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ORIGINAL PAPER

The Challenges of Using Organic Municipal Solid Waste as Source of Secondary Raw Materials

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Abstract

The diversity of molecules with different functionalizations allows targeting of various end products, such as biomaterials, biobased plasticizer, food additives and fertilizer. The heterogeneity of organic municipal solid waste (OMSW) streams, however, challenges the formulation of reliable statements regarding the share of functionalized molecules. The aim of this study was the assessment of OMSW as source of functionalized molecules when hydrolysis was carried out enzymatically, thermo-chemically as well as thermo-chemically and enzymatically. Results revealed that OMSW is only quantitatively assessable at carbohydrate, protein and lipid levels. This is due to a changing seasonal and spacial composition, and consequently different hydrolytic products. However, also the treatment had an impact on the quantity. Depending on the treatment 230–640 mg g^{-1} carbohydrates, 150–250 mg g^{-1} lipids and 80–200 mg g^{-1} proteins were quantified in food waste and organic street waste. The intensity of treatment had an impact on the quality of sugars. When wastes were treated enzymatically glucose, fructose and sucrose were found. Using thermochemical treatment glucose can be the only product. Contrarily, lipid and fatty acid as well as protein contents seemed not affected by the treatment.

Keywords Municipal solid waste · Hydrolysis · Secondary raw materials · Characterization

Abbreviations

FAN	Free amino nitrogen
HPLC	High performance liquid chromatography
Nd	Not detected
OMSW	Organic municipal solid waste
U	Enzyme units
Total-C	Total-carbon content
Total-N	Total-nitrogen content

Statement of Novelty

Nowadays, biotechnological and chemical processes are used for converting OMSW as a whole into biochemicals and energy-rich compounds. It is beneficial that such an approach does not require a separation of constituents beforehand. The potential of the organic material as source of functionalized molecules, such as sugars, amino acids and fatty acids, however, cannot be conserved. This, however, is needed to develop new and innovative utilization processes. The novel aspect of this study is the consideration and assessment of OMSW as direct source of functionalized molecules. An assessment can only be carried when sufficient data on the presence of functionalized molecules is available, which also allows a conclusion on the presence of functionalized molecules in different waste streams.

Introduction

Sustainable chemistry investigates processes in order to apply resources efficiently and to achieve a holistic use in chemical and/or biotechnological processes [1]. Organic municipal solid waste (OMSW) is currently either composted or directly/indirectly energetically used. The indirect energetic use of organic materials is based on the conversion of highly functionalized molecules (Table 1) into methane and carbon dioxide. The conversion of sugars, for instance, into methane and carbon dioxide results in a loss of functionalization [2]. Furthermore, 50% of the carbon is lost as carbon dioxide [3]. Instead of using OMSW

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Table 1 Composition of OMSW and by hydrolysis obtainable secondary raw materials

Composition	Secondary raw materials (functionalized molecules)	Building blocks (selection)
Starch, hemicellulose, cellulose	Glucose, fructose, xylose, arabinose, lactose, sucrose	Lactic acid, propionic acid, succinic acid, fumaric acid, malic acid, furfurale
Proteins	Alanine, arginine, asparagine, aspartic acid, cysteine, glutamic acid, glutamine, glycine, histidine, isoleucine, leucine, lysine, methionine, phenylalanine, proline, serine, threonine, tryptophan, tyrosine, valine	
Lipids	Glycerol, myristic acid, palmitic acid, palmitoleic acid, stearic acid, oleic acid, linoleic acid, alpha-linolenic acid	
Lignin	Coumaryl alcohol, coniferyl alcohol, sinapyl alcohol, coumaryl aldehyde, coniferyl aldehyde, sinapyl aldehyde	
Polyphosphate	Phosphate	

Secondary raw materials can be converted into building blocks and final products by chemical and biotechnological industries. Amino and fatty acids and phenols can be used directly as building blocks, while sugars require first fermentative and/or catalytic conversions

energetically, a direct use as source of highly functionalized molecules is indicated.

OMSW is heterogeneous and may consist of food and kitchen wastes, paper, coffee and tea residues, grass and green clippings. An average composition (w/w) of OMSW of 17.5% lipids, 17.7% proteins, 17.1% starch, 10.5% free sugars, 18.6% cellulose, 9.7% lignin and 8.6% hemicellulose [4] illustrates, that the easily hydrolysable parts, such as starch, lipids and proteins, may represent 62.8% of the waste matter. Recalcitrant parts, such as cellulose, hemicellulose and lignin may represent 36.9%. It is suggested that the utilization of organic matter should follow a cascading principle in order to develop a biobased society [5]. An option to utilize organic matter materially is its use as feedstock in biotechnological processes. Particularly in fermentative processes [6–10], the easily hydrolysable waste constituents can serve as substrates for microorganisms with or without hydrolysis carried out beforehand.

