

Chemical characterization methods for biodegradable organic wastes with relevance for the composting process. Case study

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Received:
02.11.2021

Accepted:
15.12.2021

Published:
17.12.2021

Abstract

The study aimed to develop some methods for the compositional characterization of biodegradable waste with relevance to the composting process at the laboratory level. For testing, four waste fractions were selected, such as waste based on dehydrated sludge, waste from parks (leaves, branches), market waste (mixture of vegetable and fruit) and cardboard waste. For metals and phosphorus, several microwave digestion methods were applied. The ammonium nitrogen content (NH₄-N) was determined using a number of extraction procedures (water, CaCl₂, and KCl). Three certified reference materials were used for testing the validity of the results. Recovery percentages higher than 90% were obtained. The developed and validated methods are suitable for biodegradable waste characterization used in composting processes.

Keywords: biodegradable waste, composting, metals, ICP-MS

INTRODUCTION

Bio-waste is defined as a complex mixture of different proportions: biodegradable garden and park waste, household food and kitchen waste, restaurants, catering and retail space and similar waste from food processing plants [1].

The main environmental problem induced by biodegradable waste is methane emission into the air during the decomposition of waste deposited in municipal landfills. In the EU, from total greenhouse gas emissions accounted for in 2019, approx. 9% are associated with biodegradable waste. Directive EU 2018/851 states that the Member States must ensure by the end of 2023 that bio-waste is either separated and recycled at the source or collected separately and not mixed with other types of waste. Also, according to the same Directive, the Commission calls on European standardization organizations to develop European standards for the bio-waste subject to organic recycling, compost, and fermented product, based on best available practices [2]. The amount of waste that undergoes aerobic or anaerobic treatment may be considered as recycled, provided that such treatment generates materials to be used as recycled products, materials, or substances [2].

From 1 January 2021, local public administration authorities or the administrative-territorial subdivisions of municipalities and their inter-community development associations, respectively, must implement the system of separate collection of biodegradable waste. This action was imposed in order to reduce the separate door-to-door collection of bio-waste in urban areas, doubled by the implementation of the "pay for how much you throw away" scheme and encouraging individual composting in rural households [3].

A first step in recycling biodegradable waste by composting is the characterization of waste suitable for composting. Therefore, appropriate methods for the analysis of critical compounds in biodegradable waste must be identified [3-5].

In the fermentation process, trace elements, such as copper and zinc, and soluble forms of magnesium, potassium, and calcium are essential for microbial growth. [5-6]. Metal ions are essential for microbial growth, and similarly, with other nutrients such as nitrogen and phosphorus, they influence the specific growth rates of bacteria [5-12].

The advantages of composting biodegradable waste are the following: reducing waste, recycling nutrients in soil as fertilizers, reduction of pollutants from the surrounding air, prevent climate change, soil water protection and preventing soil erosions [7-17].

Some examples of analytical techniques and digestion methods for heavy metals determination from biodegradable waste are presented in Table 1.

Table 1. Procedures used for heavy metals determination from waste – reported data

Sample waste	Analytical techniques/ Metals	Method of digestion/ Reagents	Remarks	Ref.
Food wastes: including brewer's spent grain, apple pomace, strawberry pomace, carrot pomace, orange and grapefruit peel, beetroot and potato peel, potato pulp, rapeseed cake, walnut and, hazelnut shells	AAS/ Pb, Cd, Cu, Zn, Cr, Ni, Na, K, Mg	<i>Microwave - closed system</i> 1g sample 7 mL HNO ₃ (65%) + 1 mL H ₂ O ₂ (30%)	Certified reference material (BCR-482, IRMM) was used to validate the analytical measurements. The closed microwave digestion method is adequate and accurate for determining Pb, Cd, Cu, Zn, Cr, Ni, Na, K, Mg in food waste.	[6]
Biomass waste coming from agriculture, forestry, and related industries (waste horticultural and wine)	ICP-MS/ AAS/ Al, Mg, Ca, K, Na, Fe	<i>Microwave - closed system</i> 0.25g -1g sample 8mL HNO ₃ +2mL H ₂ O ₂	The concentration obtained for Al, Mg, K, Ca, Na and Fe by microwave digestion in a closed system analyzed using the ICP-MS technique were better than the AAS technique.	[7]
Biomass waste coming from agriculture, forestry and food industries	ICP-MS/ As, Cd, Pb	<i>Microwave - closed system</i> 0.5g ash+10mL HNO ₃	How the ash sample is prepared from the biomass is therefore crucial for obtaining reliable and reproducible results.	[8]
Sewage sludge	AAS/ Cd, Cu, Cr, Pb, Ni, and Zn	a) <i>Microwave - closed system</i> 0.5g dried sample (105°C) + 6mL HNO ₃ +2mL HCl b) <i>Microwave - closed system</i> 0.5g roasted sample (600°C) + 6mL HNO ₃ + 2mL HCl c) <i>Wet acid digestion open system</i> 1g dried sample + 12mL HNO ₃ +4mL HCl	Generally, method (b) was the most effective. Methods (b) and (c) yield complete decomposition of the matrix. As a result, the precision of measurement substantially increases. Microwave acid digestion in a closed system allows decomposing samples more quickly than with conventional mineralization.	[9]

