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Review of chemical characterization methods and data for compositional analysis of fruit wastes: current status and opportunities

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Abstract: Fruit waste (FW), mainly from agroindustry, is currently left behind in landfills despite its rich composition. The bioactive compounds (e.g., oils, polyphenols), carbohydrates, and lignin present in this biomass type require comprehensive characterization (i.e., identification and quantification) before they can be used as raw materials in biorefineries. This review collected information from scientific papers on FW compositional analysis methods and characterization data; the information needs to be compiled in a systematic, standardized, and comprehensive way to understand and quantify the true potential of FW as feedstocks for biorefineries. The information gathered in this review allowed us to identify the biomass fractions that could be valorized further depending on the kind of FW (peels, seeds, or seed vessels, and pomace or mixed residues). Fruit waste differs from conventional lignocellulosic biomass due to the presence of higher amounts (>5%) of extractives – pectin, and starch. This review describes current compositional analysis methodologies to identify possible strengths and weaknesses that could affect the adequate selection of valorization platforms. As no current methodology allows the composition of FW to be described thoroughly, this work identifies procedures applicable to biorefineries that use FW. Possible improvements are suggested to fill methodological gaps in the quantification of samples with large amounts of extractives and pectin. The standardization of methods for FW's quantification is fundamental for the adequate integration of different valorization platforms into biorefineries. It is essential to consider all the substances present in FW to exploit fully their potential for new value-added molecules, including oils, polyphenols, and pectin. © 2024 Society of Industrial Chemistry and John Wiley & Sons Ltd.

Supporting information may be found in the online version of this article.

Key words: fruit wastes; compositional analysis; waste valorization; biorefineries

Introduction

The global population has been increasing since the Industrial Revolution. This has led to an increase in energy consumption and resource exploitation. Fossil fuels have been a reliable source of energy and everyday products; nonetheless, economic growth has been achieved at the expense of environmental damage, deterioration in health, and ongoing social inequality.¹ As a result, the long-term stability of modern societies is at risk due to their reliance on oil, the supplies of which are becoming depleted.² There is consequently a need for sustainable solutions in a (complete) circular economy model that reduces consumption, and that reuses and recycles waste materials and replenishes the supply chain.³

Biorefinery systems that use biomass-based raw materials to obtain value-added products through biological or chemical conversion processes are an attractive concept. These complex arrangements can be used to valorize waste biomass such as fruit waste (FW) (a kind of vegetable biomass comprised of peels, pulp residues, and seeds – the fruit's nonedible parts). Agroindustry and municipalities are the primary sources of this organic waste, which is discarded at each step of these food value chains.⁴ After most of the pulp is removed, transformed into various food products, and conserved, most of the fruit's weight is left behind as FW, which is sent to landfills without further valorization.⁵ Even though FW is usually considered a nonhazardous waste,⁴ it could cause undesired emissions, secondary wastes, acid gases, and other toxic substances (i.e., dioxins and furans produced by incineration), leading to serious environmental and health risks.⁶ In 2018, 866 million tons of fruits were produced on average in the world, with Asia leading (~57%), followed by the Americas (~19%), Africa (~13%), Europe (~10%), and Oceania (~1%),⁷ which provides an abundant source of biomass with potential use in biorefineries, especially for the top-producing countries in each region. Some examples are India, China, Indonesia, Thailand, Brazil, the USA, Mexico, Colombia, Iran, Egypt, Nigeria, Turkey, Spain, and Italy. Consequently, FW could be an important feedstock for biorefineries due to its rich content of carbohydrates and bioactive compounds, which could be transformed into high-value products such as compost, vermiculture, pectin, enzymes, essential oils, antioxidant compounds, edible fungi, dietary fiber, bioethanol, biogas, and other products from

thermal valorization,^{8–15} with multiple uses in cosmetics, pharmaceuticals, foods and feeds, and bioenergy.

Fruit waste has a heterogeneous composition that requires systematic, standardized, and comprehensive characterization to understand and quantify its true potential as a feedstock for biorefineries. Adequate characterization of FW would make it easier to know the exact amounts of relevant compounds necessary to design its processing routes and select valuable and marketable products.^{4,6} Even though FW has a similar composition to conventional lignocellulosic biomass in terms of cellulose, hemicellulose, lignin, protein, and ash,¹⁶ it also has high pectin, starch, and extractives content (>5% w/w).^{17–20} The presence of these particular substances makes fruit-derived biomasses both attractive and challenging in terms of their quantification and valorization.²¹

Hence, a comprehensive and systematic collection of compositional analysis data of FW is required in order to harmonize characterization methods for application to biorefineries. For that reason, the aim of this review is to collect information systematically about the compositional analysis of FW, which is relevant to the concepts of biomass valorization and biorefineries. It is also an aim of this review to establish and suggest improvements to the current methodologies of compositional analysis to construct better decision criteria for biorefinery design when FW is used as a raw material. It is expected that this work could serve as a basis for future work providing information on how best to address a comprehensive and complete characterization of FW and provide compositional data for multiple FWs.

Research method and structure of the review

The procedure followed in this study is based on the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) guidelines for systematic reviews and meta-analysis.²² Figures 1 and 2 show a schematic representation of the steps taken and decisions made during the literature review process. The literature analysis included internationally indexed scientific papers (excluding conference papers) published in the last 20 years (March, 2003–March, 2023) in databases including Google Scholar, Scopus, ScienceDirect, SpringerLink Journals, Pubmed, Taylor & Francis Journals, and American Chemical Society Publications. Only peer-reviewed articles were considered.

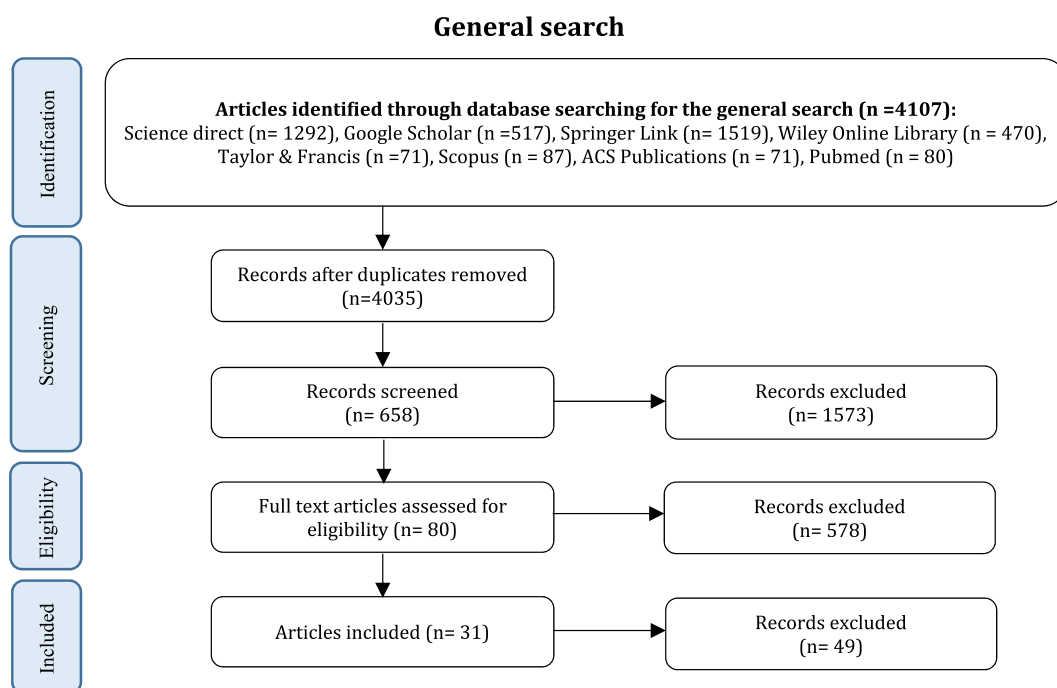


Figure 1. Flow diagram used to select papers based on the PRISMA guidelines for the general search.

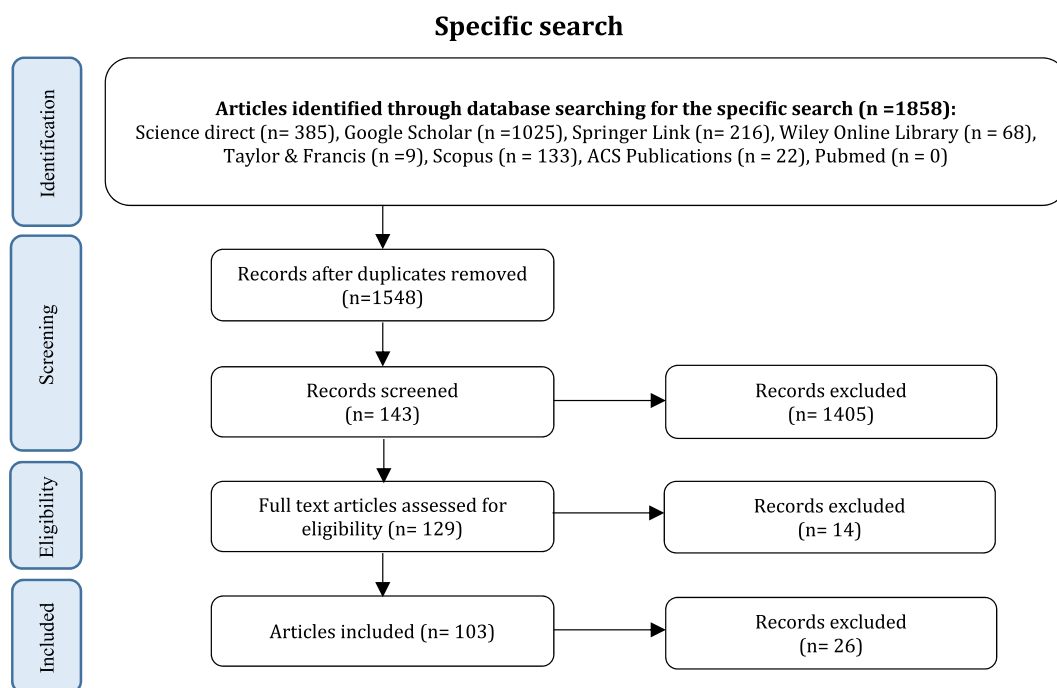


Figure 2. Flow diagram used to select papers based on the PRISMA guidelines for the specific search.

First, a general search was done to identify potential publications relevant to the topic using the words 'Fruit', 'Waste', and 'Biorefineries'. Next, articles related to the compositional analysis of FW were selected and screened,

checking first the titles and abstracts mentioning fruit feedstock and then selecting relevant articles with quantitative data on the composition of FW, as shown in Figs 1 and 2. A secondary search included more specific keywords to

ensure that relevant information was not excluded from this review. Supporting Information, [Table S1](#), shows in detail the combination of keywords used and the number of results obtained for each database during the two search steps. In all cases, duplicate articles and publications not associated with the topic of this review were discarded (i.e., papers reporting information not related to FW and their composition). The exclusion criteria also considered the removal of papers that did not include experimental data or that presented incomplete or partial results (i.e., those papers not intended to measure the composition) or papers that reported data obtained using methods that are not standardized (i.e., not validated by internationally recognized organizations). The review will first present a description of the composition of vegetable biomass. Then a compilation of the available standardized methodologies to characterize FW's composition will be shown. After that, a collection of compositional data on FW that has been reported in the literature (using these methods) will be presented. Finally, the information gathered will be used to discuss and propose a unified methodology for the compositional analysis of FW for their use in biorefineries.

Composition of vegetable biomass

Vegetable cell-wall composition is complex and varies depending on taxonomical groups, tissues, cell types and layers, and the age of the plant.²³ Most vegetable biomass contains different proportions of structural polysaccharides, starch, lignin, proteins, silica, water-soluble carbohydrates, organic acids, and other secondary metabolites.^{24,25}

The first plants (*Bryophytes*) evolved from green algae and adapted to life on land by developing a dermal tissue called a cuticle, which prevents water loss and gives protection. At this point, plants also had several structural polysaccharides, such as cellulose, hemicellulose, and pectin. Later, lignin allowed the first vascular plants (ferns) to grow higher and have stronger tissues.²⁶ The evolution of gymnosperms (plants with exposed seeds) gave rise to the abundance of tissues that sustain the growth of seeds and contain starch, lipids, and proteins.²⁴ More recently, angiosperms (seed plants with fruits and flowers) evolved an enclosure rich in sugars, pectin, and phytochemicals, which attracts pollinators (or dispersers) and protects the seed from predators and harsh environmental conditions.²⁴

It is currently possible to observe the effect human beings have had on how certain plants grow. For example, large forest areas have been dedicated exclusively to wood harvesting. Agriculture has transformed wild plants into new varieties that are more edible and palatable. Consequently,

most of the residues that human activities left behind consist of biomass from gymnosperm and angiosperm plants. Conifers (*Pinophytes*), like pines and spruces, are examples of gymnosperms used to obtain what are known as softwoods. On the other hand, monocotyledons (like grass, maize, sugar cane, bamboo, rice, water hyacinth, pineapple, banana, açai, and yams) and dicotyledons (like olive trees, carrots, coconuts, apples, oranges, tomatoes, strawberries, sunflowers, and roses), which pertain to the angiosperm clade, have been used to produce most foods and feeds, hardwoods from dicot trees, and energy by incinerating the remaining biomass. Thus, the abundance of specific plant groups that have been affected by human interference has defined and restricted the substances found in vegetable biomass and, consequently, the yields of possible byproducts that can be accessed and retrieved.