Another utilization approach might be the separation of present functionalized compounds (Table 1) as secondary raw materials directly from hydrolyzed organic matter, which can be converted into final products, such as biomaterials, biobased plasticizer, food additives and fertilizer [11]. It is crucial for the efficiency of OMSW-based utilization processes that the whole process is flexible and runs stable irrespective the waste applied [12]. It is hypothesized here that this might be achieved by carrying out a sequential hydrolysis of hydrolysable parts. The advantage is that functionalized molecules can be separated sequentially after every hydrolytic step and the inhibition of hydrolytic performance by waste constituents, such as the protection of carbohydrates by lipids [13], as well as products can be avoided. Nevertheless, the direct utilization of OMSW through the recovery of all functionalized molecules is challenging. The variable composition makes a continuous adaption of the pretreatment (hydrolytic approach) and separation necessary. Furthermore, there is no sufficient database regarding the presence of functionalized molecules in OMSW.

The investigation of hydrolytic processes and inventory of functionalized molecules, obtained by hydrolysis of OMSW, is expected to lead to highly wanted basics in order to develop more efficient utilization processes. Information regarding the effect of hydrolytic processes on the presence and recovery of functionalized molecules contributes to the development of technical processes which can be applied where organic residues appear, such as near food processing and agriculture industries as well as municipalities. It has never been more important to develop efficient utilization strategies of organic residues in order to cope with the challenges to cover the demand of resources in the future.

Therefore, the aim of this study was to carry out a characterization of functionalized molecules, such as sugars, long- and short chained fatty acids, proteins and amino acids, obtainable from OMSW using different hydrolytic treatments and an assessment of separation techniques in order to recycle those molecules back in the sense of a circular economy as secondary raw materials. For this purpose food waste and street waste have been collected and waste constituents sequentially or in one-batch approaches enzymatically, thermo-chemically or thermo-chemically as well as enzymatically hydrolyzed.

Materials and Methods

Organic Waste

Food waste (leftover food), made of potatoes, noodles, bread, meat, vegetables, was randomly collected from the canteen at Leuphana University of Lüneburg (Germany) at different times in May 2017 and April 2018. Directly after, the waste was blended and stored at − 18 °C until further usage.

Street waste was randomly collected from waste bins located in Lüneburg at different times in October and November 2017. Organic waste was mixed with inorganic material and the organic fraction (15–22%, w/w) was separated by hand. The organic fraction was predominantly made of thrown away food, such as buns, sandwiches and sausage. After collection it was blended and stored at − 18 °C until further usage.

Enzymes

All enzymes used were obtained from ASA Spezialenzyme GmbH (Wolfenbüttel, Germany). The activities of the different enzyme formulations reported by ASA Spezialenzyme GmbH were: > 30 U mL^{-1} for cellulase (TXL); 1.200 U mL^{-1} for glucoamylase (AN); > 300 U mL^{-1} for exo-polygalacturonase, > 3000 U mL^{-1} for endo-polygalacturonase and > 300 U mL^{-1} for pectinase present in pectinase (L-40) formulation from *Aspergillus niger*; > 400,000 U g^{-1} for xylanase and > 900 U g^{-1} for cellulase present in xylanase (2) formulation; > 18,000 U mL^{-1} (glyceryl tributyrate) and > 13,000 U mL^{-1} (olive oil) for Lipase (FE-01). For the protease (S-02) formulation no activity was provided. Hydrolytic treatments were carried out at provided optimal temperature and pH conditions as shown below.

Reference Compounds

To assess the complexity of organic materials, homogeneous reference compounds, such as starch powder, cellulose in form of paper tissue, protein powder and butter were hydrolyzed enzymatically or thermo-chemically as well as enzymatically. Reference compounds were chosen in order to illustrate the recalcitrant character and resistance against hydrolytic treatments. Focus was laid on digestibility and released monomers, such as glucose, fructose, sucrose, amino acids, fatty acids and phosphate. All hydrolyses were carried out as batch processes.

Paper tissue was collected from a tissue dispenser at a university restroom, cut into small pieces and 5 g mixed with 500 mL demineralized water. Thereafter, the suspension was added to a 1 L EloFerm bioreactor (Biotronix GmbH, Berlin, Germany). Temperature was set to 50 °C and pH to 4.5 before cellulase was added. Enzymatic hydrolysis was carried out for 24 h. Furthermore in a second approach, to 5 g of cut paper tissue 500 mL of 0.6% (v/v) sulfuric acid was added. The suspension was autoclaved for 15 min at 121 °C in a Schott flask and the resulting suspension enzymatically hydrolyzed as described before for 24 h. Samples were taken regularly.

Similar to the paper tissue hydrolysis, in a first approach, starch or protein powder was hydrolyzed using glucoamylase at 55 °C and pH 4.5 or using protease at 60 °C and pH 3.0 for 24 h, respectively. The solid-to-liquid ratio was 5.4% (w/w) for starch powder and 1.4% (w/w) for protein powder due to solubility issues. In a second approach, both powders were first thermo-chemically treated in presence of 0.6% (v/v) sulfuric acid and autoclaved for 15 min at 121 °C in a Schott flask. The solid-to-liquid ratio was again 5.4% (w/w) for starch powder and 1.4% (w/w) for protein powder. Afterwards, enzymatic hydrolysis was carried out as described above. Samples were taken regularly.