The study aimed to develop and implement some characterization methods of biodegradable waste relevant to the composting process.

MATERIALS AND METHODS

Equipment

Millipore Milli-Q Ultrapure Water System
Microwave Digestion System Ethos Up Milestone
ICP-MS type 7900 Agilent with Mass Hunter 4.4 software
AAS Perkin Elmer PinAAcle 900T
UV-VIS type 205 Specord Analytic Jena

Reagents and calibration

Calibration curves for As, Cd, Cr, Ni, Cu, Zn, Pb, K were performed using a Multielement CRM (100 mg/L), traceable to NIST, containing 21 elements. Quality control of the analytical results was performed with a Multielement standard (100 mg/L) containing 23 elements, from Sigma Aldrich.

The sodium salicylate, sodium citrate dihydrate, sodium nitroprusside, sodium dichloro isocyanurate, sodium hydroxide, HCl and, H₂SO₄ used for the UV-VIS spectrophotometric method were purchased from Sigma Aldrich. To achieve the NH₄-N calibration curve, was used ammonium chloride of 1000 mg/L (Sigma Aldrich quality). All the reagents used in the experiments were of quality grade.

For in-house validation experiments were used three different certified reference materials (CRMs) traceable to NIST, with ammonium nitrogen, total phosphorous, and metals content: sewage sludge (LRAC 4000), tomato leaves (1573a), and apple leaves (1515). In order to assess and develop the method, four types of waste were chosen: waste based on dehydrated sludge (P1); waste from parks (leaves and branches) (P2); market waste (mixture of vegetable and fruit scraps) (P3); cardboard waste (P4).

The methods and sample preparation

This complex study aimed at characterizing specific waste fractions in terms of metal content, total phosphorus, and ammonium nitrogen and the implementation at the laboratory level of the tested analytical methods. The samples preparation consisted in samples drying at 105°C and grinding at 40mm. Four digestion methods were applied to determine the total metals and phosphorus content (Table 2). For ammonium nitrogen content determination were applied three extraction method: in water, in CaCl₂ and in KCl.

The minor elements (As, Cd, Cr, Ni, Cu, Zn, Pb) from the waste fractions were determined using the ICP-MS technique in the concentration range 10÷50µg/L. The total phosphorus content was determined using the ICP-MS technique in the range of 100÷500µg/L. Total potassium was determined using the AAS technique in the range of concentrations 1÷5mg/L. The matrix effect-removing agent for the AAS technique was 1% LaCl₃. Ammonium nitrogen (NH₄-N) was determined using UV-VIS technique in the range of concentrations 5÷40 µg/L N. The waste fractions were dried at 105°C and crushed under 40mm. The same sample preparation methods were applied for CRMs.

Digestion step

After the drying stage at 105°C and calcination at 550°C, the samples were further mineralized according to Table 2. The methods were adapted on the literature data presented in the introduction and according to the specific standards for bio-waste and sludge.

Table 2. Digestion methods

Waste fraction	Sample weight, g	Method	Steps	Power, W	Temp., °C	Time, minutes
P1- Waste based on dehydrated sludge	0.25-0.50	I Method	1	1600	160*/100**	30
		7.5mL HCl + 2.5mL HNO ₃	2	1600		35
			Cooling		-	-
P2- Parks waste (leaf-branches)	0.50-1	II Method	1	1400	120*/100**	20
		9mL HNO ₃ + 1mL H ₂ O ₂	2	1400		25
			Cooling		-	-
P3 - Market waste	0.50-1	III Method	1	1500	140*/100**	25
		8mL HNO ₃ + 2mL H ₂ O ₂	2	1500		30
			Cooling		-	-
P4- Cardboard waste	0.50-1	IV Method	1	1200	110*/100**	15
		10mL HNO ₃	2	1200		25
			Cooling		-	-

* The temperature inside the vessels controlled by a temperature sensor in the reaction vessel 1;

** The temperature on the outside of the vessels controlled by IR sensor.

Analyses

The metals As, Cd, K, and total phosphorus were determined from the dried solid sample [18].

