CONAMA 2024
CONGRESO NACIONAL DEL MEDIO AMBIENTE

De biorresiduos urbanos a fertilizante y enmienda de suelo: el potencial del biochar a partir de rechazos de compostaje

From Urban Biowaste to Fertilizer and Soil Amendment: The Potential of Biochar from Composting Rejects

FROM URBAN BIOWASTE TO FERTILIZER AND SOIL AMENDMENT: THE POTENTIAL OF BIOCHAR FROM COMPOSTING REJECTS

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1. RESUMEN (ES) / EXECUTIVE SUMMARY

El presente estudio tiene como objetivo evaluar la aptitud de los rechazos de compostaje provenientes de la planta de digestión aerobia de LIPOR, empresa de gestión de residuos del área metropolitana del Gran Oporto que sirve a una población de 1 millón de personas, para la producción de biochar destinado a uso agrícola. A través de un análisis termogravimétrico se determinaron las condiciones óptimas de producción de biochar frente a gases y aceites de pirólisis, abarcando temperaturas de entre 200°C y 700°C. Asimismo, se caracterizaron las cenizas, los sólidos volátiles y totales, el carbono fijo, la humedad y el poder calorífico del biochar obtenido de las 5 muestras entregadas.

En una segunda fase, habiendo identificado las mejores condiciones de proceso en temperaturas de 300ºC, 350ºC y 400ºC y rampa de 5°C/min, se han generado 3 muestras de biochar producidas de la mezcla de las 5 muestras de rechazos entregadas. El biochar se ha caracterizado en términos de micro y macronutrientes así como de hydropfobicidad. Los resultados inherentes a la concentración de nitrógeno son muy prometedores, siendo esta de entre 2.05% y 2.38%, o sea por encima del mínimo exigido para fertilizantes orgánicos sólidos por el reglamento antes mencionado (PFC 1(A)(I)). Esta concentración de nitrógeno no es habitual en el biochar comercial y, junto a las concentraciones de potasio y de fósforo detectadas, muestra claramente el potencial del biochar procedente de rechazos de compostaje no solo como enmienda orgánica, sino como aporte de nutrientes al suelo.

2. INTRODUCTION (EN)

Many European cities and regions have been engaged in the valorisation of municipal biowaste for decades. However, this practice continues to require R&D solutions to prevent urban biowaste ending up in landfills. Improving the efficiency and yield of biowaste valorisation schemes has become even more critical since January 1, 2024, when the amendment to the Waste Framework Directive (EU 2008/98/EC, Article 22) mandated the selective collection of biowaste across all EU municipalities.

Composting remains the most common method for biowaste valorisation. [1] This process generates secondary waste streams of varying particle sizes and a moisture content. Composting rejects typically consist of sieved-off particles, primarily small branches, wood fragments, and variable fractions of plastics, glass, and metals, depending on the biowaste pre-treatment and composting processes. Unfortunately, these rejects are often sent to landfills or incinerated, resulting in the loss of their high carbon content and the missed opportunity to return it to the soil. The size and humidity of these side streams render them incompatible with composting process specifications. Nonetheless, they are highly suitable for alternative value-added valorisation routes.

Pyrolysis, the decomposition by heating of an organic material in the absence of oxygen, offers a promising alternative. During the pyrolysis process, hemicellulose breaks down at 200–250 ºC, cellulose decomposes between 240–350 ºC, and lignin undergoes pyrolysis at 280–500 ºC. In general, wood pyrolysis requires temperatures of at least 300–375 ºC [2][3] and its performance at moderate temperatures (below 500 ºC) shifts the thermodynamic balance toward the production of a solid phase: biochar. Biochar can have different applications but for the purpose of this work we focus on its agronomic properties. Actually, biochar enhances soil physical, chemical, and biological properties, contributing significantly to soil health.[4] Additionally, depending on the chemical composition of the input biowaste and the pyrolysis conditions, biochar can provide valuable nutrients.

This study's objective is to explore the suitability for agronomic applications of biochar obtained from composting rejects from selectively collected municipal biowaste. The rejects were sourced from LIPOR's composting facility in Baguim do Monte, Portugal. LIPOR is the public waste management company of Greater Porto Area and their facility processes biowaste from approximately one million households, the HoReCa sector and green areas. Their product is Nutrimais®, a high-quality compost very demanded by the local primary sector and innovation in nutrient recycling is one of LIPOR's key R&D priorities.

