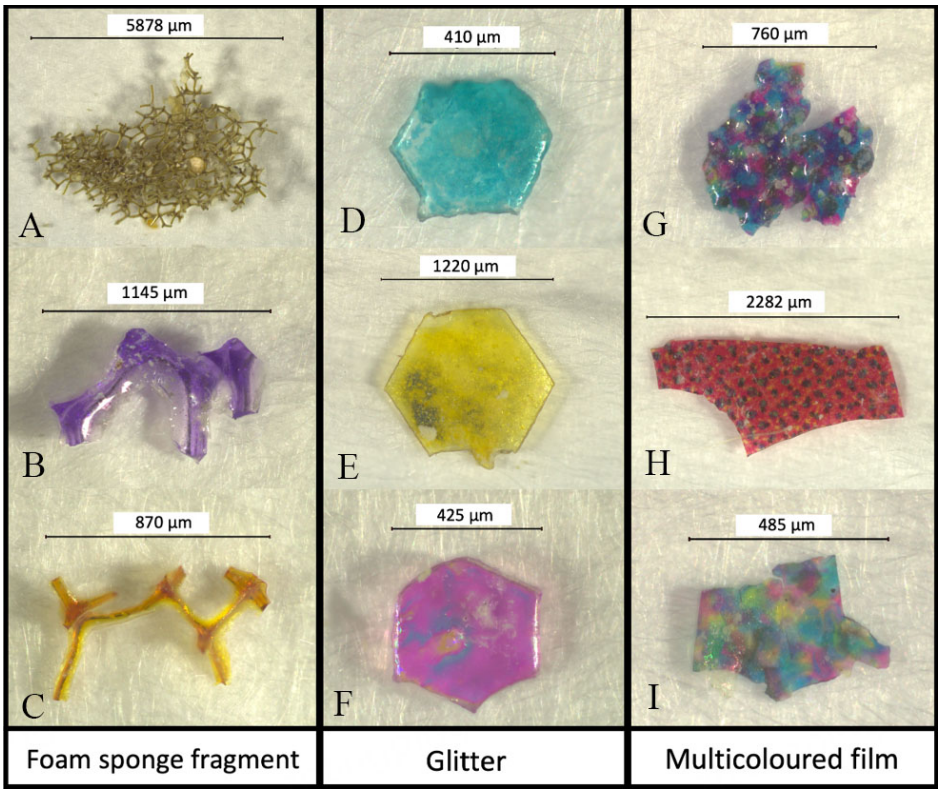

Figure 3. Examples of microplastic foam sponge fragments (A-C), glitter (D-F), and multicolored films (G-I) in biowaste samples.

use of these products is required before compostable plastics can be considered viable solutions to assist in reducing plastic pollution.

Polypropylene was the main polymer type across biowaste sample types [Figure 4], comprising 47.5% in vermicompost, 42.2% in bagged compost, 32.7% in bulk compost, and 29.3% in biosolids. The average polymer type proportions at each facility/bagged compost brand are presented in the Supplementary Materials [Supplementary Figure 4, Supplementary Table 4]. Polypropylene was the most predominant polymer in 15/20 biowastes, with PE (3/20) and PMMA (2/20) the most abundant at the remaining. In this study, PET and PU were present in higher proportions (13.1% and 6.3%, respectively) in biosolids compared to the other biowaste sample types. The high proportion of PE in biosolids in this study is comparable to two previous biosolids studies where PE was dominant; however, in four other studies, PET was the most abundant polymer type detected in biosolids [Table 1]. The dominant proportions of PE and PP in the composts of this study are comparable to those of a previous study, where PE was the most abundant polymer in compost (42.7%), followed by PP (31%)[17]. Polystyrene was found to be more abundant in bulk compost than in other biowaste types in this study, and was particularly highly abundant in composts from Spain and Germany[60,61].

Microplastics size

The majority of microplastics were in the size range of 100-300 µm (33.5%) and 500-1,000 µm (21.0%) [Supplementary Figure 5, Supplementary Table 5]. Approximately 18.7% of all microplastics were > 1,000 µm, with the largest microplastic detected at 26,540 µm in its longest dimension. Only 6.1% of the total microplastics were < 100 µm, and the smallest microplastic detected was 18 µm in the longest dimension. Bulk compost contained, on average, a higher proportion of microplastics < 100 µm (11.3%) compared to

Table 1. Estimate of microplastic loading in each amendment, based on an application rate of 10 tonnes per ha and the estimated microplastic concentration in the topsoil after each amendment application

Sample type	Biosolids	Vermicompost	Bulk compost	Bagged compost
MP abundance in amendment (particles/g)	2.71	2.69	1.94	1.10
MP loading in amendment (particles/ha)	2.71×10^7	2.69×10^7	1.94×10^7	1.10×10^7
[MP in topsoil] increase per application of amendment (particles/kg)	13.9	13.8	9.9	5.6

MP: Microplastics.

