Available Sampling Methods for Plastic Waste

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Given the rapid development of plastics recycling in recent years, the need for guidelines for sampling and material characterization is steadily emerging. However, there still exists a considerable scarcity of methods that enable proper material data acquisition. It was found that neither the literature nor the standards provide a comprehensive practice that considers the distinctive characteristics of plastic waste and applies it to different situations along the value chain. Two variants of the proposed plan were evaluated based on the flake size distribution and the apparent density of four different pretreated polyolefin (PO) waste materials. Combining stratified random sampling with composite sampling yields a good sampling technique for rigid PO waste. Moreover, the analysis of a composite sample adequately conveys the true material properties of a sublot or lot.

waste management plastics recycling waste sampling

1. Introduction

The total production of plastics had grown to 368 million metric tons (Mt) in 2019 [1][2]. This number is expected to rise to 460 Mt by 2030 [3]. While, over 50% of the global production of plastics takes place in Asia, Europe accounts for only 16%, coming after China and the countries of the North American Free Trade Agreement (NAFTA) [1][2]. By 2015, the carbon footprint of plastics had increased by a factor of two with respect to the estimations of 1995 [4]. Hence, ample pressure from the public and policy makers has been imposed for solutions towards a more sustainable value chain in the plastics industry. Since the environmental impact of plastics is often lower than that of other alternatives, a total ban of plastic products is definitely disadvantageous [4][5]. It was estimated that the production of plastics and other means of recovery, such as incineration, annually generate around 400 Mt of CO_2. However, the recycling of plastics can reduce the extraction of fossil fuels and inhibit the greenhouse gas emissions [5][6][7][8]. Therefore, several governmental bodies and policy makers established action plans to emphasize the importance of plastics circularity through reusing and recycling [6][9]. In 2018, the European commission set an ambitious action plan and a strategy to enhance plastics recycling and encourage further developments in this field. The plan aims to recycle over 50% of plastic waste in Europe and to make all packaging plastics either reusable or recyclable by 2030 [6][7]. Furthermore, the plastic circulation strategy, that was introduced by the Japanese government in 2019, is another example of the increasing awareness of plastics circularity. The strategy dictates a complete transition to reusable and/or recyclable plastic packaging by 2025 followed by a target to reuse or recycle 60% of all plastic containers and packaging products by 2030. Moreover, it also aims to fully recycle or reuse all plastic waste by 2035 [9].

1.1. Life Cycle of Polyolefin Products

PO materials have become the most widely used polymers in the plastic industry. POs, mainly polyethylene (PE) and polypropylene (PP), dominate the European market covering around 50% of the total plastics demand [1]. The low costs and the diverse property profile of the products made them a perfect fit for packaging applications [10]. Packaging represents the largest share with 39.6% of the total production of plastics in Europe [1]. Consequently, packaging products account for 61% of postconsumer plastic waste. In 2018, 17.8 Mt of postconsumer plastic waste (PCPW) from packaging applications were collected. However, only 42% of the collected waste was recycled [1][11].

Plastic packaging can be divided into flexible and rigid packaging. Rigid packaging (RP) includes a wide spectrum of products, such as thin-walled containers, bottles, bottle caps, transport boxes, etc. These products are normally produced by various processing technologies (e.g., injection molding, extrusion, blow molding) and, based on the desired property profile of the end product, proper material selection is required [12]. Melt flow rate (MFR) of the polymer is usually used as an indicator to determine the compatible processing technology for a certain product [13][14][15]. Gall et al. [14] carried out a survey of relevant material selection guides and data sheets from several polymer suppliers to investigate the diversity of materials that are used for cap and closure applications in the beverage industry. In another study, Eriksen et al. [13] presented a link between typical packaging products and suitable processing methods based on the MFR value of various PO grades. Both studies agree that MFR values of different PE and PP types, which are typically used for packaging applications, vary greatly depending on the processing technology as well as the product requirements. According to Erikson et al. and Gall et al. [13][14], the spectrum of MFR values of some packaging products may range from as low as 0.3 up to 30 g/10 min in the case of high-density polyethylene (PE-HD) and it expands to well above 50 g/10 min for PP.