To the best of our knowledge, no previous works assess the agronomic properties of biochar from the same feedstocks, instead focusing on other feedstocks, mainly wood and food industry byproducts, like cereal straw or sawdust. This work provides insight into how pyrolysis temperature and operating conditions influence some properties and yield of biochar for agronomic applications.

3. EXPERIMENTAL PROCEDURE

5 samples of compost refuse, grabbed from 5 different locations of the composting tunnels were divided into quarters following the procedure described in the UNE-EN 15413 standard, see Figure 1. Samples were characterised individually in terms of material composition, immediate analysis, elemental composition and calorific values. The 5 samples where later submitted to TermoGravimetric Analysis (TGA) to evaluate weight loss with temperature and heating rate. In a second stage, the samples were mixed up and lab-scale biochar batches were produced and characterized. More details on the experimental procedure are reported here below.

Figure 1. manual division in quarters of the samples of compost rejects

Characterization of the materials composition. Each quarter was characterized according to the composition of 13 different fractions (results in Table 2). Metal particles and seashells fragments were set aside to avoid damages to the mills.

Pretreatment of the 5 compost refuse samples. Due to the morphological characteristics of the sample, mechanical pre-treatment was necessary to adapt it to the needs of the experimental equipment. It consisted of crushing and grinding. The grinding was carried out in two phases, i) grinding with a stainless-steel cutting mill up to 4 mm, and then ii) with a centrifugal mill a size of 2 mm was achieved.

Characterization of compost rejects. Thermogravimetric analysis (TGA) of each pretreated sample was performed to evaluate weight loss rate and collect data on ashes, volatile solids, total solids, fixed carbon, humidity. Experiments were performed in quadruple replicates at a temperature range of 30ºC – 950ºC (as per European Biochar Certificate directions). Calorific value and elemental composition of all samples were performed to foresee the behaviour of the feedstock at different pyrolysis temperatures (T), and to estimate the energy balance of the pyrolysis process.

Evaluation of the effect of compost refuse composition on the yield of biochar. The 5 pre-treated compost refuse sample underwent a TGA to determine the rate of weight loss of the sample as a function of temperature and heating rate in an inert atmosphere (N2) to simulate pyrolysis conditions. In each test, a quadruplicate of the 5 rejection fraction samples sent by LIPOR was analysed. Explored conditions were 5 Heating temperature (T = 200°C, 300°C, 400°C, 500°C, 700°C) and 3 heating rates (ΔT = 2°C/min, 5°C / min, 10°C/min, being the latter chosen as per European Biochar Certificate directions) [5]. 75 TGA curves and biochar yield data (5 samples x 5 temperatures x 3 heating rate) were obtained to get information on the operating conditions to be chosen for the pyrolysis laboratory tests.

Lab scale production of 3-9 biochar batches and characterization. Based on the results of the previous phase, 3 operating conditions with different T and holding times are chosen for 1 compost refuse samples. This sample is a mixture of all grounded samples characterised in the previous phases that was crushed to a granulometry of 2 mm and dried in an oven to 85% dry solid (15 % humidity). ΔT was set on 5 °C/min.

- Test A - HOOP. Temperature: 300°C; Heating rate: 5°C/min; Time at T300: 30 min.
- Test B - HOOP. Temperature: 350°C; Heating rate: 5°C/min; Time at T350: 60 min.
- Test C - HOOP. Temperature: 400°C; Heating rate: 5°C/min; Time at T400: 90 min.

Table 1 summarizes the control software programming data of the experimental equipment.

Table 1. Test conditions

Test reference	Test A	Test B	Test C
Stage 0 - Time (min)	10	10	10
Stage 0 - T (°C)	25	25	25
Stage 1 - Time (min)	55	65	75
Stage 1 - T (°C)	25 - 300	25 - 350	25 - 400
Stage 2 - Time (min)	30	60	90
Stage 2 - T (°C)	300	350	400
Ramp (°C/min)	5	5	5
Max pressure (bar)	1	1	1

The amount of sample was limited by the volume of the reactor (600 mL), which must be filled to a maximum of 2/3 of its capacity (400 mL). Approximately 115 g of compost refuse were processed per experiment (density = 0.290 g/mL).