[Figure 3\(a\)](#) shows a graphical representation of the cell-wall composition of dicotyledonous and nongramineous monocotyledonous plants. [Figure 3\(b\)](#) also gives a visual representation of the cell-wall composition of gramineous monocotyledonous plants and gymnosperms. In [Fig. 3](#), the first structures, located outside epidermal plant cell walls, are cuticles, which are made of a hydrophobic polymer matrix that contains cutin, a polyester of hydroxy and epoxy fatty acids (C16 or C18 chains, or both), and cutan, a wax composed of several aliphatic and aromatic compounds.³⁰ After that, the middle lamella is found as an interface between neighbor cells, allowing the passing of intercellular signals, nutrients, and gases.³¹ Following that, it is possible to observe the primary cell wall supported by cellulose and kept together by crosslinked glycans. Hemicelluloses link together cellulose microfibrils but avoid direct contact with them.³¹

Depending on how those crosslinks occur, it is possible to classify primary cell walls. Type I primary cell walls contain a large amount of pectin, which surrounds cellulose (glucan) and hemicellulose (xyloglucan).^{24,27,32} This type of cell wall can be observed in dicot plants and non-gramineous monocots, mostly in their edible tissues.^{23,24} In this kind of cell wall, pectin binds through calcium bridges (low esterification) and hydrophobic bonds (high esterification), which also crosslink with phenolics and plasma membrane proteins.^{24,32} Cellulose and hemicellulose provide rigidity to the cell wall and are intertwined with pectin polymer, which provides fluidity but is stabilized by phenolic compounds and proteins.^{24,31}

On the other hand, Type II primary cell walls are common in gramineous monocotyledons (grasses) and gymnosperms.^{23,24} This type of cell wall contains lignin, which embeds cellulose microfibrils and hemicellulose in the form of xylans (such as glucuronoarabinoxylan,

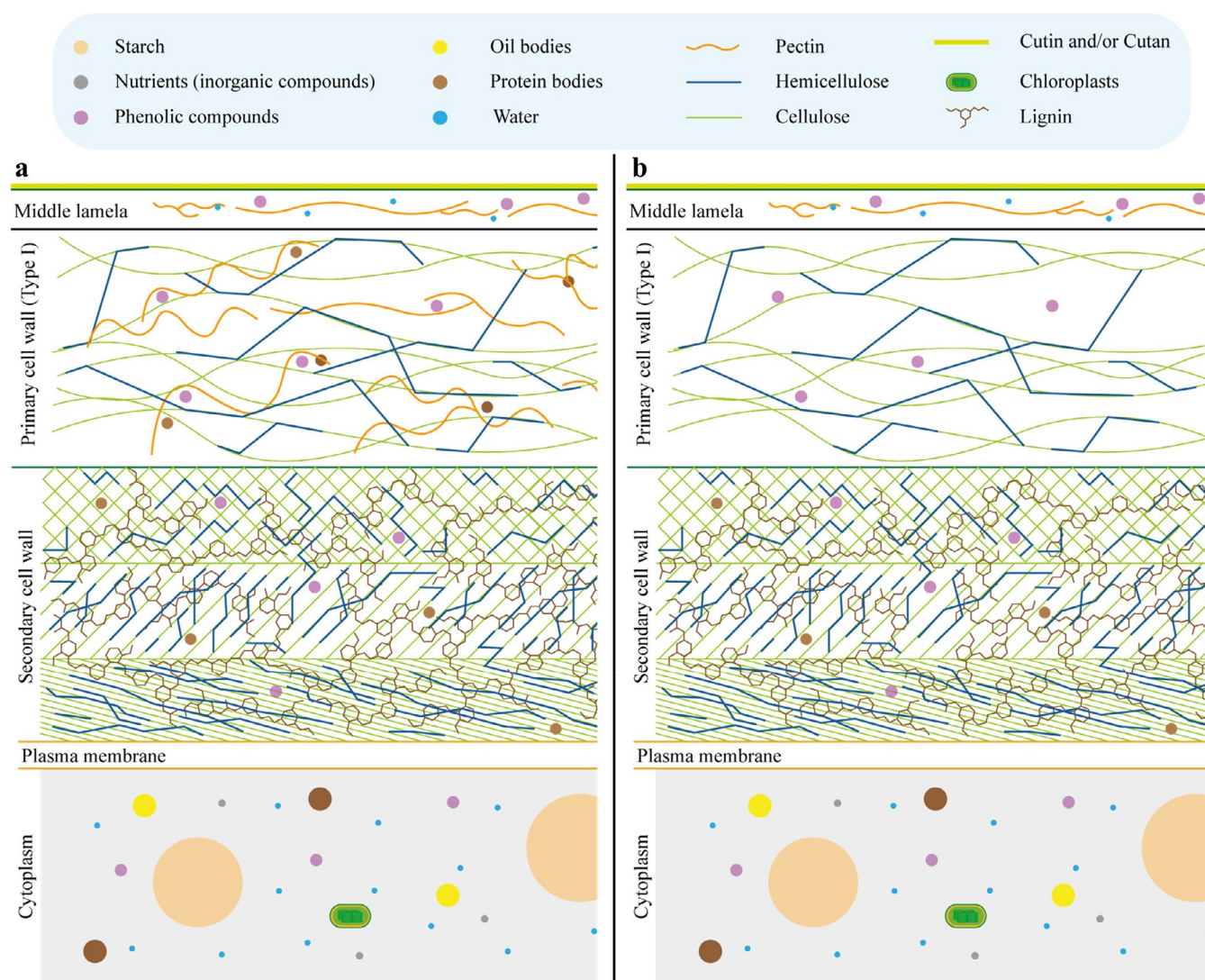


Figure 3. Graphical representation of the cell-wall composition of (a) dicotyledonous and nongramineous monocotyledonous plants and (b) gymnosperms and gramineous monocotyledonous plants.^{24,25,27–29}

arabinoxylan, and glucomannan).^{24,32} Pectin and structural proteins are absent in this cell wall, as represented in Fig. 3(b). It is important to note that the main hemicelluloses are xyloglucans, xylans, mannans, and glucomannans in dicots. Arabinoxylans predominate in monocots (wheat, barley, and grasses).³¹

Another structure that can be seen in Fig. 3 is the secondary wall. This is composed mainly of cellulose, hemicellulose, and lignin.²³ The cellulose in secondary walls is arranged in different layer configurations of the microfibrils embedded in lignin.²³ These cell walls are deposited in specialized tissues such as the xylem and the sclerenchyma, which help transport water and give structural strength to the plant.^{27,32} It is evident then that the term 'lignocellulose' could be regarded as an overgeneralization that does not represent the variety

observed in plant taxonomic groups. Instead, it tends only to represent fairly the overall composition of gymnosperms and nongramineous monocots.

For the lignification process to happen, lignin precursors (i.e., monolignols such as *p*-coumaroyl alcohol, coniferyl alcohol, sinapyl alcohol, and caffeoyl alcohol) cross cell membranes using mostly passive transport mechanisms and are deposited in plant secondary walls giving strength.^{27,28,32} Apart from monolignols, other substances such as enzymes, sugars, organic acids, and most polyphenols (dimeric phenolics and flavonoids) can cross cell membranes.²⁸ Passive transport occurs through the ~5 nm pores of guard cells, limiting the kind of molecules that can permeate.²⁴ These bioactive compounds are produced inside cell walls and are stored in vacuoles and organelles in the form of starch

granules (10–100 µm), oil bodies (0.5–5 µm), protein bodies (0.5–5 µm), carotenes, and chlorophyll inside the cell wall of chloroplasts (3–5 µm),³³ as shown in Fig. 3. It is clear that cell walls impede the release of molecules for energy storage and energy production, such as lipids (e.g., oils, fats, and terpenoids) and starch, which are vital for survival.

The only way to access these substances is to divide them into smaller structures capable of passing through the cell or releasing them by applying external forces that break cell walls. For example, starch gelatinization would require thermal and mechanical treatments (milling or grinding), which cause cell wall rupture.²⁴ On the other hand, phenolic compounds such as flavonoids and phenylpropanoids, mainly located in central vacuoles, can travel along primary and secondary walls due to their small size, and can be found even in the external waxes (cuticles) and accumulate in trichomes.²⁹ These bioactive compounds help plants to protect themselves from biotic (predators) and abiotic (radiation, pollution, heavy metals) sources of stress, send signals to other plants, create symbiotic relationships with other organisms, and attract insects (pollinators and dispersers).²⁹ In Fig. 3, it is possible to see that phenolic compounds are present in both primary and secondary cell walls between the cellulose fibrils.³² Consequently, the heterogeneous composition of vegetable biomass requires systematic, standardized, and comprehensive characterization to understand and quantify their true potential as feedstocks for biorefineries. Knowing the exact amounts of relevant compounds is key guidance for setting realistic expectations, designing processing routes, and selecting valuable and marketable products.⁴

Characterization methods for compositional analysis of fruit wastes

Multiple methods have been reported in the literature for the compositional analysis of FW, some of which are standardized. These standardized methods and the contributions made by several authors have been described by Sluiter *et al.*¹⁶ Although most of those methods were initially developed for lignocellulosic feedstocks and industries (agriculture, biomass, and papermaking), they have also been slightly adapted or used directly for the quantitative characterization of FW, which might significantly differ from conventional lignocellulosic biomass in terms of their composition (mainly in the contents of pectin, extractives, and starch) and physical–chemical properties. Table 1 shows a summary of standardized methods (i.e. validated with inter-

laboratory studies), reported in the literature, which can be used for the compositional analysis of FW. However, due to the structural differences between lignocellulosic biomass and FW, the direct application of these characterization methods into FW may lead to possible data gaps (e.g., incomplete characterization) and/or inconsistencies across methods, especially determining and quantifying extractives, pectin, and starch. The following subsections briefly describe the methods that have been used to obtain FW's composition.

The National Renewable Energy Laboratory – laboratory analytical procedures

The National Renewable Energy Laboratory (NREL) developed standardized methods^{54,56,58,60,61} to analyze the chemical composition of lignocellulosic biomass, driven by growing interest in producing biofuels and biochemicals. These methods assess biomass potential as a carbon source for holistic utilization via catalytic conversion, thermochemical processes, or fermentation. The main goal is to achieve a comprehensive description of the constituents using the concept of summative mass closure (SMC). This approach ensures that the total measured substances add up to 100%, with a maximum allowable variation of ±5% between different laboratories.⁶² The NREL is the most commonly used method for FW as it provides the most accurate description of the composition due to its quantitative nature, which can also be coupled with spectral data from near-infrared (NIR) spectroscopy to form predictive regression models. This is still under development.^{63,64} One advantage of this method is the use of a standard sample preparation procedure that guarantees the same pretreatment conditions for all the samples, which helps to assure the replicability of these protocols compared to other methods (Table 1).

The NREL method uses water and ethanol to measure water-soluble carbohydrates (WSC), polyphenols, waxes, fats, resins, gums, sterols, and nonvolatile hydrocarbons. However, the extraction of these substances from the sample may be incomplete for samples with high nonpolar compound content.⁵⁸ Structural monosaccharides are identified by acid hydrolysis and measured by high-performance liquid chromatography (HPLC). The latter has the advantage of including a set of sugar recovery standards (SRS) that consider possible losses due to over-hydrolysis.¹⁶ It is important to note that the NREL recently published a protocol for starch quantification based on enzymatic hydrolysis and uses gravimetric and chromatographic measurements for that purpose.⁶⁵ However, starch and pectin have not been historically quantified in the NREL

Table 1. Characterization methods of lignocellulosic biomass that have been used for FW.

Type of method	Methods			
	NREL	TAPPI [34]	Van Soest [35–38]	AOAC [39–53]
Sample preparation	Quantitative Dried at 45 °C (moisture <10%), milled to 1 mm mesh [54]	Semiquantitative Milled to a 0.4 mm mesh. Samples must be wood (T 257) or pulp (T 210)	Semiquantitative Samples homogenized to 1 mm mesh. Extractives, proteins, and starch are removed from homogenized samples	Semiquantitative Industrial grinders, blenders, or food processors are used to obtain a homogeneous mixture
Total solids	Dried at 105 °C until constant weight [55]	Dried at 105 °C until constant weight. For samples of wood (T 264), pulp (T 210), paper, or paperboard (T 550).	Dried at 100 °C	Dried at 100 °C [37]
Ash	Ignition at 575 °C using a muffle ramp [56]	Ignition at 525 °C (T211)	Ignition at 525 °C	Ignition of the sample (525–600 °C) depending on the type of sample (Table S2)
Protein	Kjeldahl method [57]	Not measured	Kjeldahl method	Kjeldahl method, with different variations depending on the type of sample (Table S2)
Extractives	Two-stage Soxhlet extraction (water and ethanol). Water soluble carbohydrates in water measured by HPLC. Ethanol extracts are measured gravimetrically [58]	Soxhlet extraction with a mixture of ethanol and benzene (1:2 v/v), dichloromethane, or acetone. Extracts are measured gravimetrically (T 264 or T 204).	Not specified. Usually obtained by using water and other solvents (petroleum ether mostly)	Crude fat determined by Soxhlet extraction with petroleum ether
Structural carbohydrates	Two-stage acid hydrolysis using H ₂ SO ₄ . Stage 1: 30 °C, 72% w/w H ₂ SO ₄ . Stage 2: 121 °C, 4% H ₂ SO ₄ . [59] Hydrolyzed sugars are measured by HPLC and used to calculate glucan, xylan, galactan, arabinan, and mannan	T 203: Alpha-cellulose (NaOH 17.5% w/w and 9.45% w/w at 25 °C), beta-cellulose (potassium dichromate), and gamma-cellulose (by difference) T 223: Pentosans obtained by boiling samples in HCl 3.85 M. Furfural is collected on the distillate and determined using the orcinol-chloride reagent T 249: Two-step acid hydrolysis to obtain monomeric sugars that are converted to alditol acetates that can be measured using gas chromatography	TDF is treated with a neutral detergent solution (sodium lauryl sulfate, decahydronaphthalene, and sodium sulfite) that removes noncell wall polysaccharides in a refluxing apparatus that allows obtaining the amount of neutral detergent fiber (NDF) NDF is treated with an acid detergent solution (H ₂ SO ₄ 0.5 M, cetyltrimethylammonium bromide, and decahydronaphthalene) that removes hemicellulose and allows the determination of acid detergent fiber (ADF)	It is possible to measure any of the following fractions depending on the version of the protocol (Table S2): TDF, NDF, insoluble dietary fiber (IDF), soluble dietary fiber (SDF), ADF, ADL. Total carbohydrates measured by difference
Lignin	Determined gravimetrically after acid hydrolysis. Corrected using protein content [59]	Determined gravimetrically after acid hydrolysis (T 222)	Determined gravimetrically after the acid hydrolysis of the ADF fraction (cellulose and lignin), which leaves behind acid detergent lignin (ADL)	Determined gravimetrically after the acid hydrolysis of the ADF fraction (cellulose and lignin), which leaves behind acid detergent lignin (ADL)