The first type of rigid plastic waste is usually generated during the production of RP products. This fraction never reaches the consumer, thus it is referred to as pre-consumer plastic waste, or more widely known as postindustrial plastic waste (PIPW) [16][17]. PIPW typically includes the residues of the production process, such as injection molding runners and sprues [18]. Such waste streams are often composed of mono polymers and not contaminated by other materials, thus they can be recycled easily and are usually used to produce high quality recyclates [19]. On the other hand, PCPW is generated after the intended use and disposal of a product by the end consumer (household or commercial) [16]. Hence, the composition of such waste streams is more complex, since they are contaminated and consist of various polymers that originate from multiple applications and have distinct property profiles (e.g., MFR, color, density) [20].

It is believed that the contamination level of PCPW is highly dependent on the employed collection system and varies from one country to another [19]. In general, there are several collection strategies for PCPW including single-source separation, whereby certain PCPW materials are separated directly by the consumer at disposal or commingled collection, in which all different waste materials are disposed of and collected together [21]. In Austria for instance, two collection schemes for plastic waste are implemented. The first scheme is by collecting all packaging PCPW in the yellow bag/bin after disposal. However, in some regions, only plastic bottles are collected for recycling, whereas the other types of light-weight packaging are collected together with the residual solid waste to be used for energy recovery [22]. Once the plastic waste is collected, it goes to the waste management facilities, where it is sorted and treated to be prepared for the next recovery processes [19][20]. While the above mentioned individual process steps are describing a formal collection system, as typically found in EU countries, in low- and middle-income countries (e.g., in Africa, Asia, Latin America) an informal collection system (ICS) is usually employed [23][24]. Presumably, depending on the specific ICS, the contamination level can vary significantly. However, according to a study carried out by Gall et al. [25], the implemented ICS in Nairobi, Kenya, leads to recyclates with a relatively low contamination level, although it relies on waste pickers.

There are several recovery paths for the plastic waste, including mechanical recycling, chemical recycling, dissolution, or energy recovery via incineration or re-oiling processes [11]. Usually, the appropriate recovery method is selected depending on the contamination and degradation levels of the plastic waste [26]. However, the current research is mainly focusing on the sampling and characterization of pretreated rigid PO flakes for mechanical recycling.

1.2. Mechanical Recycling of Plastic Waste

The EU directives of waste management have established new regulations and policies to eliminate landfill and replace it with more sustainable means of disposal and recovery of plastic packaging [27][28]. Consequently, a lot of effort has been made to make plastic production circular and transform it from the old “take-make-dispose” model into the “reduce-reuse-recycle” model. Extending the service life of polymeric materials through reuse and recycling is essential for a circular economy (CE) of plastic packaging [11][29][30]. Amongst the different methods of plastics recovery, mechanical recycling of PCPW is still considered the most desirable approach and the most sustainable practice after reusing [17][31]. Mechanical recycling, or secondary recycling, usually refers to processes that transform the plastic waste into secondary raw materials by physical means (e.g., shredding, extrusion) [26][28]. The regranulation of plastic waste into pellets via extrusion is the most common recycling technology due to its relatively inexpensive operational costs, applicability to different polymers, high efficiency, and the possibility of enhancing the quality of the polymer melt by the addition of degassing and filtration units to the extruder [28][31][32]. In general, the operational sequence of mechanical recycling always starts with the collection of PCPW and ends with the regranulation process. However, certain pretreatment steps, such as sorting, shredding, washing and drying are usually implemented before extrusion to improve the quality of the input materials, thereby the quality of the resulting recyclates [17][33]. Hence, mechanical recycling can, practically, be divided into preparatory steps, whose product is pretreated flakes, and the regranulation process, which converts the flakes into recyclates. However, the order and the repetition of the pretreatment steps may differ depending on the source and the contamination level of the plastic waste [19].