The **pyrolysis yield** intended as the weight distribution of oil, gas and biochar, was measured as the percentage by weight of each of the products obtained based on the initial weight of the sample.

In the last phase of this work, the biochar was characterised as follow:

- Moisture – In accordance with UNE-EN ISO 18134-3:2016 by the indirect drying method.
- Volatile matter – In accordance with UNE-EN ISO 18123:2016 standard, using a muffle furnace HD-230 PA.
- Ashes – In accordance with UNE-EN ISO 18122:2016 standard, using a muffle furnace HD-230 PA.
- Elemental composition (C, H, N, S, Cl) – The elemental composition of the biochar samples was examined through Energy Dispersive Spectrometer.
- Gross calorific value and Net calorific value – By the bomb calorimetric (Parr-SERIE) in accordance with UNE-EN ISO 18125:2018
- Hydrophobicity – drop permeation test
- Metal micro-nutrients – Inductively Coupled Plasma Optical Emission spectroscopy (ICP-OES) Thermo Fisher Serie 6500.
- Micronutrient (Ca+, K+, Fe+, Na+) – Ion chromatography.

As per the gas and liquid phases, the quantities obtained were insufficient to perform a characterization of the elemental composition and the calorific value.

The **energy consumption** of each batch production was estimated from the electrical consumption of the reactor.

4. RESULTS

Characterization of compost rejects.

As shown in Table 2, the fraction 'Putrescible waste - garden waste' constitutes the majority in all samples, with an average of 90.94±2%. The total amount of materials of biological origin, calculated as the sum of this fraction and the 'fine fraction,' averages 94.34% across the five samples. Notably, the samples exhibit high homogeneity, with the largest variation observed in the 'Unspecified combustibles – Bones' fraction, where sample 5 (3.13%) shows double the average of the total set. Figure 2 shows examples of some of the identified fractions.

Table 2. Material composition of the 5 compost reject samples

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Putrescible waste - Garden waste	90.60%	93.85%	88.99%	91.49%	89.79%
Paper / cardboard	0.00%	0.00%	0.00%	0.00%	0.00%
Composites	0.00%	0.00%	0.00%	0.00%	0.00%
Textiles	1.02%	0.11%	0.00%	0.00%	0.00%
Plastics	1.63%	0.87%	2.79%	1.12%	1.15%
Wood	0.00%	0.00%	0.00%	0.00%	0.00%
Glass	0.27%	0.06%	2.92%	0.15%	0.00%
Metals	1.34%	0.00%	2.50%	0.28%	0.74%
Unspecified combustibles - Bones	0.36%	0.87%	0.00%	1.64%	3.13%
Unspecified non-combustibles - shells	2.44%	0.26%	0.00%	2.34%	0.63%
Hazardous waste	0.00%	0.00%	0.00%	0.00%	0.00%
Other waste	0.00%	0.00%	0.00%	0.00%	0.00%
Fine fraction (20 mm)	2.34%	3.99%	2.80%	2.98%	4.55%

Figure 2. Image of the different fractions of the sample 1

After the material composition characterization, samples were crushed to 2 mm and characterized by immediate analysis, elemental analysis and calorific value. The results are collected in Tables 3, 4 and 5 and again highlight he homogeneity of all the samples. Moreover, it has been observed that the reported data are like those of biomass obtained from agricultural activities.

Table 3. Immediate analysis

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Moisture - Uncrushed (%)	43.09	40.98	41.59	39.53	38.67
Moisture - Crushed to 2 mm (%)	39.82	34.94	32.91	32.80	25.57
Volatile matter, d.w. (%)	56.50	58.92	61.17	59.13	64.10
Ash, d.w. (%)	19.99	18.59	16.01	17.79	11.84
Fixed carbon, d.w (%)	23.51	22.48	22.82	23.08	24.06

Table 4. Elemental composition

Elements	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
C (%)	62.70	60.75	58.75	58.32	61.25
H (%)	3.00	2.66	2.58	2.46	2.33
N (%)	8.46	8.38	8.09	8.11	7.97
O (%)	5.85	9.61	14.57	13.32	16.62

Table 5. Calorific values

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Gross calorific value, d.w. (*) (kJ/kg)	26,831	26,961	26,739	26,852	27,379
Gross calorific value w.w. (*) (kJ/kg)	15,270	15,913	15,621	16,239	16,791
Net calorific value, d.w (kJ/kg)	26,184	26,380	26,175	26,317	26,869
Net calorific value (kJ/kg)	13,853	14,571	14,275	14,952	15,533

(*) d.w. = dry weight; w.w. = wet weight

Evaluation of the effect of compost refuse composition on the yield of biochar.