In order to assess the impact of complexity of organic waste on hydrolytic performance, organic waste was simulated by mixing 65% (w/w) starch powder, 20% (w/w) butter and 15% (w/w) protein powder. The mixture (20 g dry weight) was resuspended in 780 g demineralized water. First for lipid hydrolysis, temperature was adjusted to 40 °C, pH set to 7.5, and 1 mL lipase was added. After 4 h, temperature was increased to 55 °C, pH set to 4.5, and saccharide hydrolysis was initiated by adding 1 mL of glucoamylase. After 4 h, temperature was increased to 60 °C, pH set to 3.0 and 1 mL protease was added for protein hydrolysis. Hydrolysis was stopped after 24 h in total. Samples were taken regularly. In a second approach, artificial organic waste was first thermo-chemically treated in presence of 0.6% (v/v) sulfuric acid and autoclaved for 15 min at 121 °C in a Schott flask, and followed by sequential enzymatic hydrolysis as described above.

Organic Waste

Free Organic Molecules

The liquid phase of food waste was first investigated for the presence of organic molecules by centrifuging 1 mL of blended food waste (solid-to-liquid ratio 24%, w/w) at 19,000×*g* for 5 min. Obtained solution was subjected to HPLC.

Blended street waste of 0.1 g (dry weight) was resuspended in 1 mL demineralized water and vortexed for 5 min. Afterwards the suspension was centrifuged at 19,000×*g* for 5 min. Obtained solution was subjected to HPLC.

Sequential Hydrolysis of Waste Constituents

Enzymatic hydrolysis of food wastes 1 and 2 was carried out at a 2.4% (w/w) solid-to-liquid ratio in a 1 L EloFerm bioreactor (Biotronix GmbH, Berlin, Germany). The individual components were hydrolyzed sequentially in order to ensure an optimal performance of hydrolytic enzymes as described for the artificial waste in "Reference Compounds" section. For food waste 1 a 24 h reaction time was considered for each enzyme. Due to the fast reaction, reaction time was shortened to 4 h for food waste 2.

In a second approach, food waste 2 was first thermo-chemically treated in presence of 0.6% (v/v) sulfuric acid and autoclaved for 15 min at 121 °C in a Schott flask, and followed by sequential enzymatic hydrolysis as described for the artificial waste in "Reference Compounds" section.

Separate Hydrolysis of Waste Constituents

The characterization of food waste 2 and street wastes regarding dry matter, ash, total carbon (C)- and nitrogen (N)-contents has been carried out separately as described in "Analytics" section.

As a tough treatment and for comparison, street wastes were thermo-chemically hydrolyzed by resuspending ca. 0.1 g dry material in 3 mL 2.5 M H_2SO_4 and autoclavation for 15 min at 121 °C. Released sugar monomers were determined by HPLC.

Analytics

Analysis of total carbon—(C-content) and nitrogen—(N-content), water, ash, carbohydrate and lipid contents was carried out for both wastes and performed in triplicate or single measurements. Mean value and standard deviation are presented for triplicate measurements in "Results and Discussion" section.

In order to determine the dry matter, aliquots were weighed and dried at 105 °C in a compartment dryer (ST 5028, Heraeus, Hanau, Germany) until constant weight.

Ash content was quantified by heating 1 g dry waste for 4 h at 550 °C in a muffle furnace (Muffle furnace LT 5/12, Nabertherm, Bremen, Germany) and weighing the remainder.

Glucose, fructose and sucrose were analyzed with HPLC (LC-10AD pump, SIL-10AD auto-sampler, CTO-10AD oven, CBM-20A communication module, Shimadzu, Kyoto, Japan): 10 µL of sample was injected in an Aminex (Bio-Rad, Hercules, California, USA) HPX-87H column (300 mm × 7.8 mm) and eluted isocratically with 0.4 mL min^{-1} of 5 mM H_2SO_4 at 27 °C. Detection was carried out by a refractive index detector (RID-20A, Shimadzu, Kyoto, Japan) at 40 °C. For each analyte, calibration curves were generated with pure solutions of known concentration.

Total C- and N-contents were measured with an elemental CN analyzer at 1150 °C (Elementaranalysator vario Max CN; Elementar Analysensysteme GmbH, Hanau, Germany). Protein content was estimated by multiplying the N-content with 5.6 [14].

Lipid extraction was carried out by adding 5 mL CH_3OH/$CHCl_3$ (2:1, v/v) to 0.1 g dry waste or butter containing tridecanoic and nonadecanoic acids as internal standards and shaking for 24 h. After centrifugation, the supernatant was decanted and stored at − 18 °C. To the pellet 5 mL CH_3OH/$CHCl_3$ (1:1, v/v) was added, shaking continued for another 24 h and the suspension was centrifuged. Both supernatants were combined and 2 mL demineralized water was added to remove non-lipid components. The organic phase was collected, evaporated at room temperature under nitrogen flow and the mass of the crude oil extract was measured.