1.3. The Inherent Heterogeneity of Plastic Waste

Despite its high efficiency and relatively low costs, mechanical recycling of PO waste still has several issues to overcome. The major challenge is the low performance of the regranulates in comparison to their virgin counterparts, which limits their applicability to numerous applications and deems them to be used for inferior products [26][34]. This is mainly due to two factors. The first is the possible material degradation, which is induced by the high temperature and shearing during the extrusion process, while the second factor is driven by the heterogeneity of the plastic waste [17][26]. PCPW is substantially heterogeneous and often contaminated by other polymers and chemicals [19], since it is generated from different sources and different products [13]. The high heterogeneity of PCPW is a consequence of several factors that can be summarized as: 1. Using immiscible polymers in the production of plastic products [35]; 2. Various product types that can be produced by different processes, thus higher feedstock variability in regard of the property profile and morphology of the materials [36]; 3. Variety of features in the product designs, such as colors, which increases the complexity of the composition of a waste stream as well as difficulty in sorting certain colors (i.e., black) due to the limitation of the sorting technologies [37][38]; 4. The use of multiple inseparable polymers in some packaging applications (e.g., multilayer films or coffee caps), which always lead to a certain degree of polymer cross-contamination [39][40].

The possibility of producing high-quality recyclates that can substitute virgin materials is highly dependent on the quality of the incoming waste and its ability to meet the requirements of particular applications as well as the operational conditions of the recycling process [41][42][43][44]. In their assessment of the management system of plastic packaging in Austria, the researchers of [29] investigated the polymer composition of the waste flow and found that the plastic waste is mainly dominated by PE-LD, PET, and PP. Similarly, the researchers of [40] investigated the source of household rigid plastic waste in Denmark to study the composition of such waste streams and to evaluate their degree of heterogeneity. They found that the waste consisted of a variety of packaging products from various applications (e.g., shampoo bottles, beverage bottles, food trays), which are made of different polymers with different designs and colors. Both studies suggested that such compositions are not easily converted into high-quality recyclates. The reason is that these polymers cannot be sufficiently sorted by the available technologies (e.g., density sorting, NIR) because of the similar densities of some polymers (e.g., POs), black pigmentation, and the size and shape of the flakes [17][37]. Consequently, the heterogeneity of the plastic waste will still be present even after the pretreatment processes. Another study was carried out by Luijsterburg and Goossens [45] to examine the impact of the collection system on the composition and selected properties of postconsumer PO recyclates. They found that the collection system did not have a significant impact on the quality of the recyclates. However, the properties of the recyclates were considerably influenced by the sorting and the reprocessing step.

Therefore, taking into account the specifications of the final product, the collection of detailed information about the composition and the basic properties of the input materials throughout the recycling process is essential to determine the processing parameters and control the quality of the recycled output [13][43]. A typical recycling process in a recycling plant starts with the pretreatment (e.g., shredding, sorting, washing) of sorted plastic waste. The pretreated plastic waste is transferred into regranulation. Afterwards, the resulting recyclates might undergo some post-treatment steps (e.g., decontamination, compounding) before being converted into new products [33]. Compounding, with virgin materials and/or additives, is usually used to improve the quality of the recycled plastics to fulfil product requirements [15][46]. Several organizations and initiatives introduced guidelines that include sets of requirements and test protocols to evaluate the reliability of the waste supplier as well as the recyclability of the input materials in the recycling industry [47][48]. However, the sampling procedure is often overlooked despite its importance for the generation of reproducible test results that can be used to improve the process and the quality of the end product [49][50].

2. Available Sampling Methods for Plastic Waste

Proper characterization and testing of input materials are crucial for the generation of accurate datasets. These datasets are used to improve the recycling process and technologies, thus, to produce recyclates that meet the quality attributes of specific applications. In general, there is still a lack of information in terms of material characteristics, which creates the need for more studies to be performed in this field [51][52][53]. When it comes to sampling procedures, the gap is even bigger since most of the research is based on the analysis of household waste at the source to provide an overview of the composition of different waste streams in different regions [54][55][56][57]. Such studies are often built from a holistic perspective to deliver statistical analyses for the decision and policy makers without considering the technological and engineering aspects of the plastics recycling process nor the final quality of the recycled materials [58][59].