(Continues)

Table 1. (Continued)

	Methods			
	NREL	TAPPI [34]	Van Soest [35–38]	AOAC [39–53]
Starch	Not usually measured. NREL published a recent protocol to measure starch based on AOAC methods that employ enzymes to hydrolyze starch but improved by using HPLC and gravimetric quantification of the fractions	Not measured	Not measured	Measured in AOAC methods since (YEAR)
Pectin	Not measured	Not measured	Van Soest recommended a procedure using metahydroxybiphenyl to measure galacturonic acid [37]	Not measured
Advantages	Samples are prepared in uniform conditions The ash ramp reduces sample losses Water-soluble carbohydrates are quantified from extracts SRS correct sugar content and consider over-hydrolysis Measured fractions of structural monosaccharides can be related to cellulose and hemicellulose contents	Useful to measure pulp quality (alpha, beta, and gamma cellulose). Useful to determine yields of paper and other derived materials	Useful to determine fibrous and nonfibrous fractions Removes extractives and starch to avoid possible interferences with fiber measurements Proposes a method to measure pectin	Useful to determine digestible and nondigestible fractions Relevant to obtain nutritional information Removes extractives and starch to avoid possible interferences with fiber measurements It tends to include a measurement of resistant and nonresistant starch, depending on the version of the method (Supporting Information, Table S2)
Disadvantages	It has problems with samples with a high amount of nonpolar extractives Even though polar and nonpolar extractives are obtained, only WSC are identified It does not consider the quantification of starch and pectin Starch could cause an overestimation of the cellulose content	It was explicitly designed for wood, pulp, paper, and paperboard samples. Variations exist in sample preparation depending on whether the sample is wood, pulp, or paper Protein is not measured, which could result in overestimating the lignin content. Extractives are not identified and are removed only to facilitate other tests Measurement of alpha, beta, and gamma cellulose is semiquantitative and is only valid for applications related to the paper industry Loss of sugars due to over-hydrolysis is not considered in T249 The selection of the method to measure structural carbohydrates is arbitrary. It does not consider the quantification of starch and pectin	It was designed specifically to determine the digestibility of forages The preparation of samples is not clearly specified and could be performed using different equipment Extractives are not usually quantified It does not consider the quantification of starch The quantification of the TDF and NDF fractions is semiquantitative and cannot be directly correlated with the cellulose, hemicellulose, and lignin contents	It was designed specifically to determine the digestibility of forages The preparation of samples varies depending on the type of sample (Table S2) and could be done using different equipment, particle sizes, and initial moisture contents Quantification of total carbohydrates or WSC is obtained by difference from the measured fractions The quantification of the TDF, NDF, ADF, IDF, and SDF fractions is semiquantitative and cannot be correlated directly with the cellulose, hemicellulose, and lignin content It does not consider the quantification of pectin

Laboratory Analytical Procedures (LAPs) (Table 1), which is a clear disadvantage that could lead to inaccurate results and measurements in FW due to starch, pectin, and nonpolar extractives in those samples. For instance, starch may cause an overquantification of cellulose due to its hydrolyzation into glucose which could be detected and measured as glucan in this protocol. Depending on the concentration of pectin in the samples, some could remain after acid hydrolysis and could interfere with lignin measurements. Finally, remaining nonpolar extractives (fats and oils) might cause incomplete hydrolysis of structural carbohydrates because they also react with sulfuric acid.

The Technical Association of the Pulp and Paper Industry methods

As part of the primary goal of the pulp and paper industry to improve constantly the production yields of high-quality and high-strength pulp (i.e., bleachability and delignification), the Technical Association of the Pulp and Paper Industry (TAPPI) has developed the sector's guidelines, standards, and methods for the compositional analysis of wood, pulp, and paper.³⁴ The highly specific development of methods and standards for these three feedstocks is very positive for the pulp and paper sector as they can be adjusted for the intended purpose; however, their applicability is limited in other industries and feedstocks. The TAPPI methods do not follow a fixed standard sample preparation procedure. Instead, the sample preparation method is decided according to feedstock type (wood, pulp, or paper). Hence, extending the TAPPI methods to other feedstocks, such as lignocellulosic biomass and FW, would most likely lead to conflicting methods and, consequently, inconsistent results regarding extractives and the quantification of structural carbohydrates.

For example, Soxhlet extractions are applied as a preparatory step for the samples before all tests regarding pulp quality (including samples of wood, pulp, and paper). Thus, all extractives are measured gravimetrically, with no differentiation between polar and nonpolar substances.³⁴ The analysis of structural carbohydrates is performed in a semi-quantitative manner by identifying alpha, beta, and gamma cellulose (recommended for bleached or delignified pulps only), which are helpful in papermaking but are inaccurately associated with the actual the amounts of cellulose and hemicellulose. This is because structural carbohydrates are measured by determining the fractions that are either soluble or insoluble under a series of pH variations, with the analysis aimed at distinguishing between the amorphous and crystalline regions of cellulose. The aim of this method is to provide useful information for the pulp and paper industry,

so TAPPI recommends its use only for bleached or delignified wood pulps.⁶⁶ Other approximations have been proposed by TAPPI, including measuring pentosans and monomeric sugars. These are similar to the NREL-LAPs but without including SRS, resulting in underestimating sugars by not considering possible losses during hydrolysis.

Contrary to the recommendations made by TAPPI,³⁴ these methods have been used to measure the content of extractives, structural carbohydrates, and lignin in FW,^{67–76} where interferences in the matrix during estimations may occur due to the presence of pectin and starch (which are not measured). Even though some analyses (e.g., total solids, ash, and lignin) are performed quantitatively, it is possible that the sum of all measurements might not result in a detailed and complete description of the composition using TAPPI methods. Some reports fail to achieve a complete SMC (~100%),^{67,68,73–75,77–79} and in the cases when a complete SMC has been achieved, the measurement of the protein content of the samples is missing.^{69–72} It is important to note that this method is the only one that does not include procedures to measure protein (Table 1).

The Van Soest method

The agricultural industry has aimed to describe forage digestion and its effects on animals' nutrition. Van Soest (in 1963) established a baseline of methods to quantify feeds' composition,³⁵ which have been modified over the years until 1991.³⁷ The methods focus on the compositional analysis in terms of the physical and biological properties relevant to the dietary balance of monogastric species and ruminants, which includes the study of forages and starchy foods and feeds.³⁷ Although the Van Soest method was the first approximation to determine fibrous and nonfibrous fractions of feedstocks, the same author emphasized (in the 1991 publication)³⁷ that this method was obsolete and should be regarded as a historical piece from which future procedures could be developed and improved. Nonetheless, several authors until this day continue to use Van Soest's procedures to measure a wide variety of feedstocks, including foods,⁸⁰ feeds,^{81,82} forages,⁸³ and wastes.⁸⁴ In this method, samples are prepared by removing extractives, proteins, and starch, which are not quantified. The remaining fraction is called total dietary fiber (TDF) or crude fiber (CF) which consists of cellulose, hemicellulose, lignin, gums, β -glucans, pectin, and resistant starch.^{36,38} From this fraction, it is possible to determine the neutral detergent fiber (NDF) (mostly cellulose, hemicellulose, and lignin) and the acid detergent fiber (ADF) (cellulose, and lignin) using detergent solutions (Table 1). In all cases, the fiber fractions are used to calculate the difference

between the amount of cellulose, hemicellulose, and lignin; however, this way might be inadequate, as reported by Sluiter *et al.*,⁸⁵ who demonstrated that the gravimetric quantification of the TDF, and NDF fractions is semiquantitative and cannot be correlated directly with the cellulose, hemicellulose, and lignin content. Another complication with this method is that it does not specify a standard sample preparation procedure, making comparisons between different authors difficult. Nonetheless, since Van Soest suggested a method for pectin quantification (Table 1), a first approximation to measuring all the soluble and nonsoluble fiber constituents was made, which could be helpful when describing FW.

Official methods of analysis of the Association of Official Agricultural Chemists

The contributions made by Van Soest to the agricultural industry resulted in collaboration with the Association of Official Agricultural Chemists (AOAC) to produce several AOAC Official Methods that have been continuously updated until the present day.⁸⁶ The AOAC Official Methods include the study of forages, starchy foods, and feeds to identify relevant substances to mammalian digestibility. In the AOAC methods (Table 1), samples are prepared with food processors (grinders or blenders) to obtain a homogeneous mixture from which crude fats can be determined by Soxhlet extraction with petroleum ether. Total carbohydrates and WSC are calculated indirectly using the other measured components. In the AOAC methods, neither pectin nor WSC are measured, even though they are nutritionally relevant. The solid residue after the petroleum ether extraction can be used to quantify starch, crude protein (Kjeldahl method), TDF, NDF or Insoluble Dietary Fiber (IDF), Soluble Dietary Fiber (SDF), ADF, acid detergent lignin (ADL), and ash. The quantification process of the fiber fractions is accomplished by using different enzymes (heat-resistant and pancreatic amylases) that enhance starch removal. Some versions of the AOAC methods allow the quantification of starch in terms of resistant and nonresistant starch, which gives valuable nutritional information about the feedstocks (more information about the specific methods and their versions can be found in Supporting Information, Table S2). Different versions of these procedures have been published (the oldest version available is from 1984 and the most recent one was published in 2019), and authors have been using both oldest and updated versions of the methods, which increases the uncertainty of the comparisons that can be made. However, AOAC methods provide a useful approach to measuring starch in the composition of FW (mainly pomaces and seeds),

enabling differentiation between glucose derived from this polymeric carbohydrate and that produced by acid hydrolysis of cellulose. A summary of the specific AOAC methods found in this review is presented in Supporting Information, Table S2.

Existing characterization methods

In general terms, the TAPPI, AOAC, and Van Soest methods are primarily semiquantitative protocols that were initially developed to provide the compositional analysis data of very particular biobased industries. However, they were never intended to characterize broader vegetal biomass types, which became a common trend in the scientific literature.⁶² It is important to note that neither of these procedures proposes methodologies to identify the nature of the extracted compounds (apart from the measurements of WSC of the NREL). Even though NREL procedures aim to explain all the biomass components quantitatively, there are possible errors when these protocols are used to measure complex feedstocks due to the presence of extractives, pectin, and starch. This is particularly relevant when the composition of the biomass varies considerably from the reference materials used to standardize each of the procedures, which are primarily conventional lignocellulosic feedstocks (wood and pruning residues). Nevertheless, valuable proposals to measure pectin and starch made in the Van Soest and AOAC methods could be used to enhance NREL procedures in a way that is useful for the characterization of FW.

Several authors have reported the composition of FW using the methods mentioned above. However, it is necessary to be careful when using these data, given each method's limitations and disadvantages, as mentioned before and shown in Table 1. This information could be useful to provide an initial idea of the possible substances present in samples of FW but it would be insufficient and inadequate to use these characterization data in biorefineries. The following section will present a collection of compositional data reported in the literature gathered during the systematic review process.

Composition of fruit wastes as reported in the literature

The composition of FW, gathered from literature, is organized and displayed in Table 2 according to the NREL protocols, in Table 3 for TAPPI, and in Tables 4–6 for the AOAC and Van Soest methods (for peels, seeds, and mixed residues, respectively). The information for most NREL methods was related directly to chemical, biochemical and thermochemical valorization platforms. On the other hand, data gathered

Table 2. Reported composition of fruit wastes for the NREL protocols (dry weight basis).