The individual fatty acids were converted to their respective fatty acid methyl esters by dissolving the crude oil extract in 0.2 mL $CHCl_3$, 2 mL CH_3OH and 0.1 mL concentrated hydrochloric acid. The solution was heated at 100 °C for 1 h. After cool down, 2 mL hexane and 2 mL demineralized water were added, the solution was shaken and after phase separation the hexane phase isolated. 1 µL of the hexane phase was injected for GC/EI–MS analysis (Trace 1310 gas chromatograph interfaced with a single-quadrupole ISQ, Thermo Scientific, Waltham, Massachusetts, USA). As column, a Select FAME fused silica capillary column (50 m × 0.25 mm ID, 0.25 µm film thickness, Agilent Technologies, Waldbronn, Germany) was used with helium as carrier gas. Ionization was conducted with an electron energy of 70 eV, the temperature of the ion source was set to 250 °C. Scans were recorded over the range of m/z 60–400.

Release of amino acids was measured as free amino nitrogen (FAN) in supernatants obtained from proteolytic treatment of protein powder and waste using a modified version of the EBC-ninhydrin method [15]. First, two reagents were prepared. For reagent A, 1 g $Na_2HPO_4 \cdot 12H_2O$, 0.6 g KH_2PO_4, 0.05 g ninhydrin and 0.03 g fructose were dissolved in 10 mL demineralized water. Reagent B contained 0.2 g KIO_3, 60 mL demineralized water and 40 mL absolute ethanol. For analysis, 20 µL sample, 50 µL A and 30 µL demineralized water were combined and heated at 90 °C for 5 min. Then 900 µL of B was added and absorption at 570 nm (Ultrospec III, Pharmacia, Uppsala, Sweden) was measured. A calibration curve with glycine as standard was used as reference.

Determination of amino acids after proteolytic treatment of food waste and protein powder was carried out using the conversion of amino acids into corresponding alpha-hydroxy acids [16, 17]. Alpha-hydroxy acids were analyzed using HPLC as described above. Peaks were identified by combining retention time with reference compounds.

Phosphate concentration was determined photometrically via generation of molybdenum blue. At first, four separate solutions were prepared: (I) sulfuric acid (2.5 M), (II) potassium antimonyl tartrate solution (1.3715 g K(SbO)$C_4H_4O_6 \cdot 1/2H_2O$ in 500 mL demineralized water), (III) ammonium molybdate solution (20 g $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 500 mL demineralized water) and (IV) ascorbic acid solution (1.76 g ascorbic acid in 100 mL demineralized water). Molybdenum reagent (V) was prepared by combining 2.5 mL (I), 0.25 mL (II), 0.75 mL (III) and 1.5 mL (IV). Sample (100 µL), 900 µL demineralized water, 10 µL (III) and 160 µL (V) were mixed. After incubating at 60 °C for 15 min, absorption was measured at 880 nm (Ultrospec III, Pharmacia, Uppsala, Sweden). In the case of fermentation broth, 40 µL of sample was taken and 960 µL demineralized water was added.

Results and Discussion

Reference Compounds

It can be seen from the results shown in Table 2 that an intensive treatment and hydrolysis result in higher contents of constituents and yields of hydrolytic products, respectively. When paper tissue was treated enzymatically no glucose was detected. However, when the same paper was first thermo-chemically treated followed by enzymatic hydrolysis then 139 mg glucose per g paper was released. Additional di- and oligosaccharides were detected but not identified. Starch has a less recalcitrant structure than cellulose and was applied as powder rather than a fiber, and thus 451 mg glucose could be released per g when hydrolysis was carried out enzymatically. With a precedent thermo-chemical treatment, the yield could be increased to 780 mg g^{-1}. Furthermore, the release of FAN increased by a factor of around 7 when protein powder was pretreated prior to enzymatic hydrolysis (Table 2). Nevertheless, the release of carbohydrates, FAN and phosphate from artificial waste shown in Table 2 was similar or did only slightly increase when thermo-chemical treatment was applied beforehand.

The fatty acid profile of butter was dominated by oleic and stearic acid with contents of about 635.9 mg g^{-1} and 300.2 mg g^{-1}, respectively. Fatty acids present at contents of 1.4 mg g^{-1}, 17.8 mg g^{-1} and 44.0 mg g^{-1} were dodecanoic, myristic and palmitic acids, respectively (Table 3). The glycerol content of butter was estimated at 100 mg g^{-1}. The total lipid content was 100% (w/w).

Free Organic Molecules

Regarding the separation of functionalized molecules, it was first investigated whether functionalized molecules can be directly obtained from wastes by separation of liquid and solid phases. After washing of street wastes and analyzing the resulting washing water with HPLC no considerable

Table 2 Constituents in reference substances and artificial waste using different hydrolytic treatments

Constituent (mg g^{-1})	Paper[a]	Paper[b]	Protein powder[a]	Protein powder[b]	Starch powder[a]	Starch powder[b]	Artificial waste[a]	Artificial waste[b]	Butter[c,e]
Carbohydrates (glucose/fructose/sucrose)	Nd	139.3[d]	–	–	450.8[d]	779.3[d]	203.2/18.6/25.9	205.7/77.6/38.5	–
FAN	–	–	0.6	4.5	-	–	3.9[e]	2.9[e]	–
Phosphate	–	–	–	–	–	–	1.3[e]	1.9[e]	–
Glycerol	–	–	–	–	–	–	~20	~20	~100

The contents/yields are based on total weight of reference substances or artificial waste