According to Dahlén and Lagerkvist [54], there is no international or European standard for the sampling and characterization of household waste, which has led to various sorting and sampling approaches and hindered the reproducibility and comparability of results between different studies [55][57]. In their research [54], provided an overview of the most common methods in composition studies of household waste in general and analyzed the applicability of the theory of sampling (TOS) for the collection of samples from household waste. They concluded that, for a proper sampling procedure, it is necessary to divide the investigated flow into strata, define the number of samples and sample size, define the sampling location, and to select the component characteristics to be investigated. Another study carried out by Glass and Dominy [60] examined the influence of acceptance sampling before the sorting step on the quality of recycled polyethylene terephthalate (PET). In their study, they focused on the recovery level of polyvinyl chloride (PVC), whose presence deteriorates the quality of recycled PET. They inferred that knowing the PVC level in a feedstock through acceptance sampling combined with the recovery rate of the sorting process helps to predict if the desired quality can be achieved.

Reliable sampling provides information about a whole lot through the inspection of a representative sample with a manageable size. However, only proper unbiased sampling will yield such representative samples [61]. In other words, the sole inspection of a sample is not enough to ensure its representativeness, thus, the whole sampling procedure has to be evaluated [62][63]. There is still no consensus on how to perform proper sampling in the plastics recycling industry to ensure a constant quality of the recycled plastics. Sampling methods from the most prominent norming organizations, including the International Organization for Standardization (ISO); Deutsches Institut für Normung (DIN); American Society for Testing and Materials (ASTM), were selected based on a set of criteria to be researched. To be selected, the standards had to be available in English or in German and to explicitly address sampling. Additionally, they had to be directed at plastic waste, waste management, heterogeneous waste, or at bulk materials in general.

ISO has not yet composed any standardized method for the sampling of plastic waste. However, it established ISO 11648 as a two-part technical practice for the statistical sampling of bulk materials, for which no standards have yet been introduced [64][65]. The first part of the standard [64] outlines the necessary technical terms and statistical methods that can be used for the sampling of bulk materials in general, while the second part provides a guideline of the basic sampling techniques from particulate materials. The aim of this part is to create a consistent approach to attain representative primary increments, from which test samples can be prepared without introducing systematic errors. According to the standard, the operational sequence of the quality inspection of bulk materials usually consists of sampling, sample preparation, and analysis and testing of the sample. To have an acceptable level of precision, four points should be taken into consideration along this sequence. First, the primary increments of the sample should be collected in an unbiased fashion; second, avoid the introduction of systematic errors during sample preparation; third, the relevant quality characteristics should be defined; fourth, performing the appropriate analytical techniques on calibrated equipment according to standardized methods.

To collect unbiased increment samples, all particles of a bulk material must have equal chances to be picked out of the lot. Hence, a reliable sampling scheme reduces the effective bias to zero and thus ensures that the sampling procedure is based on correct selection probabilities [62][65]. Theoretically, bulk materials are always three-dimensional. However, in industrial operations materials are usually described by a one-dimensional model, as the other two dimensions are presumed of less importance and neglected. Thereby, stratified systematic sampling can be carried out for the collection of the primary increments. However, if a systematic error can possibly be introduced, it is strongly recommended to use stratified random sampling within defined intervals instead [65]. Consequently, ISO suggests a sequence of eleven steps to be followed in the development of a sampling practice for regular QC activities. In certain experimental cases, such as determination of variability, the sample increments can be investigated individually. Nonetheless, in regular sampling, the increments of a lot or a sublot are combined to constitute a gross or composite sample, from which portions are then taken for the sample preparation of the respective test methods [65].