Fruit waste	Cellulose	Hemicellulose	Lignin	Pectin	Extractives	Protein	Ash	SMC	Reference
<i>Peels</i>									
Avocado peel	29.76±1.18	27.30±1.24	4.72±0.13	NR	37.10±0.34	NR	1.12±0.05	100±2.94	[87]
Banana peels	52.43±2.81	37.19±1.64	7.74±0.93	NR	7.26±1.58	NR	1.03±0.17	105.65±7.13	[88]
Coffee pulp and outer skin	23	20	22	NR	16	NR	19	100	[89]
Musambi peel	25.4	9.4	23.6	17.1 ^a	NR	NR	NR	75.5	[90]
Orange peel	27.14±0.81	14.88±1.00	18.5±2.45	18.50±2.45 ^b	19.15±1.31	6.89±0.13	3.71±0.32	108.77±8.47	[91]
Orange peel	21.23±1.20	12.08±0.42	14.77±1.75	23.02±1.40 ^b	5.56±0.37	6.74±0.20	3.19±0.25	86.59±5.59	[92]
Orange peel and pulp	18.6±0.1	14.3±0.2	6.5±0.6	18.6±1.9 ^c	38.0±0.5	NR	3.7±0.1	99.7±3.4	[93,94]
Pineapple peel	20.9±0.6	31.8±1.9	10.4±1.0	NR	28.1±2.5	3.9±0.2	5.9±0.06	101±6.26	[95]
Pineapple peel	20.15±1.64	29.39±2.13	6.35±0.28	NR	NR	NR	5.05±0.10	60.94±4.15	[96]
Pomegranate peels	19.40±0.2	13.51±0.3	24.48±0.3	29.10±0.2 ^d	5.08±0.1	4.62±0.5	3.81±0.1	100±1.6	[97]
Pomegranate peels	19.72	14.56	25.22	25.34 ^d	6.08	5.27	3.81	100	[98]
<i>Seeds/seed vessels</i>									
Açaí seed	43.81±3.39	25.89±2.49	24.56±0.58	NR	7.71±0.06	5.27±0.14	1.18±0.04	108.42±6.7	[99]
Açaí seed	13.05±1.46	42.67±1.81	15.91±6.71	NR	22.31±0.51	NR	7.54±0.11	101.48±10.6	[100,101]
Annatto seed	18.81±0.73	11.34±1.20	13.92±0.35	16.00±0.04 ^b	28.46±1.91	8.71±0.39	5.39±0.07	102.63±4.69	[100,101]
Avocado seed	6.97±0.38	51.50±2.14	1.93±0.04	NR	38.67±1.95	NR	0.94±0.06	100±4.57	[87]
Castor seed cake	19.0±0.3	17.9±0.7	41.6±0.6	NR	21.1±1.3	NR	NR	99.6±2.9	[102]
Coconut fiber/husk	25.61±0.43	23.48±0.20	32.22±2.39	NR	NR	NR	NR	81.31±3.02	[103]
Coffee ground (spent)	9.78±0.7	47.82±1.6	11.37±0.2	NR	14.56±0.1	14.67±0.1	1.81±0.1	100±2.8	[104]
Coffee ground (spent)	12.40±0.79	39.10±1.94	23.90±1.70	NR	2.29±0.30 [*]	17.44±0.10	1.30±0.10	96.43±4.93	[105]
Coffee husk (parchment)	29.17±1.51	28.96±2.44	22.35±0.96	NR	17.67±1.98	NR	4.6±0.47	102.75±7.36	[106]
Coffee silverskin	23.77±0.09	16.68±1.30	28.58±0.46	NR	3.78±0.40 [*]	18.69±0.10	5.36±0.20	96.86±2.55	[105]
Cherimoya seed cake	22.05	17.78	25.63	NR	2.15	29.53	2.84	99.98	[107]
Hazelnut seed	15.4±1.5	11.3±0.6	26.2±0.7	10.6±2.4 ^e	24.6±1.7	8.0±0.2	5.0±0.3	101.1±7.4	[108]
Hazelnut shell	18.7±0.5	23.2±0.7	46.7±0.2	5.3±1.7 ^e	1.2±0.3	2.8±0.1	0.9±0.1	98.8±3.6	[108]
Olive stone	21.10	31.43	40.88	NR	6.04	NR	0.55	100	[109]
Olive stone	20.10	29.92	38.87	NR	10.54	NR	0.57	100	[110]
Peach stone	17.6±2.0	16.2±0.1	45.0±3.6	NR	2.8±0.1	NR	1.2±0.3	82.8±6.1	[111]
Walnut endocarp	20.9±1.1	16.2±0.6	45.4±1.2	NR	7.1±0.2	NR	0.6±0.0	90.2±3.1	[111]
<i>Pomace and mixed residues</i>									
Andean blackberry pulp (spent)	43.99±0.46	20.87±1.39	20.26±1.65	NR	13.68±1.18	NR	1.20±0.01	100±4.69	[112]
Apple pomace	22.71	15.79	19.80	NR	18.16	5.21	1.40	83.07	[113]

Table 2. (Continued)

Fruit waste	Cellulose	Hemicellulose	Lignin	Pectin	Extractives	Protein	Ash	SMC	Reference
Apple pomace	21.0 ± 1.0	11.1 ± 0.9	24.7 ± 0.1	14.4 ± 0.6 ^c	21.1 ± 1.3	NR	2.2 ± 0.1	94.5 ± 4.0	[114]
Grape pomace	8.04 ± 0.42	8.2 ± 0.19	44.46 ± 0.02	NR	28.62 ± 1.54	12.68 ± 0.12	4.52 ± 0.07	106.52 ± 2.36	[115]
Papaya waste	6.3 ± 0.1	1.5 ± 0.1	5.1 ± 0.1	NR	77.6 ± 2.9	4.6 ± 0.4	7.6 ± 0.2	102.7 ± 3.8	[116]
Tomato pomace	7.66	7.51	37.34	NR	36.02	NR	NR	88.53	[117]

Note: Methods reported for pectin quantification: ^aSudhakar and Maini, ¹¹⁸ ^bBitter and Muir, ¹¹⁹ ^cQuantification using HPLC. ^dRosli et al., ¹²⁰ ^eMelton and Smith, ¹²¹

Abbreviations: NR, not reported; SMC, summative mass closure.

*Fat content was measured using Soxhlet extraction with petroleum ether for 1 h, according to the AOAC method no. 920.39.⁴⁶

reporting the TAPPI, Van Soest, and AOAC methods were used in applications associated with the elaboration of materials, foods, and feeds, limiting the area of application of the analyses reported to those particular industries. A decision matrix (Table 7) was included by the authors to show the convenience of the methodologies discussed for quantifying the compounds of FW and how they could complement each other. The decision matrix evaluated each method's ability to quantify the substances present in biomass by assigning scores on a scale from 0 to 10 based on their performance.

Fruit waste was classified into peels, seeds or seed vessels, and pomace or mixed residues. Such waste has a high total carbohydrate content (61% to 97% in peels, 17% to 94% in seeds and seed vessels, and 52% to 92% in pomace and mixed residues) in dry weight. From these carbohydrates, pectin represents up to 29% of the composition of peels, 11% in seeds and seed vessels, and 15% in pomace and mixed residues. Fiber could represent from 60% to 75% of the total carbohydrates, depending on the kind of FW. The composition of the remaining FW consists of extractives (0.1% to 38% in peels, 1% to 72% in seeds and seed vessels, and 0.1% to 78% in pomace and mixed residues), protein (0.1% to 13% in peels, 3.5% to 46% in seeds and seed vessels, and 0.35% to 18% in pomace and mixed residues), and ash (1% to 24% in peels, 0.5% to 8% in seeds and seed vessels, and 0.5% to 8.5% in pomace and mixed residues). The ranges listed for each substance were obtained from the values in Tables 2–6. It is important to note that the composition of pomace is highly variable and depends on the extraction processes and the nature of the biomasses (i.e., the proportions of peels, seeds, seed vessels, and remnant pulp in these mixtures).

Total carbohydrates and WSC

It is important to note that authors using NREL procedures do not explicitly report the WSC content (sucrose, lactose, glucose, fructose, and galactose) explicitly, as seen in Table 2. However, it is included as part of the total extractives content. The NREL procedures do not have a method to quantify pectin and starch. In this method, starch is hydrolyzed and quantified as glucan, which could cause an overestimation of cellulose content.⁶⁵ Pectin is not included in the NREL procedures but some authors have employed different methodologies, reported in the literature, to obtain approximate content.

In the case of the TAPPI methods, as the presence of WSC, starch, and pectin are not expected in wood and pulp, they are not determined. This is a concerning point

Table 3. Reported composition of fruit wastes for the TAPPI methods (dry weight basis).

Fruit waste*	Cellulose	Hemicellulose	Lignin	Pectin	Extractives	Protein	Ash	SMC	Reference
Açaí seed	17.74±0.11	56.55±0.36	16.78±0.10	NR	NR	NR	NR	91.07±0.57	[75]
Almond shells	18.19±0.19	35.99±1.23	31.24±0.29	NR	3.11±0.32 ^a	NR	0.81±0.09	89.34±2.12	[67]
Almond shells	23.7 ^a	31.2 ^a	28.8	1.4 ^a	5.7 ^b	NR	0.7	91.5	[68]
Argania nutshells	48.10	7.56	34.58	NR	NR	NR	0.54	90.78	[77]
Banana peel	15.80	14.57	12.33	NR	57.3	NR	7.24	107.24	[76]
Cactus seeds	27.2	0.01	20	NR	NR	NR	3.28	50.49	[74]
Cactus seeds	27.17±2.33	0.01±0.00	37.25±3.18	NR	NR	NR	3.28±0.39	67.71	[79]
Date pits	21.2	28.1	19.9	NR	NR	NR	NR	69.2	[78]
<i>Jatropha curcas</i>	55.52	16.88	21.38	NR	NR	NR	6.22	100	[69]
Olive pomace	13.8 ^a	22.2 ^a	31.2	0.5 ^a	34.4 ^b	NR	7.3	109.4	[70]
Olive stones	15.3 ^a	29.4 ^a	42.1	1.1 ^a	13.7 ^b	NR	0.6	102.2	[70]
Peach pits	56.7 ^c (24.7 ^d)		39.6	NR	3.5	NR	1.5	101.3	[71]
Pineapple waste	41.48	36.2	13.22	NR	NR	NR	6.05	96.95	[73]
Pineapple peel	42.14	22.88	11.08	NR	23.9	NR	9.18	109.78	[76]
Walnut shell	47.78 ^c (26.51 ^d)		49.18	NR	NR	NR	2.13	99.09	[72]
Walnut endocarp	21.7 ^a	24.7 ^a	29.9	3.3 ^a	10.6 ^b	NR	0.7	90.9	[67]

Abbreviations: NR, not reported; SMC, summative mass closure.

^aDetermined by HPLC or GC from structural monosaccharides produced in acid hydrolysis (TAPPI T 249): cellulose (glucose), hemicellulose (xylose, mannose, galactose, arabinose, and acetyl groups), pectin (rhamnose, galacturonic acid, and glucuronic acid).

^bSoxhlet extraction with dichloromethane, ethanol, and water.

^cHolocellulose.

^dCellulose.

^eEthanol-toluene extractives (TAPPI T244-om-93).

*Apart from olive pomace, pineapple waste/peel, and banana peel, the rest of the wastes presented here are seeds and seed vessels.

considering that all biomasses in Table 3 could have some pectin (measured as galacturonic acid, glucuronic acid, and rhamnose in TAPPI T 249³⁴) and starch content.

The content of WSC could contribute significantly to the total composition, with values reaching up to 82% in peels, 93% in seeds and seed vessels, and 92% in pomace and mixed residues (ranges obtained from Tables 4–6). However, the exact amount of WSC, as described in the AOAC methods, is unknown because this value is calculated straightforwardly and is not quantified⁴⁵ by discounting crude fat, crude protein, and ash from the total weight of the sample.

Even though total carbohydrates and WSC represent most of FW composition, all procedures fail to describe them thoroughly. The WSC content in the NREL procedures is considered as part of the extractives, in the TAPPI methods it is not determined, and in the AOAC methods it is not measured but obtained indirectly. This fraction is very interesting for biofuels, materials, food and feed products, and other applications. Based on the NREL procedures, it would be convenient to quantify WSC using Soxhlet extraction and HPLC, due to its abundance in FW and its potential uses. To describe FW fully, it would be necessary to

evaluate the effect of the extraction time using samples with a large amount of WSC (i.e., samples where pulp leftovers are abundant), which would be an improvement on the existing NREL procedure.

Quantifying starch is fundamental for an accurate representation of its composition – a challenging task considering that, in the NREL and TAPPI methods, this substance is usually not measured and it could also interfere with cellulose measurement.⁶⁵ In the case of the AOAC methods, Tables 4–6 show that the starch content is reported only in three studies,^{175,184,193} which do not differentiate between resistant and nonresistant starch, essential to understand how starch is digested (in food and feed products). Nonetheless, most versions of the AOAC standards published since 1995 include procedures for starch removal and its colorimetric quantification before fiber measurements.^{45,52} Although most of the investigated studies using AOAC methods include treatments with amylases and amyloglucosidases to remove starch, its content is not reported in most cases which is counterintuitive because the AOAC methods are focused in food and feed products. The starch content was reported only for Juçara seed (~13%), grape pomace (2.3%), and apple pomace (0.2%).^{175,184,193}

Table 4. Reported composition of fruit wastes (peels) for the AOAC and Van Soest methods (dry weight basis).