Nd: not detected; –: not analyzed

[a]Hydrolysis was carried out enzymatically

[b]Hydrolysis was carried out first thermo-chemically and second enzymatically

[c]Hydrolysis was carried out chemically as part of the transesterification step for fatty acid quantification

[d]Only glucose was identified

[e]Lipid content was considered 100% (w/w)

Table 3 Fatty acid contents based on total weight of lipids detected in waste materials

Fatty acids (mg g^{-1})	Food waste 1	Food waste 2	Street waste 1	Street waste 2	Butter
Dodecanoic acid	Nd	Nd	3.2	16.4	1.4
Myristic acid	Traces	38.5 ± 0.2	4.9	15.7	17.8
Palmitic acid	288.3 ± 64.8	339.3 ± 9.1	384.9	311.2	44.0
Palmitoleic acid	Nd	Nd	0.3	1.6	Traces
Stearic acid	46.1 ± 9.6	54.4 ± 0.4	51.9	75.2	300.2
Oleic acid	364.9 ± 76.4	430.7 ± 2.8	435.9	393.2	635.9
Linoleic acid	106.8 ± 17.0	127.5 ± 6.8	109.4	174.8	Traces
Alpha-linolenic acid	Traces	8.7 ± 6.0	7.0	10.3	Traces
Eicosanoic acid	0.7 ± 0.2	0.9 ± 0.1	2.4	1.5	Traces

Nd not detected

amounts of detectable compounds were found. The supernatant of food waste fraction did reveal concentrations for glucose, fructose, sucrose and lactic acid of 25.3 g L^{-1}, 13.8 g L^{-1}, 11.2 g L^{-1} and 7.2 g L^{-1}, respectively, at a solid-to-liquid ratio of 24% (w/w).

Sequential and Separate Hydrolysis of Organic Waste

Street and food wastes were randomly collected at the same location, but at different times. All waste materials predominantly consisted of carbohydrate, lipid and protein, which made it an easily hydrolysable material.

While chemical composition of all wastes was similar, the quantity varied due to different hydrolytic treatments. The biggest deviation of all materials was found for the dry matter (Table 4). The lowest dry matter of 217 mg g^{-1} had food waste 2, while the highest dry matter of 461 mg g^{-1} was found in street waste 1. Ash content (Table 4) was around 62 mg g^{-1} in both food wastes and street waste 2 and 92 mg g^{-1} in street waste 1. Even though no phosphatases were applied, 5.2 mg phosphate was recovered per g of food waste 2.

When materials were enzymatically or thermo-chemically as well as enzymatically treated, carbohydrate content was 200–300 mg g^{-1} in food waste 2 and in both street wastes, while food waste 1 contained around 600 mg g^{-1}. Fructose and sucrose yields differed between all wastes. The pure thermo-chemical hydrolysis with 2.5 M H_2SO_4 resulted in glucose yields of 417 mg g^{-1} and 568 mg g^{-1} in street waste 1 and 2, respectively. There was neither fructose nor sucrose detectable after chemical treatment.

Food waste 1 and street waste 2 had a similar C-content of about 495 mg g^{-1} (Table 4). The C-content of food waste 2 and street waste 1 was 467 mg g^{-1} and 482 mg g^{-1}, respectively. N-content ranged from 14 mg g^{-1} in street waste 1, around 24 mg g^{-1} in food waste 1 and 2, and 36 mg g^{-1} in street waste 2. Correspondingly, also the protein contents ranged from 79 to 204 mg g^{-1}. All amino acids present in the hydrolysate of food waste 1 could not successfully be separated and identified. Clearly separated and identified were asparagine, valine, lysine, cysteine and tryptophan. Additionally present, but not clearly identified owing to very similar retention times, might be serine, threonine, glutamic acid, phenylalanine, asparagine, glycine, alanine, proline, tyrosine, leucine and isoleucine (not shown). Because of difficulties to separately detect all amino acids only food waste 1 was investigated.

The lipid content was between 215 and 252 mg g^{-1} in food wastes 1 and 2, respectively, and 171 mg g^{-1} and 158 mg g^{-1} in street wastes 1 and 2, respectively. The glycerol content was estimated at 10% (w/w) of the lipid content. The fatty acid profiles for food and street wastes were similar. The contents of fatty acids based on the total weight of lipids, however, slightly differed (Table 3).

Table 4 Constituents in organic waste materials based on dry weight

Constituent (mg g^{-1})	Food waste 1[b]	Food waste 2[c]	Food waste 2[b]	Street waste 1[c]	Street waste 1[e]	Street waste 2[c]	Street waste 2[e]
Total-C	495.5 ± 0.7	466.9		481.6 ± 8.8		495.0 ± 2.7	
Total-N	23.8 ± 0.2	24.9		14.1 ± 0.5		36.4 ± 2.2	
Carbohydrates (glucose/fructose/sucrose)	604.3/21.5/19.3	290.6/65.3/18.9	223.3/66.7/35.9	227.9/29.8/10.6[d]	417.3[f]	218.1/6.2/Nd[d]	567.6[f]
Lipid	214.7 ± 12.8	251.5 ± 24.6		170.6 ± 59.3		157.5 ± 17.1	
Glycerol	~ 22	~ 25		~ 17		~ 16	
Protein[a]	133.3 ± 1.1	139.4		78.9 ± 2.8		203.8 ± 12.3	
Dry matter	242.5 ± 0.4	216.9 ± 0.1		461.0 ± 44.2		302.0 ± 88.7	
Ash	60.6 ± 0.6	64.5 ± 0.5		91.8 ± 6.9		61.8 ± 19.0	
Phosphate	–	5.2		–	–	–	–