TGA were performed exploring different heating rates and pyrolysis temperature. For each value of heating rate, the maximum weight loss obtained at each temperature and the test time that has been necessary to achieve these weight losses have been represented in Figures 3 (ΔT = 2°C/min), 4 (ΔT = 5°C/min) and 5 (ΔT = 10°C/min). No significant differences have been observed in the behaviour of each of the 5 samples analysed. As foreseen, weight loss increases with temperature, for each temperature, the time to reach the maximum weight loss will be proportional to the heating rate and most of the weight loss of the sample takes place below 400°C. Above this temperature, the variation in weight is very small.

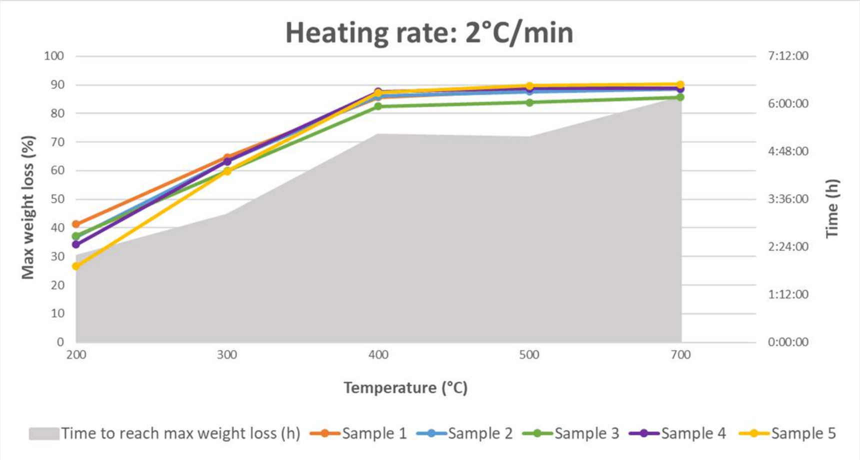

Figure 3. Weight loss vs. temperature, and time to maximum loss. Heating rate: 2°C/min

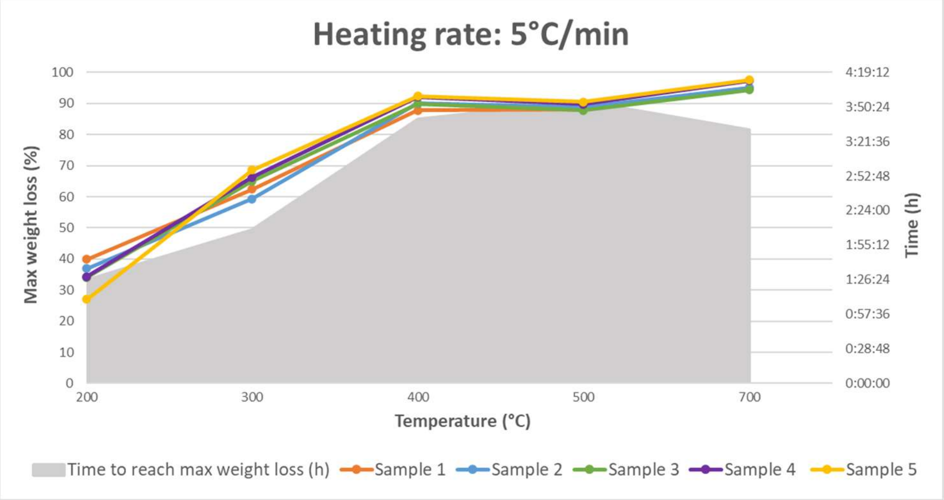

Figure 4. Weight loss vs. temperature, and time to maximum loss. Heating rate: 5°C/min