Fruit waste	TC	WSC	STA	CF/TDF	SDF	PEC	IDF/NDF	HEM	ADF	CEL	LIG	CFat/EE	CP	Ash	SMC	Reference
Achachairú peel ^h	77.61 ± 0.07	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	8.89 ± 0.07 ⁷	9.52 ± 0.10	3.98 ± 0.03	100 ± 0.27	[122]
Avocado peel ^f	61.12 [*]	7.98 ± 0.66	NR	53.14 ± 0.17	NR	NR	NR	NR	NR	NR	NR	35.22 ± 0.58 ⁴	0.25 ± 0.01 ¹²	2.94 ± 0.05 ¹⁷	99.53 ± 1.47	[80]
Banana peel ^c	78.84 ± 4.13	23.6 [*]	NR	54.80 ± 1.56	NR	NR	NR	NR	NR	7.20 ± 0.83	NR	2.13 ± 0.03	7.72 ± 0.83	11.31 ± 3.21	100 ± 6.53	[123]
Banana peel ^f	62.09 [*]	50.13	NR	11.96	NR	NR	NR	NR	NR	NR	NR	4.85	9.34	23.72	100	[124]
Banana peel ^f	78.51 ± 0.11	21.65 [*]	NR	56.86 ± 1.16	19.39 ± 1.19	10.98 ± 1.61 ¹⁹	37.14 ± 1.34 ⁹	NR	NR	NR	NR	2.30 ± 0.12 ⁵	5.64 ± 0.27 ¹³	13.55 ± 0.11 ¹⁶	100 ± 5.8	[125]
Banana peel ^f	56.21 [*]	18.4 [*]	NR	NR	NR	13.9 ± 0.46 ²⁰	NR	9.76 ± 0.52	NR	11.97 ± 0.31	2.18 ± 0.05	29.45 ± 1.29	5.13 ± 0.02	9.21 ± 0.02	100 ± 2.67	[126]
Banana peel ^f	55.23 [*]	14.09 [*]	NR	NR	NR	15.9 ± 0.26 ²⁰	NR	10.19 ± 0.12	NR	12.17 ± 0.21	2.88 ± 0.05	29.83 ± 0.29	5.13 ± 0.01	9.81 ± 0.42	100 ± 1.36	[127]
Banana peel ^f	46.9 [*]	NR	NR	NRE	NR	NR	28.5	NR	18.4	10.6	7.8	NR	NR	NR	54.7	[128]
Banana peel ^d	66.3	29.4	NR	NR	NR	NR	35.1	20.7	23.6	14.9	8.2	7.5	12.0	16.4	102.2	[81]
Banana peel ^f	NR	NR	NR	NR	NR	NR	NR	21.56	NR	13.98	9.28	NR	NR	NR	44.82	[129]
Banana peel ^g	NR	NR	NR	19.45 ± 0.44	NR	NR	NR	NR	NR	NR	NR	1.20 ± 0.62	7.57 ± 0.64	11.84 ± 0.37	38.98 ± 2.07	[130]
Cactus pear peel ^g	95.5 [*]	24.52 ± 2.13	NR	70.98 ± 2.05 ⁹	33.93 ± 1.57 ¹⁰	NR	37.04 ± 1.66 ¹⁰	NR	NR	NR	NR	0.12 ± 0.01 ¹	0.09 ± 0.01 ¹⁵	4.29 ± 0.99 ¹⁸	100 ± 5.19	[131]
Cashew bagasse ^f	NR	NR	NR	NR	NR	NR	NR	16.2	NR	12.7	34.5	NR	NR	NR	63.4	[15]
Calá-manga peel ^g	61.57 ± 0.02	NR	NR	28.23 ± 0.02	NR	NR	NR	NR	NR	NR	NR	0.03 ± 0.0009	6.42 ± 0.02	3.75 ± 0.01	100 ± 0.07	[132]
Dragon fruit peel ^g	74.51 [*]	8.92 ± 1.15	NR	65.59 ± 1.65 ⁹	23.96 ± 1.32 ¹⁰	NR	41.63 ± 2.97 ¹⁰	NR	NR	NR	NR	1.31 ± 0.16	6.30 ± 0.18	17.56 ± 0.29	99.68 ± 3.43	[133]
Dragon fruit peel ^g	77.88	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	0.96	8.65	12.5	100	[134]
Durian peel ^f	NR	7.22	NR	NR	NR	NR	NR	19.45	NR	22.53	10.21	6.45	1.73	3.7	74.0	[135]
Feijoa peel ^g	NR	NR	NR	48.3 ± 0.0	8.1 ± 0.4	NR	40 ± 2	NR	NR	NR	NR	1.5 ± 0.3	2.27 ± 0.05	2.09 ± 0.02	54.16 ± 0.37	[136]
Grape peel ^a	77.55 [*]	22.70 ± 0.03	NR	54.85 ± 0.01	NR	NR	NR	NR	NR	NR	NR	6.39 ± 0.01	12.27 ± 0.02	3.79 ± 0.01	100 ± 0.08	[137]
Grape peel (Burmese) ^c	82.51	15.24 [*]	NR	67.27 ± 0.39	10.86 ± 0.17	NR	56.41 ± 0.38	NR	NR	NR	NR	1.53 ± 0.15	9.10 ± 0.11	6.86 ± 0.12	100 ± 0.77	[138]
Hog plum bagasse ^m	54.20 ± 0.01	NR	NR	25.73 ± 1.00	6.67 ± 0.06	NR	19.06 ± 0.11	NR	NR	NR	NR	2.05 ± 0.10	7.29 ± 0.08	2.92 ± 0.03	92.19 ± 1.22	[139]
Jackfruit peel ^a	83.55 [*]	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	2.9	7.07	6.49	100	[140]
Lemon peel ^l	NR	NR	NR	NR	NR	31	NR	1.6	NR	21.2	0.4	NR	5.1	NR	59.3	[141]
Lemon peel ^h	95.12 ± 0.04	5.97 ± 0.00		89.15 ± 0.00	NR	NR	NR	NR	NR	NR	NR	0.31 ± 0.06	0.88 ± 0.09	3.69 ± 0.03	100 ± 0.36	[142]
Sweet lime peel ⁿ	46.84	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	5.61	13.02	4.98	70.09	[143]
Sweet lime peel ^l	NR	NR	NR	NR	NR	NR	NR	25.18	NR	17.07	7.34	NR	NR	NR	49.59	[129]
Mandarin (K'innow) peel ^{l,j}	NR	21.78 [*]	NR	NR	NR	22.88 ± 1.24 ²²	NR	3.88 ± 0.27	NR	10.72 ± 0.36	1.91 ± 0.15	29.66 ± 1.48	5.65 ± 0.34	3.52 ± 0.19	100 ± 4.03	[144]
Mandarin peel (Khas) ^c	89.73 [*]	51.91 [*]	NR	37.82 ± 0.33	9.23 ± 0.23	NR	28.57 ± 0.41	NR	NR	NR	NR	3.73 ± 0.11	8.05 ± 0.18	2.31 ± 0.09	51.91 ± 0.71	[138]
Mandarin orange peel ^l	61.86 ± 0.16	NR	NR	9.60 ± 0.13	NR	NR	NR	NR	NR	NR	NR	7.91 ± 0.19	14.97 ± 0.03	5.65 ± 0.16	100 ± 0.67	[145]

Table 4. (Continued)

Fruit waste	TC	WSC	STA	CF/TDF	SDF	PEC	IDF/NDF	HEM	ADF	GEL	LIG	CFat/EE	CP	Ash	SMC	Reference
Mango peel ^h	88.32*	73.61±0.31	NR	14.71±0.28	NR	NR	NR	NR	NR	NR	NR	1.90±0.07	4.22±0.05	5.55±0.06	100±0.77	[146]
Mango peel var. Ataulfo ^b	88.87±0.38	64.52*	NR	24.35±0.66 ⁹	4.17±0.56 ¹⁰	NR	20.18±0.77 ¹⁰	5.62±0.57	NR	8.16±0.18	6.41±0.42	1.78±0.21 ¹	5.05±0.01	4.33±0.14	100±0.74	[147]
Mango peel var. sugar ^f	87.9*	52.3±0.6	NR	35.6±0.5 ⁸	12.1±0.3 ⁸	NR	23.5±0.4 ⁸	NR	NR	NR	NR	1.6±0.0 ⁶	6.23±0.1 ¹⁴	4.2±0.0 ¹⁸	99.93±1.2	[148]
Mango peel (Mangifera odorata) ^f	92.25*	41.31*	NR	50.94±7.19	34.78±3.73	NR	16.16±3.46	NR	NR	NR	NR	0.58±0.04	4.31±0.04	2.86±0.01	100±7.28	[149]
Mango peel ^b	75.03±0.05	NR	NR	12.14±0.19	NR	NR	NR	NR	NR	NR	NR	1.75±0.25	7.23±0.12	3.85±0.34	100±0.86	[150]
Melon (Sharlyn) peels ^c	78.26*	48.67	NR	29.59	NR	NR	NR	NR	NR	NR	NR	1.58	9.07	11.09	100	[151]
Orange peel ^k	NR	NR	NR	NR	NR	NR	NR	6.61	NR	15.2	1.35	NR	NR	4.8	27.96	[15]
Orange peel ^l	78.76*	58.62±0.42	NR	20.14±0.88 ⁹	NR	NR	NR	NR	NR	NR	NR	16.20±0.18 ⁴	0.28±0.00 ¹²	4.92±0.04 ¹⁷	100.16±1.52	[80]
Orange peel ^h	89.3*	40.6±0.3	NR	48.7±0.6 ¹⁰	6.4±0.3 ¹¹	NR	42.7±0.5 ¹¹	NR	NR	NR	NR	1.5±0.1 ³	4.9±0.1 ¹²	4.2±0.1 ¹⁷	99.9±1.4	[152]
Orange peel ^g	82.96*	67.05	NR	15.91	NR	NR	NR	NR	NR	NR	NR	10.23	1.14	5.68	100	[153]
Orange peel ^l	85.35*	45.93±0.89	NR	NR	NR	18.96±0.9 ²¹	NR	5.7±0.15	NR	14.17±0.21	0.59±0.03	4.89	6.89±0.06	2.87±0.07	100±2.31	[154]
Orange bagasse ^l	NR	NR	NR	NR	NR	NR	NR	6.61	NR	15.2	1.35	NR	NR	NR	23.16	[15]
Passion fruit peel ^k	NR	NR	NR	NR	NR	NR	NR	11.8	NR	16.2	4.8	NR	n. r	6.0	38.8	[15]
Passion fruit peel ^k	NR	NR	NR	NR	NR	NR	NR	11.8	NR	25.4	4.9	NR	NR	6.0	48.1	[14]
Passion fruit peel ^l	91.86*	59.01±1.28	NR	32.85±0.02 ⁹	NR	NR	NR	NR	NR	NR	NR	0.47±0.03 ⁴	0.17±0.01 ¹²	6.32±0.08 ¹⁷	98.82±1.42	[80]
Passion fruit peel ^h	82.64*	21.57±0.54	NR	61.07±0.29 ⁷	NR	NR	NR	NR	NR	NR	NR	0.58 ¹	8.10±0.28 ¹⁴	8.68±0.14 ¹⁸	100±1.83	[155]
Passion fruit peel ^d	86.81*	23.41±0.10	NR	63.40±0.10	NR	NR	NR	NR	NR	NR	NR	0.87±0.04	4.82±0.03	7.50±0.07	100±0.34	[156]
Passion fruit peel ^g	88.3*	30.37±0.41	NR	57.93±2.72 ⁹	11.75±1.21 ¹⁰	NR	46.18±3.76 ¹⁰	NR	NR	NR	NR	0.64±0.02 ²	4.62±0.16 ¹⁴	6.44±0.14 ¹⁸	100±3.45	[157]
Passion fruit peel (yellow) ^b	87.42*	23.11±0.44	NR	64.31±0.86*	20.50±0.12	NR	43.81±0.74	NR	NR	NR	NR	0.11	4.77±0.31 ¹⁴	7.71±0.13 ¹⁸	100±1.74	[158]
Passion fruit peel (yellow) ^f	85.78±0.00	24.62*	NR	61.16±1.02	NR	NR	NR	NR	NR	NR	NR	4.20±0.03	3.40±0.06	6.61±0.24	100.89±1.35	[159]
Passion fruit peel (purple) ^l	80.71±0.00	19.03*	NR	61.68±1.31	NR	NR	NR	NR	NR	NR	NR	4.89±0.07	9.47±0.04	7.93±0.05	100±1.47	[159]
Passion fruit peel (Orange) ^f	64.86±0.00	2.72*	NR	62.14±2.62	NR	NR	NR	NR	NR	NR	NR	10.25±0.12	11.60±0.44	13.29±0.41	100±3.59	[159]
Passion fruit peel ^l	NR	NR	NR	NR	NR	NR	NR	11.8	NR	25.4	4.8	NR	NR	NR	42	[15]
Pineapple peel ^g	96.27*	25.55±1.99	NR	70.72±3.21 ⁹	24.49±1.63 ¹⁰	NR	46.23±1.79 ¹⁰	NR	NR	NR	NR	0.19±0.01 ¹	0.36±0.05 ¹⁴	3.18±1.52 ¹⁸	100±6.78	[131]

Table 4. (Continued)

Fruit waste	TC	WSC	STA	CF/TDF	SDF	PEC	IDF/NDF	HEM	ADF	CEL	LIG	CFat/EE	CP	Ash	SMC	Reference
Pineapple peel ^f	94.27*	82.61 ± 0.10	NR	11.66 ± 0.23 ⁹	NR	NR	NR	NR	NR	NR	NR	0.99 ± 0.16 ⁴	0.17 ± 0.03 ¹²	4.56 ± 0.03 ¹⁷	99.99 ± 0.55	[80]
Pomegranate peel ^f	96.2*	78.67 ± 0.32	NR	17.53 ± 0.74	NR	NR	NR	NR	NR	NR	NR	0.40 ± 0.03	0.7 ± 0.03	2.70 ± 0.23	100 ± 1.35	[160]
Pomegranate peel ^f	93.41*	76.64 ± 0.20	NR	16.77 ± 0.21	NR	NR	NR	NR	NR	NR	NR	0.21 ± 0.03	0.64 ± 0.05	2.74 ± 0.07	97 ± 0.56	[161]
Pomegranate peel ^h	79.6 ± 0.04	25.1 ± 0.02	NR	NR	15.27 ± 1.25	NR	36.36 ± 0.2	28.20 ± 1.06	NR	NR	NR	3.1 ± 0.005	15.6 ± 0.002	11.4 ± 0.03	109.93 ± 2.54	[162]
Pomegranate peel ^h	78.6 ± 0.08	24.1 ± 0.01	NR	NR	14.3 ± 1.25	NR	35.33 ± 0.33	29.30 ± 1.26	NR	NR	NR	3.3 ± 0.001	16.6 ± 0.005	12.4 ± 0.02	110.9 ± 2.86	[163]
Tomato peel ^f	NR	NR	NR	48.52 ± 0.67	5.12 ± 0.21	NR	43.40 ± 0.68	NR	NR	NR	NR	1.77 ± 0.04	14.47 ± 0.50	5.74 ± 0.03	70.5 ± 1.24	[164]
Watermelon peels	NR	26.0 ± 0.06	NR	NR	NR	NR	NR	12.8 ± 0.31	NR	50.01 ± 0.06	11.04 ± 0.29	3.6 ± 0.01	4.1 ± 0.02	NR	107.55 ± 0.75	[165]
Watermelon rinds ^c	72.37*	24.89*	NR	47.48 ± 0.47	15.03 ± 0.14	NR	32.45 ± 0.25	NR	NR	NR	NR	3.32 ± 0.20	16.45 ± 0.18	7.86 ± 0.17	75.11 ± 1.02	[138]
Watermelon rinds ^c	73.3*	56.02	NR	17.28	NR	NR	NR	NR	NR	NR	NR	2.44	11.17	13.09	100	[151]

Note: Methods referenced: ^aAOAC (1984)⁵³, ^bAOAC (1990)⁴⁹, ^cAOAC (1995)⁵², ^dAOAC (1997)³⁹, ^eAOAC (1999)⁴⁰, ^fAOAC (2000)⁵¹, ^gAOAC (2005)⁴⁶, ^hAOAC (2012)⁴⁵, ⁱGoering and Van Soest (1970)³⁶, ^jVan Soest et al. (1991)³⁷, ^kVan Soest (1994)¹⁶⁶, ^lAOAC (2016)⁴¹, ^mAOAC (2010)⁴³, ⁿAOAC (2006)⁴⁷. Specific quantification methods used: (i) fats: ¹AOAC 920.39, ²AOAC 948.22, ³AOAC 960.39, ⁴AOAC 963.15, ⁵AOAC 969.24, ⁶AOAC 991.36; (ii) fiber: ⁷AOAC 962.09, ⁸AOAC 985.29, ⁹AOAC 991.42, ¹⁰AOAC 991.43, ¹¹AOAC 2011.25; (iii) protein: ¹²AOAC 920.152, ¹³AOAC 950.48, ¹⁴AOAC 960.52, ¹⁵AOAC 984.13; (iv) ash: ¹⁶AOAC 923.03, ¹⁷AOAC 940.26, ¹⁸AOAC 942.05; (v) Pectin: ¹⁹Yu et al. (1996)¹⁶⁸, ²⁰Happi Emaga et al. (2008)¹⁶⁹, ²¹Sudhakar and Maini (2000)¹¹⁸, ²²Not specified. The full list of AOAC methods in the literature review can be found in Table S2.