The contents/yields are based on total weight of reference substances or artificial waste

Nd: not detected; –: not analyzed

[a]Protein content was estimated by multiplying the N-content with 5.6 [14]

[b]Waste material was sequentially hydrolyzed

[c]Waste material was separately hydrolyzed

[d]Carbohydrates hydrolyzed first thermo-chemically and second enzymatically

[e]Carbohydrates hydrolyzed thermo-chemically using 2.5 M H_2SO_4

[f]Only glucose was detected

Composition, Treatment and Functionalized Molecules

The composition of organic waste varies not only between origins, but also due to metabolic activities of microbial consortia, nutritional habits, season and temperature [4, 18, 19]. The same origin may provide organic waste with the same composition of major constituents, the quantity of each constituent, however, can vary and consequently an adaption of quantification methods might be necessary. Food wastes 1 and 2 used in this study, for instance, were collected at the same location, the glucose yield after sequential and separate enzymatic digestion, however, differed by a factor of 2, while the protein and lipid contents were similar (Table 4). In daily routine work it is rather challenging to discriminate between differences in composition due to different waste materials or due to different quantification procedures.

With regard to changing composition and most likely metabolic activity, an assessment of functionalized molecules present in organic material is a tilt at windmills. In this study, which was carried out at lab scale and where materials as well as samples were stored in a refrigerator or freezer, it was difficult to deduce whether differences in quantities should be ascribed to different composition or treatment. It seems more appropriate to estimate from the main constituents: carbohydrate, protein and lipid, which monomers and quantities may appear during storage by hydrolysis and conversion by indigenous consortia. Microbial consortia are active when temperature increases in spring and summer, and convert major constituents, such as carbohydrates, proteins and lipids, into monomers and metabolites. The supernatant of food waste investigated in this study contained significant concentrations of glucose, fructose, sucrose and lactic acid. It is not unusual that food contains free sugars and organic acids, however, the high concentrations found may indicate an active indigenous microbial consortium. Contrarily, street waste was collected in winter when temperature was between 0 and 5 °C, and neither free sugars nor organic acids were detected in supernatants. An active microbial consortium results in a continuous change of composition, which complicates not only the quantification, but also the utilization in the sense of a direct use of secondary raw materials. Organic waste serves as substrate in biomethane and biohydrogen production and methods have been developed to estimate its bioaccessible fraction [4, 18, 19]. A change of the bioaccessible fractions, such as cellulose, hemicellulose, starch, protein and lipids, significantly influences the productivity of those processes, but also the presence of functionalized molecules.

Determining poly- and oligomers in organic waste predominantly bases on degradation towards their monomers. For instance the quantification of starch is based on the chemical or enzymatic degradation and analysis of released glucose [20]. The performance of enzymes, however, is influenced by microscopic phenomena. Lipids may cover carbohydrates and proteins, and prevent them from being hydrolyzed [13]. Another aspect is solubility, since only solubilized undergo hydrolysis [21].

A method which is applicable for the hydrolysis of different materials and to increase yields of all studied hydrolytic products is the thermo-chemical treatment at 121 °C for 15 min and 0.6% (v/v) H_2SO_4 (Table 2). While the application of diluted H_2SO_4 makes more carbohydrates available to enzymes, and thus favors the hydrolysis [22], the application of concentrated H_2SO_4 does result in side-reactions. When street wastes 1 and 2 were sequentially hydrolyzed enzymatically the released products were glucose, sucrose and fructose. The glucose yield was around 220 mg g^{-1} (Table 4). When both wastes were thermo-chemically treated with 2.5 M H_2SO_4, the glucose yield was between 400 and 600 mg g^{-1}. Furthermore, the treatment caused a complete hydrolysis of sucrose to fructose and glucose, and apparently a conversion of fructose into furfural [23, 24], which may further complicate the separation of functionalized molecules.

Different treatments did not only result in different sugar yields, but also in different FAN yields (Table 2). When protein powder was digested first thermo-chemically and afterwards with protease, a seven times higher FAN yield was found compared with the pure enzymatic hydrolysis. In the case of protein powder it was observed that powder was not totally solubilized and clumps were formed which were not completely bioaccessible. Surprisingly, this difference was not found when artificial wastes (Table 2) or food waste 2 (not shown) was treated with or without heat and acid, which may indicate that processed proteins are better water soluble than unprocessed ones.

Investigating the composition of food waste and OMSW is crucial to various research questions. Most study published aim on an understanding of the effect of waste composition on product formation. The essential question thereby is how fast do organic wastes degrade and provide compounds, which can easily be converted into products of interest under given conditions ([25–32], Table 5). One utilization process, which is predominantly under investigation, is anaerobic digestion. It is of interest to the novelty of the present study that the majority of published research focuses on the use of organic waste as substrate in anaerobic digestion. Only one of the studies shown in Table 5 considered the direct use of waste constituents as feedstock in chemical processes. In this study, Li et al. aimed on a use of fatty acids as functionalized molecules in biodiesel production [33].

