



Characteristics of Hydrochar and Liquid Products Obtained by Hydrothermal Carbonization and Wet Torrefaction of Poultry Litter in Mixture with Wood Sawdust

Rafail Isemin ^{1,*}, Natalia Muratova ¹, Sergey Kuzmin ¹, Dmitry Klimov ¹, Vadim Kokh-Tatarenko ¹, Alexander Mikhalev ¹, Oleg Milovanov ¹, Antoine Dalibard ², Olayinka Ahmed Ibitowa ³, Manuel Nowotny ³, Mathieu Brulé ⁴, Fouzi Tabet ⁵ and Bernd Rogge ⁶

- ¹ Biocenter, Tambov State Technical University, Sovetskaya St. 106, 392000 Tambov, Russia; muratova-nat@rambler.ru (N.M.); tregulyaj@mail.ru (S.K.); marcelldm@mail.ru (D.K.); vadim6873@yandex.ru (V.K.-T.); malex_v@list.ru (A.M.); milowanov.o@yandex.ru (O.M.)
- ² Fraunhofer-Institut für Grenzflächen-und Bioverfahrenstechnik IGB, Fraunhofer-Gesellschaft, Nobelstraße 12, 70569 Stuttgart, Germany; antoine.dalibard@igb.fraunhofer.de
- ³ COAST-Zentrum für Umwelt-und Nachhaltigkeitsforschung, Universität Oldenburg, Ammerländer Heerstr. 114-118, 26129 Oldenburg, Germany;
 - olayinka.ahmed.ibitowa@uni-oldenburg.de (O.A.I.); manuel.nowotny@uol.de (M.N.)
- ⁴ Department of Natural Resources Management and Agricultural Engineering, Agricultural University of Athens (AUA), Iera Odos 75, 11855 Athens, Greece; mathieubrule82@gmail.com
- ⁵ Opti'Tech, Schletterstrasse 12, 04107 Leipzig, Germany; fouzi.tabet@gmail.com
- ⁶ Inter Center GmbH, Allee der Kosmonauten 32 B, 12681 Berlin, Germany; br@intercentre.de
- Correspondence: penergy@list.ru; Tel.: +7-475-263-0446

Abstract: Poultry farms with floor-standing poultry generate large amounts of poultry litter waste. The direct application of this waste as an organic fertilizer does not ensure sustainable and costefficient utilization of all waste fractions, and can also be linked to environmental hazards. Therefore, the development of new technologies is required for processing poultry litter into a safe product with higher added value. In this work, the characteristics of activated carbon derived from hydrochar, along with the liquid products obtained from hydrothermal carbonization (HTC) and the wet torrefaction (WT) of poultry litter, were investigated. Poultry litter (PL) was applied in a mixture with sawdust (SD) in the following ratios: 1:0 (PL/SD 1:0), 1:1 (PL/SD 1:1), 1:2 (PL/SD 1:2), and 2:1 (PL/SD 2:1). WT processing took place in an innovative fluidized bed system in a superheated steam medium with low overpressure (less than 0.07 MPa) at 300 °C and 350 °C for 30-45 min. Conventional HTC processing was performed in a water medium at 220 °C for 1-4 h. The hydrochar produced in the experiments was activated with steam for 1 h at 450–750 °C. The porosity characteristics of activated hydrochar were measured, including pore size, pore volume, and specific surface area, in view of potential industrial applications as an adsorbent. Additionally, the contents of 5-hydroxymethylfurfural (HMF), as high-value product, were determined in the liquid products obtained from HTC processing, as well as in the condensate obtained after WT processing. Specific surface areas of the activated hydrochars may still be too low for application as adsorbent material. Hence, its use as a biofertilizer and soil improver should be preferred. Interestingly, the liquid fraction obtained from the innovative WT process displayed a significantly higher 5-HMF content compared to the conventional HTC process.

Keywords: hydrothermal carbonization; steam torrefaction; biomass; waste treatment; activated carbon; fluidized bed

1. Introduction

In various regions of Russia, and abroad, the lack of technologies to ensure efficient disposal of biowaste, generated by agricultural, food, timber, and wood processing industries, results in environmental hazards and emissions.



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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). An innovative pathway applicable for a wide range of biowaste and organic waste is the process of hydrothermal carbonization (HTC), taking place at a temperature range of 180–250 °C and an overpressure of 2–10 MPa [1,2]. This process generates a solid product, hydrochar, which is similar in properties to brown coal. The hydrochar yield is in the range 35–60%, depending on process conditions. Hydrochar can be further converted into high-value products, such as synthesis gas and liquid fuel, soil improver, or sorbents [3].