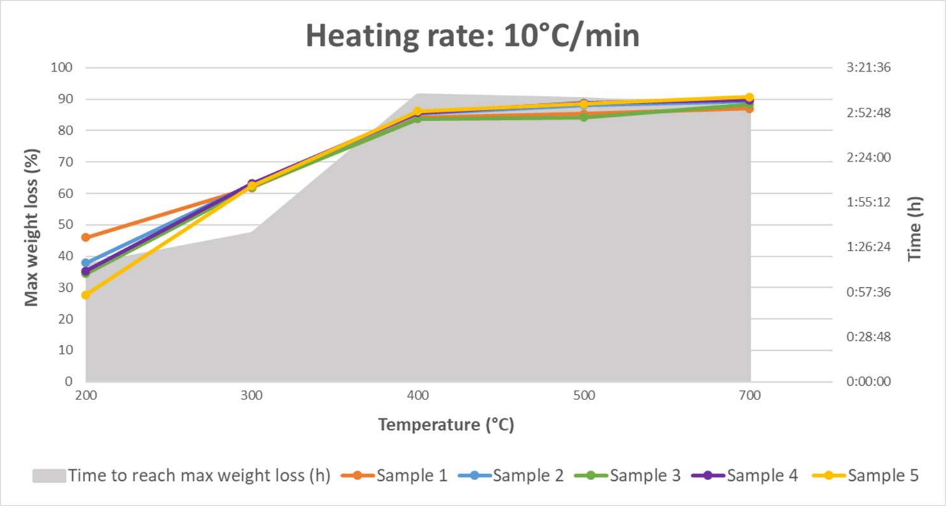

Figure 5. Weight loss vs. temperature, and time to maximum loss. Heating rate: 10°C/min

Lab scale production of 3-9 biochar batches and characterization

Figure 6. Image of the biochar obtained in Test B.

Figure 6 shows an image of the biochar obtained in Test B (350°C), while Table 6 shows the percentual distribution of the weight of the obtained pyrolysis products. In our laboratory-scale equipment up to 20% of the mass contained in the initial sample is lost and it was assumed it belongs to the gas fraction. As it can be seen from the mass balance, it is proven as foreseen that, at higher temperatures, more gas and liquid fractions are produced over biochar.

Table 6. Pyrolysis mass balance

Test reference	**Test A (300°C)**			**Test B (350°C)**			**Test C (400°C)**		
Initial quantity sample (g)	28			27			27		
Fraction	Gas	Liq.	Sol.	Gas	Liq.	Sol.	Gas	Liq.	Sol.
Quantity after test (g)	5.9	0	22.1	11.6	1.7	13.7	12.1	3.8	11.1
Balance (%)	21%	0%	79%	43%	6%	51%	45%	14%	41%

Table 7 reports the characterization values of the three biochar batches generated in Test A, B and C.

Table 7. Characterization of all fractions of the 3 batches of biochar from tests A, B and C

Test reference, solid fraction	**Initial sample**	**Test A (300°C)**	**Test B (350°C)**	**Test C (400°C)**
Moisture (%)	16.45	-	-	-
Volatile matter, d.w. (%)	60.15	47.00	25.51	17.83
Ash, d.w. (%)	16.37	20.88	34.75	34.20
Fixed carbon, d.w. (%)	23.48	32.13	39.74	47.97
Gross calorific value, d.w. (kJ/kg)	26’997	19’823	19’830	19’842
Net calorific value, d.w. (kJ/kg)	26’427	18’530	18’688	18’950
C d.w. (%)	52.60	53.83	46.95	44.02
N d.w. (%)	2.18	2.31	2.38	2.05
H d.w. (%)	6.00	5.85	5.56	4.14
H/C ratio	0.11	0.11	0.12	0.09
O d.w. (%)	22.85	17.17	10.36	15.59
P_2O_5 d.w. (%)	0.75	0.94	1.65	2.91
K_2O d.w. (%)	1.44	1.64	2.69	4.66
Ca d.w. (%)	1.38	1.86	3.19	6.21
S (mg/kg. d.w.)	1.210	2.420	1.800	2.380
Na (mg/kg. d.w.)	6.980	7.150	14.600	21.400
Mg (mg/kg. d.w.)	1.590	2.050	2.850	4.510
Cl (mg/kg. d.w.)	9.020	9.999	18.000	28.340
Co (mg/kg. d.w.)	B.L.[1]	B.L.[1]	4.0	6.0
Fe (mg/kg. d.w.)	1.727	2.520	3.959	6.674
Mn (mg/kg. d.w.)	48.2	71.4	105	196