Moisture content: P1 (82.88%); P2 (98.13); P3 (97.56) and P4 (97.12%).

The other metals (Pb, Cr, Cu, Ni, Zn) were determined from the sample residue (ash) obtained by samples calcination at 550°C [24]. Residue (ash) content: P1 (50.89%); P2 (12.82); P3 (19.19) and P4 (3.26%).

In-house validation method for the determination of NH₄-N

Three extraction methods were used to determine ammonium nitrogen content (NH₄-N), as can be seen in Figure 1. The optimal method for the determination of ammonium nitrogen in the waste samples was validated in-house. The repeatability, intermediary precision, quantification limit (LOQ), detection limit (LOD), recovery and expanded uncertainty were evaluated.

A five-point calibration curve was drawn for the ammonium nitrogen content from a 1mg/L ammonium nitrogen standard solution: 5µg, 10µg, 20µg, 30µg, 40µg, using UV-VIS technique at 655nm wavelength. The correlation factor (R²) was 0.9998. To verify the ammonium nitrogen method a certified reference material for sewage sludge was used.

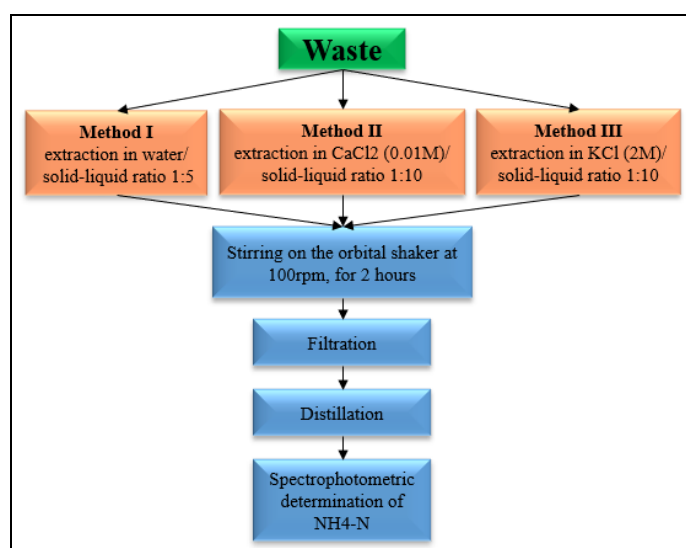


Fig. 1. Scheme for determining the ammonium nitrogen (NH₄-N) content in waste

The tested performance parameters of the study are presented in Table 3.

Table 3. The in-house validation experiments for NH₄-N

Parameters	Experiments
LOD and LOQ	10 independent fortified sample solutions with 2µg NH ₄ -N
Repeatability	10 independent fortified sample solutions with 15µg NH ₄ -N
Intermediate precision	12 independent fortified sample solutions with 15µg NH ₄ -N
Recovery	5 independent fortified sample solutions with 25µg NH ₄ -N

The extraction methods for ammonium nitrogen were performed according to the standardized methods [19-21]. With one minor exception, the samples were stirred on Shaker 2h instead of one hour and at a stirring speed of 100rpm.

The determination of ammonium nitrogen was performed by using a UV-VIS method at 655nm [22-24]. All samples were filtered, distilled, and the appropriate method for determining ammonium nitrogen was applied.

RESULTS AND DISCUSSION

In-house validation for NH₄-N

Table 4 presents the values of the performance parameters obtained for in-house validation of NH₄-N method using the UV-VIS technique and digestion method III. Validations for total phosphorus and metals content were reported in previous studies [13, 14].

Table 4. Performance parameters for NH₄-N, after application of 2M KCl extraction

Sample	LOD, mg/kg	LOQ, mg/kg	RSD _r *, %	RSD _R ***, %	Recovery, %	U _{ex} ***, %
P1	0.012	0.039	2.25	2.61	98.4	11.3
P2	0.020	0.066	1.94	2.36	97.6	10.5
P3	0.031	0.100	2.50	2.79	91.2	12.6
P4	0.023	0.080	2.43	2.64	92.8	11.5

*Repeatability test; **Intermediate precision; *** Expanded uncertainty

The method for NH₄-N analysis from the waste is precise, accurate, presenting small detection and quantification limits (Table 4). The recovery yield of NH₄-N is over 98% for dehydrated sludge. For the rest of the samples, the recovery yield is P2 (97.6%), P3 (91.2%), and P4 (92.8%).