Even though the quantification of composition of different waste streams shown in Table 5 has been carried out differently to the methods used in the present study, the composition is comparable. This indicates that waste

Table 5 Quantification of composition of different organic waste streams and motivation (aim) for quantification

Substrate	C-content (%)	N-content (%)	Cellulose (%)	Ligno-cellulose (%)	Hemicel-lulose (%)	Carbohydrate (%)	Protein (%)	Lipid (%)	Aim	Refs.
Food waste from a university canteen	49.4	3.5	–	–	–	59.0	18.1	18.0	Improving anaerobic digestion	[25]
Food wastes from different canteen	45.5–51.5	2.6–5.3	–	–	–	3.1–11.0	3.6–7.5	2.9–10.2	Studying the effect of composition on anaerobic digestion	[28]
Mixed flower and vegetable wastes	68.6	3.8	16.1	12.4	21.4	–	13.8	2.7	Improving hydrolysis for anaerobic digestion	[32]
Food waste from a canteen	51.0	4.0	–	–	–	–	–	–	Understanding enzyme activities in anaerobic digester	[26]
Organic municipal solid waste	47.7	3.1	–	–	–	–	–	–	Enhancement of waste degradation	[31]
Organic municipal solid waste	–	–	–	20.2	–	58.6[a]	8.3	6.5	Production of ethanol from organic municipal solid waste	[30]
Kitchen waste	46.1	3.2	–	–	–	–	–	–	Changes in fatty acid composition by thermal treatment	[33]
Kitchen waste[b]	46.1	3.2	–	–	–	11.8	2.5	3.5	Investigating organic degradation during anaerobic degradation	[29]
Food waste from canteens[b]	47.6	3.2	–	–	–	8.9	4.8	5.4	Investigation the effect of composition on anaerobic digestion	[27]

[a]Based on starch

[b]Based on wet weight

streams have a similar composition in common, but hydrolytic products can differ. In order to consider OMSW as a source of functionalized molecules, it is considered here to carry out the characterization using a three levels differentiation scheme (Fig. 1). The totality of organic matter is thereby considered level 1, level 2 stands for carbohydrates, proteins and lipids. The monomers obtainable are glucose, various amino acids, glycerol and fatty acids, respectively, and stand for level 3. Nevertheless, it should be admitted here that level 3 is unpredictable and underlies a continuous change.

Level 3 is associated with a certain unpredictability due to compounds originating from side reactions, metabolic products or not completely hydrolyzed materials. Yet, based on level 2, it might be possible to carry out a "superficial inventory" with an estimation of level 3, such as glycerol from lipids. With starch as starting material, one would expect that only glucose appears after hydrolysis. However, when hydrolysis was carried out firstly thermochemically and secondly enzymatically, 0.8 g glucose per g starch powder was obtained (Table 2), which indicates that a certain amount of the initially applied starch is still present as starch, oligosaccharides or hydrolysis byproducts. An unpredictable fraction is also remaining when protein powder was hydrolyzed. Despite the significant increase in FAN yield after first thermo-chemical and second enzymatic hydrolysis compared with pure enzymatic hydrolysis, there might still be a certain fraction remaining as oligopeptides and/or protein.

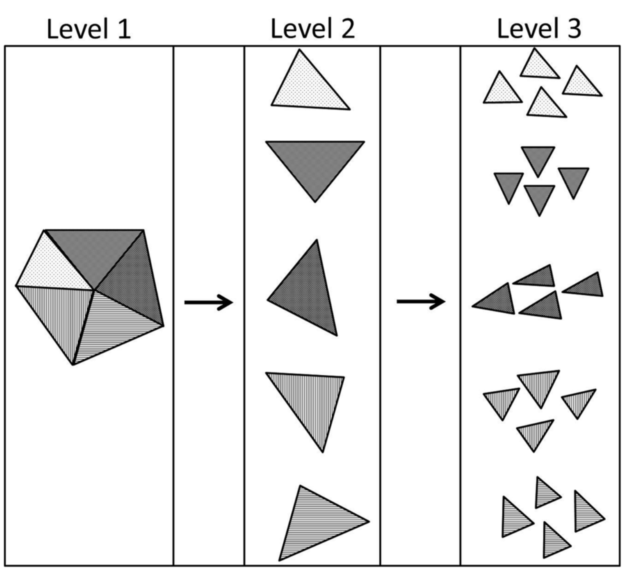

Fig. 1 Levels of characterization. Level 1 stands for the organic content, level 2 represents the organic constituents, such as starch, protein and lipids, and level 3 the monomers obtainable from organic constituents

Practical Implications

Separation of Functionalized Molecules

The complex composition of organic waste challenges a complete utilization [13]. Particularly when biological methods are applied, strategies need to be carefully designed in order to make use of the whole potential. The separation of functionalized molecules and development of tailor made direct conversion strategies, for instance catalytic approaches, for each stream may contribute to efficient and complete utilization of organic waste [34]. However, as discussed above, the heterogeneous and continuously changing composition makes a detailed characterization rather impossible. Therefore, it is recommended to only consider the quantification of carbohydrates, proteins and lipids in organic waste, and theoretical estimation of obtainable functionalized molecules after hydrolysis (Fig. 1).