In a study investigating the use of HTC for municipal solid waste processing [4], a hydrochar yield of 58% of the initial raw material was obtained. During the process, the heating value of the biomass increased 1.41-fold based on weight-heat content, or 9-fold based on volumetric heat content. Furthermore, the HTC process can be successfully applied for the processing of medical waste and plastic waste [5], since organic chlorine in polyvinyl chloride wastes can be converted into an inorganic form during the process. The efficiency of waste dechlorination increased up to 83–90% as a result of the HTC co-treatment of PVC medical waste together with lignin, or wood chips, as co-substrate. Furthermore, HTC has been successfully used for PVC processing [6] at 180–260 °C, during which active dehydrochlorination was observed. Chlorinated hydrocarbons, including chlorophenols, could only be identified at trace levels in the final products, and polychlorinated dibenzodioxins, in addition to dibenzofurans, were not detected. Hence, the HTC of municipal solid waste may be applicable as an environmentally friendly process for the removal of toxic organic products. Additionally, the effects of HTC on the distribution of heavy metals, and subsequent pyrolysis of sewage sludge, were studied in comparison with the direct pyrolysis of sewage sludge [7]. Under this configuration, HTC reduced the content of acid-soluble and recoverable fractions of heavy metals; reducing the risk of pollution by heavy metal compounds in the environment as heavy metals remained in an inactive form in the inorganic fraction of hydrochar.

Hydrochar generated by HTC can be used in high-value applications, such as adsorption processes [8,9] and the synthesis of activated carbon [10–12]. HTC produces hydrochar with higher amounts of oxygen functional groups and low levels of aromatization, which facilitates further chemical activation [10,13]. In [10], hydrochar obtained from starch and eucalyptus sawdust was exposed to chemical activation using potassium hydroxide. The researchers obtained high-quality activated carbon with high pore surface (2700 m²/g) and hydrogen absorption capacity (16.4 mmol/g) [10]. In another study, activated carbon, obtained by the chemical activation of hydrochar with potassium hydroxide, yielded CO₂ absorption rates of up to 4.8 mmol/g at 298 K and 1 atm [11]. It is reported that activated carbon generated from hydrochar originating from rye straw, displayed high pore surface (2200 m²/g) and CO₂ adsorption capacity (20 μ mol/g) [14]. Furthermore, chemical activation of rice husk hydrochar with H₃PO₄ also yielded high specific pore surface areas (2610 m²/g) [15], while chemical activation with KOH yielded an even higher specific pore surface more surface (3362 m²/g), and therefore produced higher adsorption capacities [16].

Poultry litter is among the most dangerous categories of biowaste. This is especially alarming considering the risks posed to people, and the environment in surrounding areas, in connection with the commissioning of large poultry farms which are engaged in the production of meat. The environmental hazards caused relate to the daily inflow of dozens of tons of droppings, along with the long-term accumulation of this waste in unauthorized storage facilities at the farming sites which do not possess sufficient arable land for spreading the biowaste. For example, a typical poultry farm hosting 40,000 laying hens, or 10 million broilers, respectively, generates between 35,000 and 83,000 tons of poultry litter, in addition to over 400,000 m³ of wastewater, annually, with high contents in organic components. This cycle results in potentially hazardous materials that pose a threat to public health and the environment. According to the World Health Organization, droppings and sewage from poultry enterprises can be a factor in the transfer of pathogens for more than 100 infectious and invasive diseases, including zoonoses. In addition, organic waste provides a favorable environment for the development, and long-term survival, of pathogenic microflora and can contain increased amounts of pollutants, including heavy

metals, pesticides, antibiotics, radioactive substances, weed seeds, and other contaminants. Therefore, it is extremely difficult to ensure the environmental safety of these wastes, as existing technologies for disinfecting poultry droppings are unreliable, ineffective, and very expensive.

On the other hand, opportunities exist for the development of innovative pathways converting poultry litter into high-value products. Nitrogen-containing hydrochar, which can be obtained by the HTC of poultry litter, may act as an adsorbent for electrolytes. This provides a promising candidate as a sorption material for water purification from heavy metals (for example, Pb^{2+} , Cd^{2+} , Cu^{2+} , U^{6+}) or from organic pollutants (for example, dyes) due to the abundance of surface functional groups [13,17]. This broadens the prospective uses of hydrochar obtained by the HTC of nitrogen-containing biowaste, such as manure and poultry litter, as a raw material for the production of highly efficient sorbents.

Nevertheless, the HTC process, when is carried out using state-of-the-art technologies, is a low productivity batch process, which should be carried out at elevated pressure. These harsh operating conditions, along with low performances, may significantly complicate and increase the cost of waste processing.