The lowest limits of detection and quantification were recorded for sample P1 (dehydrated sludge) and the highest for sample P3 (market waste). The results obtained were influenced by each sample composition, the extraction time, and the stirring speed. For the calculation of the expanded uncertainty the following parameters were taken into account: standard deviation of repeatability, the standard deviation of reproducibility, purity of reagents, the uncertainty of laboratory glassware, CRM uncertainty for sludge, and CRMs uncertainty for biomass, the measuring range (5÷40 µg NH₄-N) and uncertainty of UV-VIS equipment. The lowest uncertainty value was obtained for the P2 (10.5%) sample composed of park waste, and the highest measurement uncertainty was obtained for the P3 (12.6%) sample composed of market waste. The validation method for cellulosic waste (P2, P3 and P4) was performed on real waste samples due to the lack of a significant CRM for the ammonium nitrogen content.

Determination of NH₄-N using three extraction methods

Three different extraction methods were applied to a reference material with certified values for ammonium nitrogen (sewage sludge LRAC 4000). The results obtained are presented in Table 5. The certified reference material (CRM) demonstrated the accuracy of the method and the reproducibility of the results for extraction method III.

Table 5. Ammonium nitrogen (NH₄-N) content in LRAC 4000 CRM, mg/kg d.m.

Parameter	Method	Certificate value	Obtained value	Recovery Yield (%)
NH ₄ -N	Method I	4700±457	3956±633	84.2
	Method II	4700±457	4216±594	89.7
	Method III	4700±457	4617±462	98.2

± expanded uncertainty

Based on the results obtained, it can be stated that the KCl extraction method is better compared to the other two methods (extraction in water and CaCl₂). The result obtained for ammonium nitrogen falls within the measurement uncertainty of the method and certificated value. The value of ammonium nitrogen recovery by the KCl extraction method (98.2%) was higher compared to the other two extraction methods (Table 5). In addition, the ammonium nitrogen content of the sludge sample by extraction method III is higher than the other methods (Table 5).

Table 6. Ammonium nitrogen (NH₄-N) in samples, mg/kg d.m.

Sample results	Method I	Method II	Method III
P1	30.64±4.90	23.81±3.33	69.87±6.99
P2	1.556±0.25	1.209±0.17	3.439±0.34
P3	19.206±3.07	14.923±2.09	38.03±3.80
P4	0.415±0.066	0.322±0.045	0.755±0.076

± expanded uncertainty

As can be seen from Tables 5 and 6, the highest ammonium nitrogen content is found in the P1 (dehydrated sludge) sample, followed by the P3 (market waste) sample. For all the samples, the highest results were obtained using extraction method III (extraction in KCl) and, the lowest results were obtained using method II (extraction in CaCl₂). The extraction methods for ammonium nitrogen depend on the waste composition, sample pH, drying temperature, and sample extraction procedures.

Determination of total phosphorous content

To determine the total phosphorous, four digestion methods were used for organic matter decomposition. Digestion methods are described in Table 2. Total phosphorus validation procedures have been extensively presented in another article [14].

The analysis method was verified with three certified reference materials (CRM) with known total phosphorus content: apple leaves, tomato leaves and sewage sludge.

Table 7. Total phosphorus from sewage sludge LRAC 4000 CRM, mg/kg d.m.

Parameter	Method	Certificate value	Obtained value	Recovery Yield (%)
Total phosphorous	Method I	2.11±0.260	2.00±0.300	94.79
	Method II	2.11±0.260	2.03±0.274	97.16
	Method III	2.11±0.260	1.91±0.287	90.52
	Method IV	2.11±0.260	1.80±0.270	85.30

± expanded uncertainty

Table 8. Total phosphorus in tomato leaves (1573a) and apple leaves (1515) CRM, mg/kg d.m.

Method	1573a			1515		
	Certificate value	Obtained value	Recovery, %	Certificate value	Obtained value	Recovery, %
Method I	2161±28	2135±203	98.80	1593±68	1532±130	96.17
Method II	2161±28	2138±203	98.94	1593±68	1540±131	96.67
Method III	2161±28	2134±186	98.75	1593±68	1529±133	95.98
Method IV	2161±28	2133±190	98.70	1593±68	1525±136	95.73

± expanded uncertainty

The digestion method II in the presence of nitric acid and hydrogen peroxide presents highest results for the determination of total phosphorus content for all samples. In addition, applying the digestion method II, the highest recovery yields (higher than 96%) of the total phosphorus were obtained from the three certified reference materials, both for the sludge matrix and for the biomass matrix (tables 7 and 8).