In contrast, the technical committee (TC) for plastics (CEN/TC 249) at the “Deutsches Institut für Normung” (DIN) launched a series of ten publications on plastics recycling. Two of these publications are designed specifically for the sampling and sample preparation of plastic waste and recyclates [66][67]. However, these two standards are primarily derived from ISO 11648 in combination with other sampling standards of other material classes (e.g., metal ore and coal). Their advantage is that they take into consideration the characteristics and conditions of plastic waste and recyclates and provide a simplified sampling approach to the plastics recycling industry. The purpose of DIN 16010 is to set a guideline that enables plastic recyclers to calculate the risk of inaccuracy due to a sampling regime. It also aims to define the basic sampling procedures that must be followed to characterize the sampled material. Hence, it describes a system of sampling methods for testing plastic waste and recyclates and presumably covers the different stages of the recycling process. According to [66] these procedures are expected to lead to representative samples. However, some variations may occur due to certain factors, such as the mixtures of different polymers, origin of the waste, previous application, remaining content from use, moisture, or residual from inert content in the material. Nevertheless, the collected samples must be sufficiently representative of the lot or the batch to generate useful information for the user. Hence, the practice suggests using statistical tools (e.g., tstudent distribution) to evaluate the representativeness of the sample and determine the effectiveness of a sampling routine. Moreover, for the establishment of a sampling plan, it recommends following the same operational sequence in accordance with ISO 11648–part 2.

On the other hand, DIN 16011 [67] defines procedures to be followed in sample preparation for the subsequent testing of various material properties (e.g., chemical, mechanical, thermal). The standard emphasizes that the sample should remain representative after the sample preparation steps. Thus, it must be well mixed and all processes that might lead to segregation should be avoided before the extraction of the laboratory portion. This is particularly necessary when increments or subsamples are collected from different sources and then combined to form a gross or a composite sample. Additionally, the contamination behavior of the sample should be observed carefully to ensure a sufficient level of homogeneity. According to DIN 16011, the determination of the minimum size of a laboratory sample is dependent on the material properties to be measured. In addition, the minimum mass of a laboratory sample is affected by the particle size as it increases with the larger grain size of the single unit (e.g., granulate, flake). Hence, the minimum sample mass is usually determined based on the largest particle size of the material to be analyzed. For this purpose, the standard provides a table of values of various particle sizes with different precision levels for the determination of the mass of a laboratory sample. Furthermore, in the case of test methods that require the production of molded parts (i.e., mechanical tests), the sample preparation is also affected by the physical form of the materials. Therefore, size reduction might be required in the case of flakes and agglomerates. DIN 16011 provides a short guideline on the basics of sample preparation including different techniques and recommendations for size reduction, mixing, and splitting.

Nonetheless, these two technical guidelines only offer broad principles and concepts that should be taken into account when creating a sampling plan for plastic waste and recyclates. They do not, however, offer a concrete sampling procedure that could be implemented in various steps throughout the value chain of plastics recycling. Moreover, they primarily concentrate on the statistical analysis of samples, thus, they are insufficient for routine sample collection and QC activities. However, they can be employed for the quality inspection of incoming materials or waste streams (e.g., acceptance sampling).

The technical committees (TCs) of the American Society for Testing and Materials (ASTM) have published numerous standards for the sampling of specific materials, although the TC on plastics (D20) has not yet introduced any practice for the sampling of plastic waste nor did the TC on waste management (D34). However, the subcommittees D34.01.01/02 of the D34, whose responsibilities are planning for sampling and sampling techniques, established a complementary suite of 25 standards, which provide guidance and knowledge for the development of a sampling strategy for a variety of waste materials in different states and situations [68][69]. Based on the criteria proposed by the researchers, seven of these standards were selected to be research. It is known that each material has its own peculiar characteristics and problems due to certain factors, such as its state; location; degree of homogeneity; size; stratification; or segregation [70]. These factors must be considered in the development of sampling strategy for a certain material type. ASTM D4687 [61] defines a waste sampling plan as "*A scheme or design to locate sampling points so that suitable representative samples descriptive of the waste body can be obtained*". This practice provides general information on multiple aspects of waste sampling that are present in the most common situations. These aspects include safety, sampling plans, quality assurance, general sampling considerations, preservation and containerization, cleaning, logistics (e.g., packaging, shipping), and chain-of-custody procedure. The advantage of this standard is that it addresses the essential points and requirements to be considered in the formulation of a sampling plan. Nonetheless, it does not provide any comprehensive framework or sampling procedures for any specific applications or waste types.