Abbreviations: ADF, acid detergent fiber; CEL, cellulose; CF, crude fiber; CFat, crude fat; CP, crude protein; EE, ether extracts; HEM, hemicellulose; IDF, insoluble detergent fiber; LIG, lignin; NDF, neutral detergent fiber; PEC, pectin; SDF, soluble detergent fiber; SMC, summative mass closure; TC, total carbohydrates; TDF, total dietary fiber; WSC, water soluble carbohydrates.

* Data calculated using the information reported by the authors.

Table 5. Reported composition of fruit wastes (seeds and seed vessels) for the AOAC and Van Soest methods (dry weight basis).																
Fruit waste	TC	WSC	STA	CF/TDF	SDF	PEC	IDF/NDF	HEM	ADF	CEL	LIG	CFat/EE	CP	Ash	SMC	Reference
Achacharú seed ^f	71.16±0.30	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	23.59±0.04 ³	3.67±0.20	1.58±0.02	100±0.56	[122]
Almond meal ^h	NR	NR	NR	8.7	NR	NR	NR	NR	NR	NR	NR	11	48	4.51	72.21	[170]
Bush mango (<i>Irvingia gabonensis</i>) kernel ^d	21.7±0.82	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	69.09±0.36	6.65±0.36	2.56±0.05	100±1.59	[171]
Bush mango (<i>Irvingia wombolu</i>) kernel ^d	17.24±0.82	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	72.23±1.92	8.17±0.51	2.36±0.08	100±3.33	[171]
Granadilla seeds	NR	23.1	NR	26.3±0.7	NR	NR	NR	NR	NR	NR	NR	27.9±0.7 ²	19±1	2.18±0.04	97.38±2.44	[172]
Jackfruit seed ^a	81.33±0.26	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	0.81±0.19	14.04±0.25	3.82±0.08	99.63±0.78	[173]
Jatropha kernels ^d	6.22 [*]	3.02±0.02	NR	3.20±0.25 ⁴	NR	NR	NR	NR	NR	NR	NR	55.21±0.80 ¹	33.63±0.02 ⁷	4.95±0.43 ⁸	100±1.52	[174]
Juçara seed ^d	93.12 [*]	NR	12.76±0.98	80.36 ⁵	1.33±0.38 ⁵	NR	71.70±0.23 ⁵	NR	NR	NR	NR	0.25±0.02 ²	4.75±0.05 ⁶	1.88±0.01 ⁹	100±1.67	[175]
Mango seed kernel ^c	87.71±0.92	62.96 [*]	NR	24.75±1.30	ND	NR	24.75±1.30	NR	NR	NR	NR	5.59±0.06	5.31±0.06	1.40±0.07	74.73±2.41	[149]
Passion fruit seeds (yellow) ^c	71.07±0.00	5.47 [*]	NR	65.60±0.51	NR	NR	NR	NR	NR	NR	NR	12.31±0.78	13.07±0.12	3.56±0.05	100.01±1.46	[159]
Passion fruit seeds (purple) ^c	69.98±0.00	14.92 [*]	NR	55.06±0.35	NR	NR	NR	NR	NR	NR	NR	14.94±0.41	13.23±0.48	1.85±0.06	100±1.3	[159]
Passion fruit seeds (orange) ^c	61.38±0.00	9.91 [*]	NR	51.47±0.60	NR	NR	NR	NR	NR	NR	NR	19.64±0.30	15.84±0.15	3.23±0.18	100.09±1.23	[159]
Passion fruit seeds (yellow) ^b	49.44±1.07	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	32.16±0.29	17.57±0.29	1.82±0.04	100±1.69	[176]
Palm empty fruit bunch ^g	NR	NR	NR	NR	NR	NR	NR	9.5	NR	16	9.4	NR	NR	NR	34.9	[177]
Sesame seed cake ^a	39.6 [*]	28.8	NR	10.8	NR	NR	NR	NR	NR	NR	NR	30.3	23.7	6.12	99.72	[178]
Soybean cake ^{e,g}	NR	NR	NR	8.68	NR	3.11 ¹⁰	NR	NR	NR	NR	NR	NR	31.90 (n. s)	7.70 (n. s)	51.39	[179]
Tomato seed ^c	NR	NR	NR	54.24±0.66	9.48±0.47	NR	44.76±0.86	NR	NR	NR	NR	17.15±0.86	25.50±0.53	4.61±0.04	101.5±2.09	[164]
Watermelon seeds ^b	4.73	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	43.88±2.81	48.47±3.46	2.92±0.32	100±6.59	[180]
Wood apple seeds ^b	24.59	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	26.44±1.81	45.95±4.65	3.02±0.04	100±6.5	[180]
Note: Methods referenced: ^a AOAC (1990), ⁴⁹ AOAC (1995), ⁵² AOAC (2000), ⁵¹ AOAC (2005), ⁴⁶ AOAC (2006), ⁴⁷ AOAC (2012), ⁴⁵ Van Soest et al. (1991), ³⁷ AOAC (2002), ⁴⁸ Specific quantification methods used: (i) fats: ¹ AOAC 920.39, ² AOAC 945.16, ³ Bligh and Dyer (1959), ¹⁶⁷ ; (ii) fiber: ⁴ AOAC 962.09, ⁵ AOAC 985.29; (iii) protein: ⁶ AOAC 920.152, ⁷ AOAC 954.01; (iv) ash: ⁸ AOAC 923.03, ⁹ AOAC 940.26; (v) pectin: ¹⁰ da Silva et al. (2022), ¹⁸¹ . The full list of AOAC methods in the literature review can be found in Table S2 .																
Abbreviations: ADF, acid detergent fiber; CEL, cellulose; CF, crude fiber; CFat, crude fat; CP, crude protein; EE, ether extracts; HEM, hemicellulose; IDF, insoluble detergent fiber; LIG, lignin; NDF, neutral detergent fiber; PEC, pectin; SDF, soluble detergent fiber; SMC, summative mass closure; TC, total carbohydrates; TDF, total dietary fiber; WSC, water soluble carbohydrates.																
*Data calculated using the information reported by the authors.																

Table 6. Reported composition of fruit wastes (pomace and mixed residues) for the AOAC and Van Soest methods (dry weight basis).																
Fruit waste	TC	WSC	STA	CF/TDF	SDF	PEC	IDF/NDF	HEM	ADF	CEL	LIG	CFat/EE	CP	Ash	SMC	Reference
Acerola waste ^{a,o}	84.9 [*]	31.9	NR	NR	NR	NR	53.0 ⁷	14.7	n. r	29.0	9.25 ⁷	1.69 ¹	9.62 ¹⁴	3.78 ²⁰	99.99	[82]
Acerola waste	NR	NR	NR	NR	NR	NR	NR	18.24	NR	14.24	6.09	NR	NR	NR	38.57	[181]
Acerola by-product	52.0±0.6	NR	NR	34.2±0.4 ⁸	NR	NR	NR	NR	NR	NR	NR	2.9±0.2	8.3±0.3	2.8±0.0	100.2±1.5	[182]
Apple pomace ^b	84.46 [*]	28.98±1.1	NR	55.48±0.7 ⁹	11.06±0.1 ⁹	NR	43.58±0.6 ⁹	NR	NR	NR	NR	6.58±0.1	6.25±0.1	1.56±0.3	98.35	[183]
Apple pomace ^l	86.8	2.3	0.2 ²⁴	83.3 (1)	20	NR	63.3	10	NR	22.1	13.7	5.4 ¹	6.4 ¹⁵	1.4 ²⁰	100	[184]
Apple pomace ^f	91.99 [*]	25.88	NR	66.10±1.78 ⁹	20.66±0.09 ⁹	NR	45.44±0.24 ⁹	NR	NR	NR	NR	NR	3.64±0.08	4.37±0.06 ¹⁷	100±2.25	[185]
Apple pomace (Royal Gala) ^c	91.5 [*]	13.3	NR	78.2±0.6	14.33±0.61	NR	63.9±0.16	NR	NR	NR	NR	1.57±0.08	3.12±0.07	1.88±0.11	98.07±2.33	[186]
Apple pomace (Granny Smith) ^c	89.6 [*]	28.9	NR	60.7±0.23	4.14±0.21	NR	56.5±0.2	NR	NR	NR	NR	4.46±0.10	3.68±0.08	1.24±0.03	98.98±0.85	[186]
Apple pomace (Liberty) ^c	91.07 [*]	1.27	NR	89.8±0.24	8.20±0.15	NR	81.6±0.23	NR	NR	NR	NR	2.44±0.12	3.64±0.07	0.56±0.10	97.71±0.91	[186]
Apple pomace ^g	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	1.2±0.1	3.8±0.05	1.5±0.02	6.5±0.17	[187]
Apple pomace ^g	NR	NR	NR	NR	NR	NR	46.55	NR	39.32	NR	NR	6.63	8.23	1.29	102.02	[188]
Blackcurrant pomace ^a	NR	16.03±0.84	NR	59.65±0.80	NR	NR	NR	NR	NR	NR	NR	3.46±0.12	17.41±0.13	3.45±0.19	100±2.1	[189]
Blackcurrant pomace ^h	NR	NR	NR	32.3±0.4	NR	NR	NR	NR	NR	NR	NR	1.98±0.05	4.2±0.1	2.9±0.1	41.38±0.65	[190]
Carambola pomace ^b	81.57 [*]	21.4 [*]	NR	60.17±0.29	13.84±0.27	NR	46.32±0.22	NR	NR	NR	NR	1.94±0.08	13.65±0.10	2.84±0.15	100±0.62	[138]
Cashew bagasse ^b	NR	NR	NR	NR	NR	NR	NR	16.2	NR	12.7	34.5	NR	NR	4.0	67.4	[15]
Cashew apple waste ^h	76.68±1.39	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	5.83±0.76	10.36±0.08	1.52±0.08	100±2.31	[191]
Cherry pomace ^a	NR	15.02±0.41	NR	60.97±0.22	NR	NR	NR	NR	NR	NR	NR	3.44±0.23	17.96±0.26	2.61±0.01	100.31±1.14	[189]
Citrus pomace ^g	NR	NR	NR	NR	NR	NR	21.77	NR	16.19	NR	NR	1.25	9.83	3.03	52.07	[188]
Custard apple waste ^c	82.10±1.77	76.35 [*]	NR	3.71±0.59	NR	NR	NR	NR	NR	NR	NR	5.75±0.42	6.10±0.0	2.33±0.29	100±3.07	[192]
Grape pomace ^g	75.54 [*]	41.48	2.3	NR	NR	NR	31.76	NR	NR	NR	20.59	7.4	11.1	5.93	100.27	[193]
Grape pomace ^g	NR	NR	NR	NR	NR	NR	44.87	NR	38.82	NR	NR	7.23	12.25	10.29	113.46	[188]
Grapefruit waste (Ruby) ^c	80.9 [*]	18.3	NR	62.6±0.30	4.57±0.35	NR	56.0±0.17	NR	NR	NR	NR	3.24±0.05	8.42±0.12	3.22±0.01	95.78±1.0	[186]
Grapefruit waste (Marsh) ^c	89.2 [*]	45.0	NR	44.2±0.35	6.43±0.45	NR	37.8±0.21	NR	NR	NR	NR	1.04±0.01	4.46±0.04	3.27±0.05	97.97±1.11	[186]

Table 6. (Continued)