Table 9. Total phosphorus content in samples, mg/kg d.m.

Sample results	Method I	Method II	Method III	Method IV
P1	124±11.78	135±10.13	121±7.87	116±8.47
P2	32±3.04	38±2.85	31±2.02	25±1.83
P3	89±8.46	96±7.20	81±5.27	72±5.20
P4	0.12±0.011	0.18±0.014	0.09±0.006	0.05±0.004

± expanded uncertainty

Not only for CRMs higher results were obtained but also for all samples prepared by digestion method II, as it can be seen in Table 9.

Applying methods I, III and IV for the digestion of samples in a closed microwave system, lower total phosphorus recovery yields were obtained for each waste fraction. The total phosphorus content was influenced by the following parameters: the composition of the waste, the mixture of acids for mineralization, as well as the temperature and microwave power of the digestion stage. These parameters contributed to the oxidation of all forms of phosphorus (polyphosphates and organic phosphorus) from waste samples to total phosphorus.

The dehydrated sludge sample contains humic acids, while vegetable samples and cardboard waste contain cellulose, hemicellulose and many colored substances such as tannins, polyphenols and flavones that must be eliminated in digesting step. These factors influenced the results for the total phosphorus content. Depending on the sample matrix for which it is desired to determine the total phosphorus content, the digestion method is subsequently selected, taking into account the composition of the sample.

Determination of total metals content

The most important aspects of the study were the preparation and the digestion methods of all waste fractions. At the end of the study, the best digestion and extraction methods were selected for each of the waste fraction. The metals were analyzed both on the dried samples (As, Cd, K) and from the residue (ash) (Cr, Ni, Cu, Zn, Pb) obtained by calcination at 550°C.

In order to ensure the validity of the results obtained for sewage sludge (P1), park waste (P2), market waste (P3) and cardboard waste (P4), three certified reference materials were used (sewage sludge LRAAC4000, leaves of tomatoes 1573a and apple leaves 1515). The CRM for tomato leaves does not contain lead, and the CRM for apple leaves does not contain chromium and arsenic. The CRMs results obtained by the four digestion methods are presented in Tables 10 and 11.

Table 10. Total metals content from CRMs, mg/kg d.m.

Metal	LRAAC 4000 (Method I)			1573a (Method II)		
	Certificate value	Obtained value	Recovery, %	Certificate value	Obtained value	Recovery, %
As	256±9	248.5±29	97.07	0.1126±0.0024	0.1102±0.0121	97.87
Cd	122.7±0.26	122.5±12	99.84	1.517±0.027	1.512±0.151	99.67
Cr	353±12	345±39	97.73	1.988±0.034	1.986±0.238	99.91
Ni	127±4	125±15	98.43	1.582±0.041	1.575±0.189	99.56
Cu	736±33	729±95	99.05	4.70±0.14	4.64±0.46	98.72
Zn	1080±20	1076±140	99.63	30.94±0.55	29.97±3.30	96.86
Pb	119±5	117±13	98.49	-	-	-
K	4918±975	4875±585	99.13	26760±480	26751±2408	99.97

± expanded uncertainty

Based on the implementation of the method of metal content analysis we can say that the results obtained are reproducible (tables 10 and 11). The highest recovery yield was obtained for cadmium (99.84%) while the lowest one for arsenic (97.07%). The highest concentrations of metals were recorded for potassium from dehydrated sludge, while the lowest concentrations were recorded for cadmium. The uncertainty of the working method was applied to each result obtained.

Table 11. Total metals content from 1515 CRM, mg/kg d.m.

Metal	Certificate value	Method III		Method IV	
		Obtained value	Recovery, %	Obtained value	Recovery, %
Cd	0.0132±0.0015	0.0129±0.0013	97.73	0.0125±0.00125	94.70
Ni	0.936±0.094	0.929±0.1115	99.25	0.918±0.1102	98.08
Cu	5.69±0.13	5.63±0.563	98.95	5.60±0.560	98.42
Zn	12.54±0.43	12.37±1.36	98.64	12.37±1.36	98.64
Pb	0.470±0.024	0.462±0.0462	98.30	0.462±0.0462	98.30
K	16080±210	16069±1446	99.93	16069±1446	99.93