Generally, ASTM emphasizes the importance of representative samples for a successful sampling plan in all listed standards. Hence, it provides a detailed discussion on the topic of representative sampling for waste management activities in the standard guide ASTM D6044 [71]. This guide defines the representativeness in sampling and describes the attributes that a sample should possess to adequately infer the characteristics of a population. It also outlines the process by which representative samples can be obtained and identifies error sources that should be avoided. The major emphasis in this standard is given to bias, as it has a direct impact on the inference from samples to their populations. The standard differentiates between three sources of bias that must be considered in the sampling design including sampling bias, measurement bias, and statistical bias. However, the practice only provides a theoretical overview of the substantial points and factors that should be considered for obtaining representative samples.

For heterogeneous waste, ASTM D5956 [72] offers a practical guide for sampling strategies, which apply to several applications and material types. The standard merely presents a nonmathematical discussion of the issues related to the sampling of such waste materials and takes into consideration certain population attributes (e.g., degree of heterogeneity). Yet, it is consistent with inferential statistics and the sampling theory of particulate materials; thereby, it can possibly serve as a basis for statistical treatment of sampling problems. The standard thoroughly discusses the different definitions of heterogeneity and stratification from different perspectives with illustrative examples to have a basic understanding of these terms. Moreover, it describes the relationship between samples to populations and how it is reflected in the design of the sampling plan. According to ASTM D5956, the degree of heterogeneity is estimated by the evaluation of the sample attributes, particularly, the representativeness of a population characteristic and the intersample variance. Therefore, the collection of multiple samples by an appropriate sampling scheme is necessary to be able to estimate the degree of heterogeneity of a population and to have a better representation of its properties. This information, in combination with the specifications and the test requirements, can be utilized to optimize the sampling procedure.

The D34 committee offers several sampling guides that consider the spatial dimensions of a waste population. One of which is the standard guide ASTM D6009 for the sampling of waste piles [73]. This practice presents sampling methods for collecting representative samples from waste piles, such as municipal waste, for the purpose of waste characterization, treatment, or disposal. Furthermore, it addresses the factors associated with the waste body and their impact on the strategic and design consideration of a sampling plan. The standard emphasizes that the development of a sampling strategy for waste piles is highly influenced by the variability of the pile features, especially those that stem from the history of the waste, the physical characteristics of the pile, and the waste properties. For instance, the number of required samples as well as their accessibility are influenced by the variability in the pile size, shape and stability. In terms of waste characteristics, the knowledge of particle size or contaminant distribution reduces the necessary sampling requirements to define the properties of interest. Therefore, this guide takes into consideration these factors to provide an overview of potential sampling strategies and equipment that cover distinct situations of pile waste sampling. However, it only addresses in-place methods including directed sampling, simple random sampling, stratified random sampling, systematic grid sampling, and systematic sampling over time.

In certain situations, sampling from a moving conveyor is more favorable than in-place methods for the evaluation of waste piles as it ensures equal chances of being picked for all material units [73]. Therefore, D34 published a standard practice, ASTM D7204, for waste stream sampling on open and closed conveyors [74]. The practice provides a concise description of procedures for sampling from conveying systems regardless of their movement direction. Moreover, it is directed at particulate materials and slurries that can be scooped or shoveled. However, it is not applicable for large size particles. Although the standard does not make any reference to plastic waste or recycling, the procedures for sampling on a conveyor belt are generally applicable for various applications. Hence, they can be useful for developing a sampling plan for plastic waste recycling.

Finally, ASTM D6311 [75] presents a systematic approach for the selection and optimization of sampling designs for waste management activities. The standard aims to establish a practical guideline for the development steps towards an optimum sampling scheme. This guideline covers the stages from the initial design selection to the final establishment. It also outlines the factors by which the selection process is influenced, such as the performance characteristics, regulations, project objectives, physical sample problems, representativeness, etc. Furthermore, it describes the optimization process of nominated design candidates based on an eight-criterium set. Additionally, the standard provides an overview of the most popular sampling techniques used in environmental and waste management contexts.

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