Fruit waste	TC	WSC	STA	CF/TDF	SDF	PEC	IDF/NDF	HEM	ADF	CEL	LIG	CFat/VE	CP	Ash	SMC	Reference
Guarana waste ^o	NR	NR	NR	NR	NR	NR	NR	59.37 ± 1.95	NR	7.82 ± 0.68	13.49 ± 1.54	NR	NR	5.59 ± 0.99	86.27 ± 5.16	[194]
Goldenberry pomace ^h	64.8 ± 0.18	47.02*	NR	17.78 ± 0.24 ⁶	NR	NR	NR	NR	NR	NR	NR	14.58 ± 0.11 ²	16.88 ± 0.01 ¹³	3.74 ± 0.16 ¹⁸	100 ± 0.7	[195]
Guava waste ^d	91.3*	22.2 ± 0.14	NR	69.1 ± 0.17 ⁹	11.1 ± 0.09 ⁸	NR	57.7 ± 0.15 ⁹	NR	NR	NR	NR	1.4 ± 0.10	4.8 ± 0.10	2.4 ± 0.10	99.6 ± 0.68	[196]
Jackfruit waste ^f	73.97	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	4.45	17.99	3.59	100	[13]
Juçara waste ⁱ	83.1*	43.0 ± 0.5	NR	40.1 ± 0.2	NR	NR	NR	NR	NR	NR	NR	7.4 ± 0.1	7.1 ± 0.3	2.4 ± 0.2	100 ± 1.3	[197]
Juçara seedless pomace ^h	79.02*	NR	ND	79.02 ⁸	4.46 ± 0.01 ⁸	NR	74.56 ± 0.23 ⁸	NR	NR	NR	NR	13.91 ± 0.5 ³	4.59 ± 0.30 ¹¹	1.92 ± 0.10 ¹⁹	100 ± 1.14	[175]
Lemon waste (Eureka) ^c	86*	25.9	NR	60.1 ± 0.22	9.20 ± 0.23	NR	50.9 ± 0.20	NR	NR	NR	NR	1.89 ± 0.02	6.79 ± 0.15	3.47 ± 0.05	98.15 ± 0.87	[186]
Lemon waste (Fino 49) ^c	82.9*	14.6	NR	68.3 ± 0.16	6.25 ± 0.16	NR	62.0 ± 0.16	NR	NR	NR	NR	1.88 ± 0.03	7.92 ± 0.08	3.91 ± 0.06	96.61 ± 0.65	[186]
Mandarin (Kinnow) waste ^f	90.37*	72.81*	NR	NR	NR	ND ²¹	NR	4.13 ± 0.11	NR	11.90 ± 0.09	1.53 ± 0.02	21.87*	6.20 ± 0.01	3.43 ± 0.12	100 ± 0.35	[198]
Mandarin (Kinnow) waste ^f	69.12*	31.58 ± 1.42	NR	NR	NR	22.6 ± 0.34 ²²	NR	4.28 ± 0.16	NR	10.10 ± 0.42	0.56 ± 0.01	NR	5.78 ± 0.25	3.23 ± 0.14	78.13 ± 2.74	[12]
Mango waste ^h	86.58*	41.98	NR	NR	NR	NR	28.95	NR	15.65	NR	NR	2.25 (n. s)	5.59 ¹⁰	2.79 ²⁰	97.21	[199]
Mango waste ^d	NR	11.9 ± 0.41	NR	70.0 ± 0.14 ⁹	28.2 ± 0.10 ⁹	NR	41.5 ± 0.24 ⁹	NR	NR	NR	NR	5.9 ± 0.05	8.0 ± 0.24	4.2 ± 0.36	100 ± 1.2	[196]
Mango waste ^d	83.7	NR	NR	NR	NR	NR	33.7	12.9	20.8	15.5	4.7	4.7	9.5	3.1	101	[81]
Orange waste ^f	NR	NR	NR	81.80 ± 2.01 ⁹	13.35 ± 0.02 ⁹	NR	68.45 ± 0.81 ⁹	NR	NR	NR	NR	NR	9.76 ± 0.05	8.44 ± 0.11 ¹⁷	100 ± 3.0	[185]
Orange waste (Valencia) ^c	82.2*	17.9	NR	64.3 ± 0.30	10.28 ± 0.30	n. r	54.0 ± 0.23	NR	NR	NR	NR	0.89 ± 0.04	6.70 ± 0.05	2.71 ± 0.09	92.5 ± 0.78	[186]
Orange waste ^o	NR	NR	NR	29.40	NR	23.47 ²³	NR	NR	NR	NR	NR	NR	8.54 (n. s)	4.51 (n. s)	65.92	[179]
Orange waste ^b	87.42 ± 0.9	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	2.51 ± 0.15 ⁵	6.11 ± 0.16 ¹⁶	3.95 ± 0.01 ¹⁷	100 ± 1.22	[200]
Orange waste ^a	80.38	14.68*	NR	65.7 ± 0.9 ⁹	16.8 ± 0.8 ⁹	NR	48.9 ± 0.5 ⁹	NR	NR	NR	NR	2.12 ± 0.11	13.25 ± 0.11	4.25 ± 0.07	100 ± 1.82	[201]
Passion fruit waste ^{d,i,f}	90.46*	18.67	NR	71.79 ⁹	19.45 ⁹	NR	52.34 ⁹	NR	NR	NR	NR	1.00 ⁴	0.35 ¹¹	8.08 ¹⁹	100	[202]
Passion fruit waste ^d	88*	6.5 ± 0.43	NR	81.5 ± 0.25 ⁹	35.5 ± 0.26 ⁹	NR	46.0 ± 0.10 ⁹	NR	NR	NR	NR	0.8 ± 0.14	6.2 ± 0.34	5.0 ± 0.16	100 ± 1.68	[196]
Passion fruit waste ^d	72.0	30*	NR	NR	NR	NR	52.0	14.7	38.8	24.5	12.5	3.8	12.0	13.2	101	[81]
Passion fruit waste ^{h,m}	57.72*	14.99 ± 0.04	NR	42.73 ± 0.91	NR	NR	NR	NR	NR	NR	NR	26.99 ± 0.84	13.73 ± 0.05	1.55 ± 0.06	99.99 ± 1.9	[203]
Passion fruit waste ^d	80.19*	23.38 ± 2.70	NR	60.81 ± 6.5	24.60 ± 2.40	NR	36.21 ± 4.10	NR	NR	NR	NR	0.11	8.25 ± 0.38	4.45 ± 0.12	100 ± 9.7	[8]

Fruit waste	TC	WSC	STA	CF/TDF	SDF	PEC	IDF/NDF	HEM	ADF	CEL	LIG	CFatVEE	CP	Ash	SMC	Reference
Pequi waste ^e	50.77	7.45 [*]	NR	43.32±0.72	9.38±0.93	NR	33.94±1.43	NR	NR	NR	NR	0.32±0.02	3.25±0.19 ¹²	2.34±0.03 ²⁰	100±3.32	[11]
Persimmon waste ^e	NR	NR	NR	NR	NR	NR	NR	4.84±0.09	NR	6.37±0.07	1.86±0.08	NR	NR	NR	13.07±0.24	[204]
Pineapple pomace ^b	90.69 [*]	10.93 [*]	NR	79.76±0.42	17.55±0.11	NR	62.21±0.33	NR	NR	NR	NR	1.43±0.16	5.95±0.16	1.93±0.08	100±0.82	[138]
Pineapple waste ^d	90.2 [*]	14.4±0.18	NR	75.8±0.23 ⁹	0.6±0.03 ³	NR	75.2±0.21 ⁹	NR	NR	NR	NR	1.3±0.03	4.0±0.17	4.5±0.03	100±0.64	[196]
Pineapple pomace ^d	NR	32.24±2.33	NR	51±2.78	NR	NR	NR	NR	NR	NR	NR	2.16±0.94	4.88±0.48	2.33±0.42	92.61±6.95	[205]
Raspberry pomace ^f	NR	13.37±0.60	NR	NR	NR	NR	NR	10.85±0.55	NR	9.72±0.71	18.95±0.71	5.07±0.32	2.58±0.20	1.30±0.05	58.72±3.14	[206]
Pineapple waste ^d	80.3	30.2 [*]	NR	NR	NR	NR	46.1	23.5	27.1	23.3	3.3	2.2	11.0	6.5	100	[81]
Tamarind waste ^{g,o}	74.82	35.7	NR	NR	NR	NR	39.1	20.5	18.6	15.7	2.92 ⁷	2.39 ¹	16.7 ¹⁴	6.11 ²⁰	100	[10]
Tomato pomace ^f	NR	NR	NR	64.12±0.56	5.56±0.34	NR	58.54±0.75	NR	NR	NR	NR	8.83±0.24	20.14±0.25	7.01±0.50	100.1±1.55	[164]
Tomato pomace ^{h,o}	NR	NR	NR	NR	NR	NR	NR	15.17	NR	16.17	33.81	35.75	NR	NR	100.9	[117]

Note: Methods referenced: ^aAOAC (1990)⁴⁹, ^bAOAC (1995)⁵², ^cAOAC (1996)²⁰⁷, ^dAOAC (1997)³⁹, ^eAOAC (1998)⁴⁴, ^fAOAC (2000)⁵¹, ^gAOAC (2002)⁴⁸, ^hAOAC (2005)⁴⁶, ⁱAOAC (2006)⁴⁷, ^jAOAC (2010)⁴³, ^kAOAC (2012)⁴⁵, ^lAOAC (2016)⁴¹, ^mAOAC (2009)²⁰⁸, ⁿVan Soest (1973)³⁸, ^oVan Soest et al. (1991)³⁷, ^pVan Soest (1994)¹⁶⁶, ^qAOAC (1999)⁴⁰. Specific quantification methods used: (i) Fats: ¹AOAC 920.39, ²AOAC 930.09, ³AOAC 945.16, ⁴AOAC 963.15, ⁵Bligh and Dyer (1959)¹⁶⁷; (ii) Fiber: ⁶AOAC 930.10, ⁷AOAC 973.18, ⁸AOAC 985.29, ⁹AOAC 991.43; (iii) Protein: ¹⁰AOAC 920.105, ¹¹AOAC 920.152, ¹²AOAC 960.52; ¹³AOAC 978.04; ¹⁴AOAC 981.10; ¹⁵AOAC 990.03; ¹⁶AOAC 991.20; (iv) Ash: ¹⁷AOAC 923.03, ¹⁸AOAC 930.05, ¹⁹AOAC 940.26, ²⁰AOAC 942.05. (v) Pectin: ²¹Sudhakar and Maini (2000)¹¹⁸, ²²Happi Enaga et al. (2008)¹⁶⁹, ²³Pang et al. (2012)²⁰⁹, (vi) Starch: 24 AOAC 920.40. The full list of AOAC methods in the literature review can be found in [Table S2](#).

Abbreviations: ADF, acid detergent fiber; CEL, cellulose; CF, crude fiber; CP, crude protein; EE, ether extracts; HEM, hemicellulose; IDF, insoluble detergent fiber; LIG, lignin; NDF, neutral detergent fiber; PEC, pectin; SDF, soluble detergent fiber; SMC, summative mass closure; TC, total carbohydrates; TDF, total dietary fiber; WSC, water soluble carbohydrates.

^{*}Data calculated using the information reported by the authors.

Table 7. Decision matrix comparing available methodologies for the characterization of fruit wastes.

Evaluation criteria	NREL	TAPPI	Van Soest	AOAC
Sample preparation	10	3	3	3
Total solids	10	10	5	5
Ash	10	7	7	7
Protein	10	0	10	10
Extractives	7	3	0	5
Structural carbohydrates	7	3	3	3
Lignin	10	7	7	7
Starch	10	0	0	7
Pectin	0	0	5	0
Sum	74	33	40	47

Note: The scores represent how much a given method satisfies the specification: 0 = poor, 3 = fair, 5 = average, 7 = good, 10 = excellent.

Other reports mention that starch is in the remaining composition but did not provide further measurements.^{126,127}

There is a lack of information on direct quantification of starch in all compositional analysis methods, which could lead to misleading information on potential uses of certain kinds of FW due to overquantification or underquantification of fiber, WSC, and starch. In this case, a suitable option to improve completeness and reliability of the compositional analysis of FW could be to include the most recent method for starch measurement reported by NREL, which could be considered an improvement of the AOAC methods (included in AOAC 2017.16) before fiber is measured. In the NREL methodology, it is necessary to remove starch enzymatically to quantify it using HPLC and to measure cellulose gravimetrically.⁶⁵

Extractives

To determine extractives using the NREL protocol it is required to have samples with an extractives content lower than 10%.⁵⁸ In cases where the extractives in the samples are greater than 10%, as seen in FW, the solid matrix of the biomass could still retain a significant amount of the extractives, which could lead to an underestimation of the actual content. This problem also occurs for the TAPPI methods, which use strong solvents (benzene, dichloromethane, hexane, and acetone) in contact with the sample for long periods (4–6 h).³⁴ Similarly, the AOAC methods use Soxhlet extraction (4–16 h) (Supporting Information, Table S2) with petroleum ether, or with a mixture of chloroform, methanol, and water. These methodologies could also result in the underestimation

of extractive content because the use of nonpolar solvents or solvent mixtures partially dissolves certain bioactive compounds from FW.²¹⁰

In general, quantification of extractives could be improved by combining polar and nonpolar solvents while using different sequences (e.g., water, ethanol, and a nonpolar solvent) with long extraction times to maximize the removal of these substances. Measurements of WSC, polyphenols, fatty acids, triglycerides, diglycerides, and so on, need to be included in each fraction extracted; this could also help to evaluate better possible valorization alternatives and value-added products. Bioactive compounds of interest should be identified and quantified carefully because they could increase profits further in biorefineries by having a high value, even though they could be present in relatively small amounts.¹¹²

Pectin

Another substance of much interest is pectin, which takes part in developing structures that provide structural stability and protection in plants. It has been observed that the pectin content can reach up to 35% in dicotyledonous plants, 2% to 10% in grasses, and 5% in wood tissue.²¹¹ For example, hazelnut pruning (a fruit harvesting residue) has a pectin content of 9.4%.¹⁰⁸ This biopolymer is abundant in peels and pomace but almost absent in seeds and seed vessels. Consequently, some authors include a pectin quantification methodology when peels, pomace, and mixed residues are analyzed because pectin could contribute substantially to a complete composition description in these fractions. On the other hand, there is a tendency to avoid quantification in seeds where pectin could be present in negligible amounts.^{70,179}

The methods reported for pectin quantification include gravimetric methods in which pectin is hydrolyzed with HCl and precipitated with ethanol^{118,169} and colorimetric methods to measure uronic acids using carbazole^{119,209} or *m*-hydroxyphenyl.^{120,121,168} The gravimetric method is unreliable because it depends on the extraction conditions used, which, in most cases, could leave behind some pectin.²¹² When carbazole is used, an unwanted reaction occurs with neutral sugars interfering with the measure of the pectin content.¹¹⁹ On the other hand, for *m*-hydroxyphenyl, it is still uncertain if complete hydrolysis of pectin is achieved.¹²¹ Different approaches have been proposed to determine the total pectin content, including the quantification of fractions consisting of water-soluble pectin,¹²⁰ oxalate-soluble pectin,¹⁶⁸ ethylenediaminetetraacetic acid (EDTA)-soluble pectin,¹²⁰ acid-soluble pectin,¹²⁰ and nonextractable pectin (protopectin).¹¹⁸ However, a reliable methodology for pectin

quantification has not yet been standardized, as evidenced from the collected information. A possible solution for pectin quantification would be to use chemical and enzymatical hydrolysis steps combined with measurements of the individual constituents (mainly galacturonic acid, glucuronic acid, and rhamnose) by chromatography.