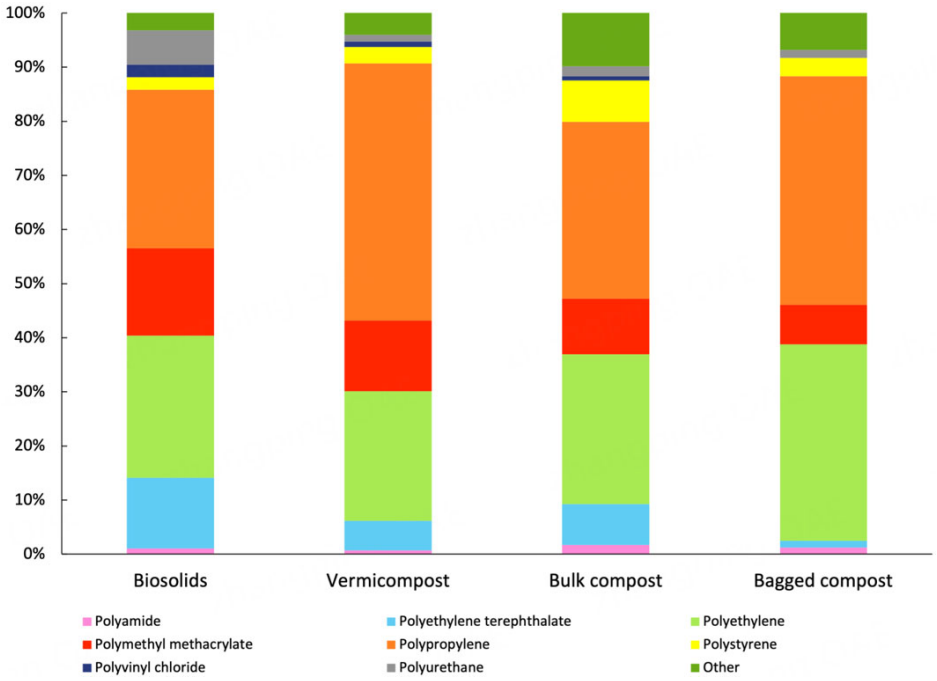

Figure 4. Proportion of microplastics polymer type identified in the four biowaste products. Other = PC, PTFE, silicone, epoxy resin, ABS, SAN, EPM, EVOH, SBR, NBR, PLA, PBAT, PCL, PVM/MA. PC: Polycarbonate; PTFE: Polytetrafluoroethylene; ABS: Acrylonitrile-butadiene-styrene; SAN: Styrene-acrylonitrile; EPM: Ethylene-propylene; EVOH: Ethylene-vinyl alcohol; SBR: Styrene-butadiene rubber; NBR: Acrylonitrile-butadiene; PLA: Polylactic acid; PBAT: Polybutylene adipate terephthalate; PCL: Polycaprolactone; PVM/MA: Poly(methyl vinyl ether-co-maleic anhydride).

other biowaste sample types, which ranged from 1.9% to 6.3%. Several previous studies reported similar findings, with the majority of microplastics in biosolids and composts < 500 µm in the longest dimension[17,62,63]. Comparison between studies is difficult due to variable extraction and analysis methods, particularly for spectroscopic methods limited by microplastic particle size.

Microplastic color

A wide range of colors were identified in varying abundances: blue (36.3%), green (22.9%), and colorless (12.9%), followed by pink (6.8%), black (4.6%), multicolored (4.5%), red (4.4%), grey (3.0%), yellow (2.3%), white (2.2%), orange (1.4%), purple (1.0%), and brown (0.1%). Within biowaste sample types, blue was the most abundant color of microplastic on average, except in bulk compost, where green was most abundant (36.3%) [Supplementary Figure 6, Supplementary Table 6]. Multicolored microplastics were detected in all sample types in percentages from 2.3%-8.2%, except for bagged compost, where none were detected. Similar to the results of this study, green, blue, and red were the most common colors in biosolids from the United Kingdom[62] and compost from China[43]. It must be noted that color is subjective and can be difficult to

distinguish between hues, and color may not be discernible for particles of decreasing size.