While the separation of lipids from all other waste constituents is relatively simple due to lower density, the separation of carbohydrates from organic acids and other constituents is not. For instance, Chen et al. studied the separation of glucose, arabinose and xylose from lignocellulosic hydrolysates using cation exchange resin [35]. Using the resin Amberlite IRP69 (Ca^+) they obtained high-purity xylose (88%) from hydrolysate and high-purity arabinose (92%) from a synthetic solution. The hydrolysate contained cellobiose, glucose, arabinose and xylose, short organic acids as well as phenolic compounds. Contrarily, the synthetic solution was less complex and contained only glucose, arabinose and xylose. The separation of functionalized molecules is particularly difficult when structure, size and charge are similar. This, for instance, applies for xylose and glucose. Therefore, Morthensen et al. first converted glucose into gluconate and second applied nanofiltration to separate both [36]. Using a pH of 9.5, 25 °C and 4 bar a throughput of 18.7 L m^{-2} h^{-1} and separation factor of 34 for xylose were obtained. Nanofiltration was also applied by Lyu et al. who separated glucose, monophenols and cyclopentenones as well as acetic acid from hydrolysates of lignocellulosic biomass [37]. They used three nanofiltration modules with different molecular cut-offs in a row in order to achieve the sequential separation. Malmali et al. have also studied nanofiltration for a separation of acetic acid and furfural from biomass hydrolysates. Even though the separation did work, the authors claimed that it is essential to select the right membrane and operation conditions [38]. The question however is, what is the right membrane and operation condition when the composition is continuously changing?

A separation of monosaccharides, organic acids and phenols from hydrolysates of lignocellulosic biomass has been carried out by Chen et al. using the anion and cation exchange resins Amberlyst A21 and Amberlite IR-120, respectively [39]. Using A21 glucose and acetic acid could be separated at purities of 87% and 98%, respectively. The resin IR-120 resulted in a separation of acetic acid and phenol, and purities of 80% and 90%, respectively, were obtained. Even using a real biomass hydrolysate from pine branch the recovery of monosaccharide and organic acid streams were 80% and 88%, respectively. For separating acetic acid and lactic acid in the organic acid stream it was suggested to apply membrane filtration [39].

Due to the promising bioactive properties of peptides obtained after protein hydrolysis, effort has been put on the separation of bioactive molecules from complex mixture. For this purpose electrodialysis with ultrafiltration has been studied for the separation of peptides and charged functionalized molecules like amino acids. According to Suwal et al. this is basically a batch process with one or more filtration membranes stacked into an electrodialytic cell [40]. The separation performance depends on number of membranes, pore size and material as well as pH and electric strength [40]. Electrodialysis with ultrafiltration was successfully tested for the separation of peptides and amino acids from marine protein sources, such as snow-crab by-product hydrolysate [40, 41]. An application of this method for the separation of peptides and amino acids from organic waste hydrolysates is therefore possible and promising approach to upcycle organic waste streams.

The ongoing progress in the field of separation technology may allow the complete and selective separation of functionalized molecules in hydrolysates from organic waste in the future. Nevertheless, due to the continuous changing composition either caused by origin or microbial activities, the applied separation techniques need to be fast, highly flexible and easily adjustable to different waste streams.

Future Work and Strategies

The policy objective of the German government gives an outlook to future research work. The German government aims in its bioeconomy strategy on a complete utilization of all components of biological resources in order to create an independence from fossil raw material suppliers. In order to reach this goal, the development of different conversion processes for primary and secondary refining as well as production of target molecules for relevant industries [42]. Thus, the chance of realization of a direct utilization of OMSW is considered as good when technical drawbacks regarding the separation of molecules are overcome. The chance of realization can further be improved when relevant industries, which apply the recovered secondary raw materials, are involved in the development of new utilization approaches. However, more solid data is needed to proof the predictability regarding quantity and quality of functionalized molecules in OMSW.

The results of the present study revealed that the presence of functionalized molecules changes due to a changing seasonal and spacial composition as well as treatment. Thus, one needs to consider that the composition underlies local differences and data regarding the composition may not be transferable directly. However, a long-term investigation period of several years may result in a data basis which can be transferred to other localities for estimating the presence of functionalized molecules.

Conclusions

From the results of this study it can be concluded that assessment of functionalized molecules in hydrolyzed OMSW has its limitation. Due to the heterogeneous and by time and location changing composition it seems rather impossible to provide a reliable detailed list of functionalized molecules. The question is finding the level of detail until which a characterization makes sense. This level is most likely the quantification of carbohydrates, proteins and lipids in waste material. Based on this level the possibly present functionalized molecules can theoretically be estimated in hydrolysates. Despite the challenges experienced, the potential of organic waste as a source of functionalized molecules is high. It is expected that more attention will be paid to the potential of organic waste in the future beyond its use as substrate in anaerobic digestion, composting or incineration. The matter, however, is finding the right separation technology, which is flexible enough to separate molecules from hydrolysates of varying composition obtained from different OMSW-streams.

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