Methods of pectin quantification are relevant to identify new sources of this substance apart from orange peels and apple pomace – the most common raw materials used for the production of high-quality pectin. In the data reported for apple pomace, tomato pomace, walnut endocarp, and almond shells/endocarp (Tables 2 and 3), some authors do not achieve a complete summative mass closure.^{68,72,113} The missing fraction to complete the whole composition for those biomasses could be pectin because it was identified in them by other authors.^{67,114} In the AOAC methods (Tables 4–6), SDF is the fraction obtained in solution after treatment with a neutral detergent mixture that solubilizes pectin and β -glucans. This allows us to hypothesize that most FW reported in this review contains considerable amounts of these substances.

Structural carbohydrates: cellulose and hemicellulose

The fiber content measured in FW (~60%–90% of their composition) makes evident the potential use of this biomass in a wide range of valorization platforms, which include the production of energy,^{88,102} materials,²⁵ and biochemicals.^{98,115} The two-step acid hydrolysis used in NREL procedures includes a set of sugar-recovery standards to correct for overhydrolysis,⁶⁰ allowing a good approximation of the relative amount of cellulose, hemicellulose, and lignin; however, the presence of starch in FW causes an overestimation of glucan.⁶⁵ The method for fiber determination (TAPPI T 203) in TAPPI methods is only useful to measure the mechanical properties of woods and pulps, which is not appropriate for FW. However, an alternative method proposed by TAPPI could be TAPPI T 249, based on two-step acid hydrolysis.³⁴ Nonetheless, this procedure does not consider sugar degradation during hydrolysis. Tables 4–6 show that some recent studies still report FW's composition using the Van Soest method, which gives only a rough estimate of fiber content in terms of NDF, ADF, and ADL.

In the case of AOAC methods, some reports tend to measure fiber based on methods 930.10,¹⁹⁵ 962.09,^{155,174} and 973.18⁸² (Supporting Information, Table S2), which use sodium hydroxide and sulfuric acid to determine crude fiber; however, these approaches do not give a clear

idea of the distribution of the structural polysaccharides in the sample.⁸⁵ Other reports use different versions of the methods based on the determination of soluble and insoluble fiber, such as methods 985.29 (the Prosky method),^{148,175} 991.42,^{80,124,131} and 991.43 (the Lee method),^{183,185,196} and 2011.25 (the McCleary method).¹⁵² Each procedure uses different enzymes and detergents, and has its own approximation to what components should be accounted for in the measured fractions (Supporting Information, Table S2).^{42,45,49} Although these methods differ slightly from each other and could be considered complementary, there is no guarantee that the fractions defined as NDF, IDF, and ADF in the AOAC methods (not a proper fiber fraction but just a preparatory step) are equivalent to glucan, xylan, mannan, arabinan, and galactan contents that are present in the sample.⁸⁵ Sometimes, procedures that determine fructans, galactooligosaccharides, and resistant starch are included to describe the composition of samples better.²¹³ At least 55% of the reports found in this review using AOAC methods do not specify the specific method used and only cite the year of publication; this demonstrates the problematic access to the latest versions of those procedures. As a result, information on fiber composition for FW using the AOAC method is not very reliable. Problems regarding these methodologies have been reported previously, describing the use of different proportions of reagents or samples, and diverse setups for filtration systems,²¹⁴ which causes high variability for both within-laboratory and among-laboratory results.

The most complete approach to measure the fiber components is the one proposed by the NREL, which could be further improved by the quantitative determination of starch, pectin, fructans, β -glucans, and other substances, which, in turn, could interfere with the actual amounts of cellulose, hemicellulose, and lignin.^{65,85}

Lignin

Lignin has multiple applications in biorefineries apart from its traditional use as fuel.^{70,111} The depolymerization of lignin is used to produce fuels and chemicals,¹¹¹ and it can be transformed into different materials such as carbon fiber, plastics, elastomers, and foams.⁹ In general, lignin quantification is the last step and it is performed similarly for all quantification methodologies.¹⁶ This substance is measured gravimetrically from the residue left behind after acid hydrolysis.¹⁶

For biorefinery applications, it would be attractive to couple lignin quantification with the measurement of

the individual phenylpropanoids present in lignin (i.e., hydroxyphenyl, guaiacyl, and syringyl). The relative amounts of lignin constituents from different biomasses can be used as a valuable input – for example, in the design of composite materials^{215,216} and cosmetic products.²¹⁷ Further understanding about the nature and specific characteristics of the lignin content present in FW would help in exploring new products that can be designed from them,²¹⁸ which maximizes the value recovered from those wastes.

Protein

Considering that most data reported in the literature and gathered in this review for FW characterization does not include protein content, an overestimation of lignin content could be expected.¹⁶ Although the Kjeldahl method is widely used for protein measurement,¹⁶ a careful selection of the nitrogen-to-protein conversion factor is recommended.⁶¹ Careful studies about the amino acid profile of each biomass are also highly recommended.

Ash

Finally, all procedures quantify ash at high temperatures (500–575 °C). Nevertheless, the most convenient form to measure ash is as NREL recommends. This method uses a temperature ramp that degrades the sample slowly, avoiding possible losses caused by rapid heating, which could result in underestimating the ash content. Short operation times are employed in both AOAC (Supporting Information, Table S2) and TAPPI methods, which could cause an overestimation of ash because a part of the sample (mostly fixed carbon) can remain unburnt. This problem can be fixed by using longer residence times to guarantee that samples of FW with high ash content (e.g., banana, dragon fruit, watermelon peels) are measured appropriately. Besides that, it must be considered that an ash content higher than 10% interferes with neutralization after acid hydrolysis due to the minerals present in samples, altering the amount of fiber measured.

Suggested sequence for chemical characterization of vegetable biomass including fruit wastes

Based on the information gathered and the discussions in the previous sections a possible sequence for a complete and reliable chemical characterization of the different fraction and components present in vegetable biomass, including FW, is presented in Fig. 4. The sequence was constructed by considering the presence of FW, like peels or seeds, in the

biomass as critical points to decide whether to use alternative/complementary quantification techniques that come from the techniques identified and described in this study (NREL, AOAC, TAPPI) and those that could be used for pectin. In the case of protein it is necessary to consider if the nitrogen source organic (e.g., proteins or amino acids present in the seed) or inorganic (nitrates or nitrites from soil/fertilizers) in order to distinguish what is truly available for recovering. Regarding extractives, it is necessary to use longer extraction times if the biomass has a content higher than 10%. For each extraction solvent, it is strongly recommended to determine extractive yields gravimetrically and recover aliquots for further analysis of bioactive compounds by HPLC or GC to ensure an accurate and comprehensive description of the fraction. Moreover, as pectin quantification methodologies are still to be standardized, it is suggested that at least two of the available methods should be used to have more certainty of its real content. Finally, the presence of starch on biomass requires not only using a method for its quantification (as in AOAC), but also a way to distinguish this substance from cellulosic glucan, which is clearly described by NREL.

Current knowledge gaps and future directions

Although significant progress has been made to determine the composition of FW, key challenges remain that limit its full valorization in biorefineries. One major issue is the absence of standardized and reliable methodologies for accurately quantifying pectin and starch, which are often underestimated or excluded. The development of robust carbohydrate analytical methods, including reliable approaches for the breakdown of pectin and chromatographic techniques for identifying and quantifying its constituents, is essential to address variability and improve the consistency of results. Current methodologies frequently neglect the effects of extractives, proteins, and ash, which interfere with acid hydrolysis and carbohydrate analysis, introducing errors in biomass characterization.

The underestimation of extractives due to the use of nonpolar solvents or solvent mixtures also highlights the need for methodologies that combine the quantification of both polar and nonpolar extractives while identifying individual bioactive compounds. Lignin characterization could also be improved by measuring the relative proportions of its phenylpropanoid units, which would enable the exploration of novel applications in areas such as composite materials and cosmetics.

Another critical gap is the limited availability of high-quality compositional datasets for FW and vegetable biomass

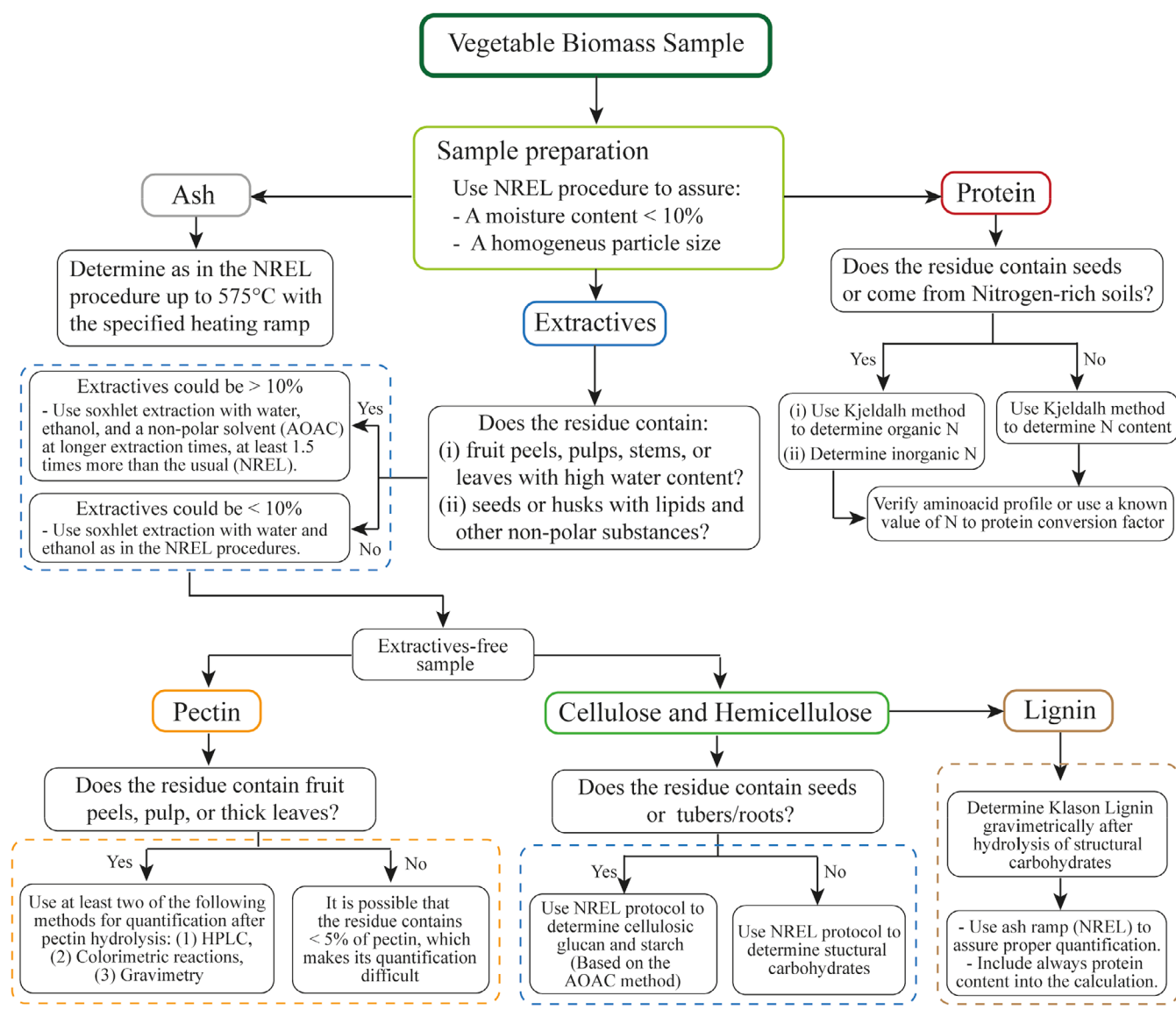


Figure 4. Proposed sequence for the characterization of vegetable biomass including FW.

in general. Expanding these datasets could facilitate a better understanding of FW's potential as a feedstock for biorefineries through the use of machine-learning techniques. Moreover, including the characterization of other plant polysaccharides like inulin could further enhance the range of valorization possibilities.

It is essential to move beyond the segmented analysis of biomass fractions and adopt a holistic approach where biomass composition is fully described and summative mass closure is achieved. Such an approach would provide a comprehensive and standardized framework for evaluating FW and comparing different feedstocks. Addressing these gaps would not only improve the accuracy and reliability of FW compositional analysis but would also strengthen the

foundation for designing sustainable and efficient biorefinery systems.

Concluding remarks

There is currently no definitive method that describes completely and accurately all substances present in FW. Several useful methodologies were identified in this literature study, which together could result in a more complete and reliable chemical characterization of FW. An improved set of procedures for compositional analysis of biomass should not consider substances as separate pieces from different puzzles but as chemical blocks that could, as a whole, be used in different industries. Finally,

the compositional analysis procedure should not depend on the needs of specific industries and should, on the contrary, be constructed from a holistic approach that minimizes wastes and that employs multiple valorization platforms. To accomplish this, a standardized basis for complete and reliable characterization of the different fractions and components of FW would be a valuable tool to study distinct alternatives for the sustainable valorization of biomass and biorefinery systems.

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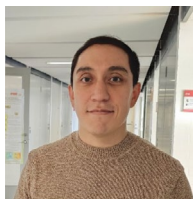
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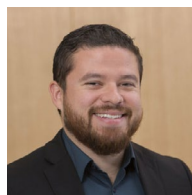
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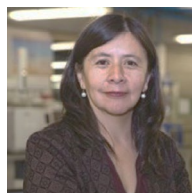
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