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[1] B.L. = below limit

Test reference, solid fraction	Initial sample	Test A (300°C)	Test B (350°C)	Test C (400°C)
Mo (mg/kg. d.w.)	17	35	27	38
Cu (mg/kg. d.w.)	17.8	27.3	45.2	75.4
Ni (mg/kg. d.w.)	8.4	19.2	23	51.5
Cr (mg/kg. d.w.)	9.0	21.0	24	61
Zn (mg/kg. d.w.)	71.5	140	216	281
Hydrophobicity. WHC (g/g)	1.28	1.32	1.45	1.55

The nutrient profile of the produced biochar is promising. The H/C ratio is well below the threshold value of 0.6 appointed in the European Biochar Certificate guidelines. [5] The reference to evaluate macro- and micro-nutrient was the consolidated version of the Fertiliser Regulation (EU) 2019/1009, on the definitions of product functional categories (PFCs) 1(A)(I) "*solid organic fertiliser*" and PFC 3(B) "*organic soil improvers*".

Total nitrogen content ranges from 2.05% to 2.38%, being well above the 1% requested by Regulation (EU) 2019/1009 when K and P are present in concentrations above 1% by mass of P_2O_5 and 1% by mass of K_2O. Batches B and C fulfil these criteria while batch A slightly lacks phosphorous (0.94%). It is worth noticing that the characterised nitrogen concentration is uncommon in commercial biochar and, together with the detected potassium and phosphorus concentrations, clearly highlights the potential of biochar derived from composting rejects not only as an organic amendment but also as a source of nutrients for soils.

The biochar batches were characterized also in terms of micronutrients. The latter were generally present in non-negligible quantities, reinforcing the assumption of a biochar with promising agronomic properties. Clorine concentration is below the maximum permitted concentration 30 g/kg dry matter, set by the BCE guidelines.

As per hydrophobicity, it should be kept to a minimum to avoid a decrease in soil water holding capacity (WHC).[6] The analysed batches shown a very low WHC, being them lower that others found in literature [7]. However, another study suggests that water holding capacity of soil was found to increase with biochar application rate, meaning that a proper biochar application protocol could compensate the hydrophobicity brought by biochar in a first application. [8] Moreover, particle size has also found to affect water movement and retention. [9] Therefore, a low hydrophobicity alone is not a critical property to good performance as a soil amendment. Finally, hydrophobicity depends on pyrolysis process conditions, besides the feedstock characteristics, entailing that process design can in some extend improve the physical properties of the obtained biochar. [10]

The performed experiment initially intended to evaluate the energy balance of the pyrolysis tests, to have an indicator of the efficiency of the process from an energetic point of view. Since the heating values of the liquid and gas fractions are not available, the energy balance of the process cannot be estimated but Figure 7 and Table 8 include the energy consumption of each of the three tests, expressed as consumption per biochar mass unit in each test. The consumption of stage 0 was not taken into account, since this stage is equivalent to heating the initial sample up to 25ºC and all tests start from the same temperature.