Estimation of microplastics applied onto land from biowastes

This study found a diverse array of microplastics, including both conventional and biodegradable polymers of varying sizes and morphotypes, in the mature finished products of biowastes, highlighting that the conditions during the treatment processes were not sufficient to biodegrade those polymers. All biowaste sampled as part of this study were destined for land application in Aotearoa New Zealand. The impact of microplastics in biowastes applied onto land will be specific to the designated land use; in this study, these include land rehabilitation, agriculture, horticulture, landscaping, and residential use [Supplementary Section 1]. Using the average abundance (MP/g) of microplastics in each biowaste type along with the parameters described in Section 2.8, between 1.10×10^7 to 2.71×10^7 microplastic particles are estimated to be applied onto the soil with the biowaste per hectare annually [Table 1]. The concentration of microplastics in the top 15 cm of soil is expected to increase by 5.6 to 13.9 MP/kg with each application of biowaste amendment [Table 1].

Previous studies have identified microplastic contamination in soils historically amended with compost (0.064-0.89 MP/g)[42,64-66] and biosolids (0.05-5.19 MP/g)[67-75]. Microplastic abundance in soils increased with successive compost and biosolid applications[65,67,75]. The persistence of microplastics in soil is of particular concern, with microplastics detected in soils 12 years after final compost application[65], and a North American and German study identified microplastics in soils 15 and 34 years after the last amendment with biosolids, respectively[71,72]. These observations prompt questions into the fate and behavior of microplastics in biowastes applied onto land, as relatively little is known. Microplastics are incorporated into soil through plowing, rain/irrigation percolation, and bioturbation by organisms and plants[70,72,76-78]. While most previous studies focused on the top 30 cm of soil, in a German study, microplastics were present at depths of 60-90 cm in soils amended with biosolids, and microplastic abundance and size decreased significantly from the 0-30 cm fraction[72]. Microplastics from amendments applied onto soils may be translocated between areas through surface and subsurface water movement and aeolian transport[79,80]. A field study undertaken in North America suggested microplastics collected from the runoff of biosolids- and manure-amended soils after natural precipitation events accounted for 0.4% and 0.3% of the total microplastics in those amendments, respectively[81].

Environmental microplastics carry both inherent and acquired chemical and pathogenic contaminant loads, along with different degrees of weathering. These factors can further influence additive leaching and particle toxicity[35,82,83], which must be taken into account when predicting toxicity. The impacts of microplastics from biowastes in the terrestrial environment are unknown. Previous ecotoxicological studies can serve as a guide to predict environmental impacts, but these are limited in terms of polymer type, particle size, additives, and degree of weathering and are not representative of environmental microplastics[84].

CONCLUSIONS

This study used a newly developed method to optimize the extraction and isolation of microplastics in complex solid organic matrices using the same extraction method, enabling, for the first time, the direct comparison of microplastics content of different solid biowastes destined for land application. In addition, vermicompost with variable feedstocks, including biosolids and industry processing wastes, were analyzed for microplastics for the first time. Microplastics contaminate valuable biowastes of biosolids, vermicompost, bulk compost, and bagged compost and were found in all samples. Distinguishable microplastics detected in biowastes included microbeads, glitter, PU foam sponge fragments, and multicolored films of remnants of packaging and embellishments, all of which have not been previously

widely reported in biowastes. However, the majority of microplastics in biowastes were fragments, films, and fibers of indiscernible origin. The most frequently detected polymer types were PP and PE, consistent with previous research. Biodegradable polymers including PLA and PBAT were detected for the first time in mature compost samples, suggesting the composting process was inadequate to allow complete biodegradation of these polymers. While the terrestrial fate and behavior of microplastics are relatively uncharacterized, previous research has demonstrated the application of biowastes is a long-term source of microplastic pollution in the terrestrial environment. From the results of this study, approximately 1.10×10^7 to 2.71×10^7 microplastics are applied annually per ha of productive soils from biowaste amendments in Aotearoa New Zealand. Mechanisms to prevent microplastics contamination of biowastes from residential, commercial, and industrial sources are urgently needed to ensure that these valuable resources can continue to be used without contributing to plastic pollution in the environment.

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Authors' contributions

Conceptualization, methodology, validation, investigation, writing - original draft, writing - review and editing, visualization: Ruffell, H.
Conceptualization, supervision, writing - review and editing, project administration, funding acquisition: Pantos, O.
Supervision, writing - review and editing: Robinson, B.
Conceptualization, supervision, writing - review and editing, project administration, funding acquisition: Gaw, S.

Availability of data and materials

The raw data supporting the findings of this study are available within this article and its Supplementary Materials. Further data are available from the corresponding author upon reasonable request.

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Conflicts of interest

All authors declared that there are no conflicts of interest.

Ethical approval and consent to participate

Human ethics was provided by the University of Canterbury Human Ethics Committee, reference number HCE 2021/17/LR-PS.

Consent for publication

Not applicable.

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