± expanded uncertainty

For market waste and cardboard waste, the working method was verified with a certified reference material based on apple leaves (1515) by two methods of digestion corresponding to the studied matrices. By method III of digestion in the presence of nitric acid and hydrogen peroxide, a higher metal content was obtained for the CRM in apple leaves compared to method IV of digestion in the presence of nitric acid. Zinc, lead and potassium were not influenced by the applied digestion method, only cadmium, nickel and copper showed higher results by digestion method III (table 11). The metal recovery were situated in the range 97.73% to 99.93% for method III and from 94.70% to 99.93 for digestion method IV (Table 11). Digestion temperature and microwave power also influenced the results obtained for the total metal content. The results obtained for the metal content of the three CRMs (sewage sludge, tomato leaves and apple leaves) fall within the measurement uncertainty of the analytical method.

The results obtained for the total metal content of all the studied samples are presented in Table 12.

Table 12. Results obtained for total metals for all the samples, mg/kg d.m.

Metal	P1 (Method I)	P2 (Method II)	P3 (Method III)	P4 (Method IV)
As	42.2±5.06	2.11±0.23	3.40±0.374	1.33±0.146
Cd	1.08±0.108	0.70±0.070	0.74±0.074	0.63±0.063
Cr	32.66±4.25	0.37±0.044	15.71±18.9	2.74±0.329
Ni	8.30±0.83	13.14±1.58	13.14±1.58	2.24±0.269
Cu	23.81±3.10	1.77±0.18	11.08±1.11	3.67±0.367
Zn	163±21.19	8.79±0.97	42.91±4.72	8.33±0.916
Pb	9.43±1.04	0.25±0.025	16.72±1.67	6.93±0.693
K	1995±239	507±46	1871±168	350±32

± expanded uncertainty

For each waste fraction, the optimal digestion method was applied to correctly determine the metal content. Initially, an attempt was made to apply each digestion method for the four waste fractions and subsequently the optimal digestion method was selected for each waste category. Table 12 show the optimal digestion methods for the four categories of waste, for which the highest metal concentrations and the most reproducible results were obtained.

The highest concentrations of metals were obtained for dehydrated sludge, followed by market waste, park waste and cardboard waste as it can be seen in Table 12. Dehydrated sludge (P1) recorded high values for As, Cd, Cr, Ni, Cu, Zn, K, with an exception for lead which is present in small quantity (9.43 mg/kg d.m) in comparison with the lead from the market waste (16.72 mg/kg d.m). The cardboard waste presents a small exception for the lead concentrations which has a high content (6.93 mg/kg d.m) in comparison with the lead from the park waste (0.25 mg/kg d.m).

CONCLUSIONS

The study consisted in the development and implementation of methods for compositional characterization of biodegradable waste with relevance for the composting process. The minor and major elements in the composition of four waste fractions were analyzed by several analytical techniques: ICP-MS, AAS and UV-VIS for the subsequent implementation at laboratory level. The parameters followed in this study were the following: sample preparation methods (drying and grinding), digestion and extraction methods and subsequently analytical techniques.

The method developed for ammonium nitrogen using UV-VIS technique has been validated internally. The highest recovery for ammonium nitrogen (98%) were obtained using method III (extraction in KCl), followed by method I (extraction in water) and method II (extraction in CaCl₂). The results obtained for the ammonium nitrogen, total phosphorus and metals content were compared with certified reference materials (CRM) to verify the validity of the experimental results. For all investigated parameters, the results are accurate, precise and reproducible.

For the metals content, recovery yields of over 97% were obtained. The best results for the metals content and for the phosphorus content were obtained using the digestion method II in the presence of nitric acid and hydrogen peroxide at a 120°C digestion temperature. Microwave acid digestion in a closed system allows decomposing samples more quickly than that with conventional mineralization and is suitable for volatile metals, which are no longer lost during the digestion stage.

The methods tested and implemented in this study for different waste fractions will help to select the optimum “secondary raw materials” for the composting stage. It is known that waste has characteristics and chemical composition that can change during composting conditions, the methods developed and presented in this paper are a useful tool for characterizing the different waste fractions that enter in the composting stage as well as the final compost after the anaerobic stage of fermentation

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Citation: Catrina, G.A., Kim, L., Serbanescu, A., Cernica, G., Bumbac, C., Sburlis, M., Chemical characterization methods for biodegradable organic wastes with relevance for the composting process. Case study, *Rom. J. Ecol. Environ. Chem.*, **2021**, 3, no.2, pp. 152-161.



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