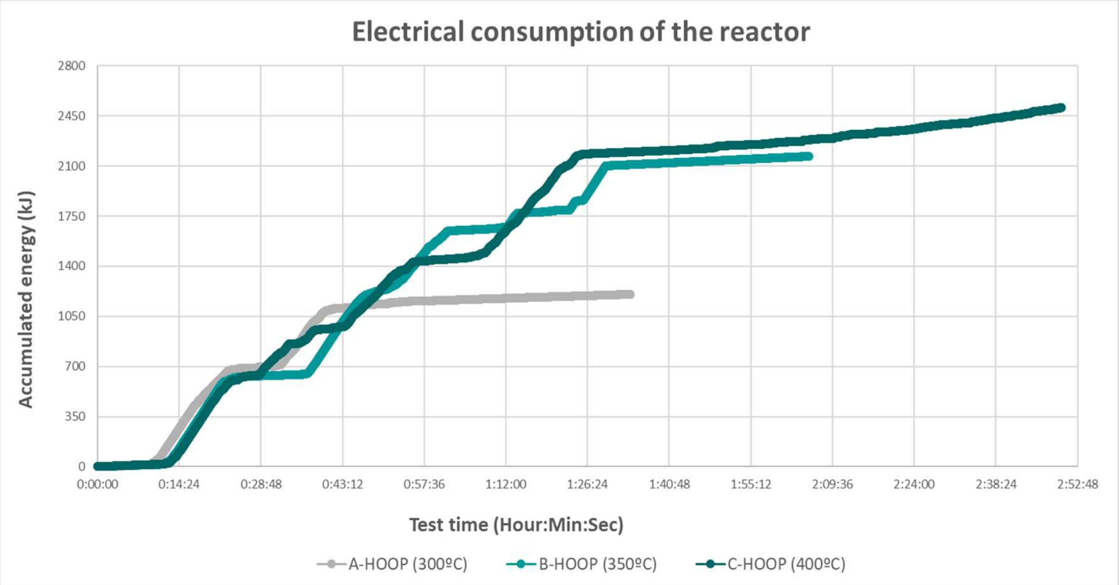

Figure 7. Electrical consumption of the reactor.

In the experimental system used, the energy contained in the generated products is not recovered by direct combustion of the gaseous and the liquid fractions generated in the process. The heat generated in that combustion would partially cover the heat demands of the process. Moreover, the experimental system used is not thermally insulated and radiation losses are not negligible. The system operates in batch, less energy efficient than one that operates in continuous regime.

Table 8. Electrical consumption

Test reference	Consumed energy (Wh)	Time	Yield (kWh/kg)
Test A (300°C)			
Stage 0	8.3	0:10:00	-
Stage 1	314.8	0:52:00	-
Stage 2	11.3	0:31:59	-
TOTAL	334.4	1:33:59	14.8
Test B (350°C)			
Stage 0	4.2	0:10:58	-
Stage 1	488.6	1:05:01	-
Stage 2	109.3	0:49:26	-
TOTAL	602.2	2:05:25	43.6
Test C (400°C)			
Stage 0	3.63	0:08:16	-
Stage 1	582.74	1:15:01	-
Stage 2	110.10	1:26:43	-
TOTAL	696.47	2:50:00	62.4

The test at 300°C would be the most efficient in terms of energy consumption. However, two additional factors must be considered to determine the most interesting process conditions: one is the quality of the obtained biochar, and another is the energy recovered through the energy valorisation of the liquid and gas fractions. Shall the reader remind that the weight of these fractions increases with process temperature and the costs of purchased energy is likely to drop when gases and liquid fractions are valorised energetically.

5. CONCLUSIONS

This work showcases that biochar obtained from compost rejects pyrolyzed at temperatures between 300°C and 400°C presents promising features for its agriculture application. The chemical characterisation of the batch samples produced in this study highlights the potential of biochar from compost rejects as "*organic soil improvers (PFCs 1(A)(I))*" and "*solid organic fertiliser PFC 3(B)*" as defined in the Fertiliser Regulation (EU) 2019/1009.

More specifically, biochar samples exhibit a nitrogen content above the minimum requested by the aforementioned regulation; potassium and phosphorus concentrations are also reaching the minimum levels for batches B and C. The five samples of compost rejects presented very similar compositions, low presence of impurities and an average content of biological material of 94.34 %.

Agronomic performances cannot be outlined without considering physical properties. In this study, we found a low hydrophobicity, which alone is not a worrying feature for two reasons. On the one side, even if biochar hydrophobicity is related to low soil water holding capacity, soil management practices can improve soil properties. On the other side, biochar physical properties like hydrophobicity can be in some extent modified depending on pyrolysis process conditions.

To fully understand the agronomic potential of biochar produced from biowaste-derived compost rejects, more investigations should be carried out to determine other key agronomic characteristics of biochar, like cation exchange capacity and porosity. Authors also suggest to complement further studies with a determination of heavy metals and organic pollutants like Polycyclic Aromatic Hydrocarbons (PHAs) and dioxin-like compounds.[11]

Overall, the findings highlight the potential of biochar derived from composting rejects as a nutrient source for soils, while underscoring the need for targeted process enhancements to maximize its environmental and economic benefits.

6. ACKONOWLEDGMENTS (EN)

This study is funded by the European Union. under the Horizon 2020 project HOOP (Grant Agreement N°101000836). Authors thank LIPOR's Departamento de Inovação, Investigação e Desenvolvimento [Research, Development and Innovation Department] for requesting this study and for providing the samples.

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