Wet torrefaction (WT) is sometimes assimilated to HTC, and involves a similar process carried out at lower operational pressures, thereupon reducing processing costs. However, a significant drawback from the reduced pressure is the longer processing time required for biomass treatment. This issue can be resolved through the integration of a fluidized bed design into the WT process. In order to optimize performance, the WT process can be performed under the specific operating conditions of vapothermal carbonization in a superheated steam environment. Earlier [18], we performed preliminary studies on the WT of ground biomass in a fluidized bed in an environment of superheated steam. These previous studies have shown that the duration of the WT process in a fluidized bed can be reduced to 15–20 min, versus 4–12 h when carrying out WT by means of stateof-the art technologies. The advantage of WT is the possibility of obtaining hydrochar, the characteristics of which are comparable to the hydrochar obtained by conventional HTC, while applying mild processing conditions, in particular lower overpressure [19,20]. Superheated steam can be used as a medium for drying biofuels and wood [21]. The use of superheated steam in the heat treatment of biomass is considered a promising alternative to traditional technologies, as superheated steam is an inert medium (at temperatures below 600 °C) [22] with interesting thermodynamic properties, allowing for high heat and mass transfers [23].

The aim of this work is to compare the characteristics of the activated hydrochar (AH) obtained by physical steam activation, as well as liquid products obtained by processing a mixture of poultry litter (PL) and sawdust (SD) with HTC and WT processes. WT is performed through an innovative time-efficient process comprising a fluidized bed operated with superheated steam as a fluidization medium. The key parameters investigated in this study were on the pore characteristics and specific surface area of the activated hydrochar, which were important parameters for its application as an absorbent material. The concentrations of organic compounds (TOC) and 5-HMF in the liquid products were also measured in view of the recovery of 5-HMF as a high-value compound from the liquid fraction.

The yields of non-condensable gases obtained from the innovative WT process have already been reported in previous studies [24,25].

2. Experimental Setup and Experimental Technique

Figure 1 presents the pilot plant for the wet torrefaction (WT) of biowaste in a fluidized bed and an environment of superheated steam. The plant consists of the following components: feedstock input bunker (1), reactor for carbonization in a fluidized bed (2), cyclone separating gases from hydrochar particles exiting the reactor (3), hydrochar collection bunker (4). The apparatus also comprises a boiler for steam generation, a superheater, and a steam cooler (not shown). The reactor (2) for wet torrefaction is connected to gas

distribution inlets supplying superheated steam under the fluidized bed. The side walls of the reactor were heated electrically to maintain the required temperature in the reactor. A detailed description of the operation of the pilot reactor for WT has been provided in [25].



Figure 1. Diagram of the vapothermal carbonization apparatus of biowaste in a fluidized bed in a superheated steam environment: (1) feedstock bunker for biomass; (2) reactor; (3) cyclone; (4) product bunker for hydrochar recovery.

Before initiating the experiment, roughly 6 L of hydrochar from a previous batch was poured into the wet torrefaction reactor. Subsequently, 2 kg of raw material, which would be carbonized, was poured into the feedstock bunker. The pre-heating of the reactor was performed while the apparatus was purged with nitrogen until the operating temperature was reached. Subsequently, nitrogen input was stopped. Saturated steam was generated and supplied by the superheater, then fed into the gas inlets. The reactor temperature was set for the desired operating conditions (310–330 °C or 350 °C). Subsequently, the biomass feedstock was fed into the reactor for wet torrefaction, and loading was completed after 15–17 min. Since the initiation of feedstock feeding, non-condensable gases (CO_2 , CO, H_2 and CH₄) were continuously sampled downstream of the condenser and characterized with a gas analyzer (Vario Plus Industrial Syngas analyzer). After the concentration of these substances decreased to the initial levels preceding the start of the biomass supply to the reactor, the experiment was stopped, and the generated hydrochar was discharged into the product bunker. The duration of the WT process ranged from 30 to 40 min and decreased with an increase in the process temperature from 300 °C to 350 °C. The entire unit was then purged with cold nitrogen and the cooled hydrochar was unloaded from the bunker for further analyses.

HTC of the poultry litter (PL) in mixture with sawdust (SL) was performed in a 250 mL stainless steel autoclave with a PTEF inlet, and subsequent physical activation of the obtained hydrochar was performed at higher temperatures in a steam medium. Each HTC experiment contained a total mass of 100 g and was carried out at 220 °C for 1, 4 and 8 h, respectively. The sealed autoclave was placed into a heated convection oven. After completing the reaction, the autoclave was taken out of the oven and placed in a fume hood at room temperature to cool the reaction products. Vacuum filtration was applied to separate liquid products from the hydrochar. The separated hydrochar was washed with 1 L of deionized water and dried overnight at 120 °C. Subsequent activation tests were carried out with steam at 450 and 750 °C for 1 h. To this effect, 1 g of hydrochar was filled into a ceramic container and placed into a tube furnace. Steam generated by the

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evaporation of the deionized water under a nitrogen atmosphere was flushed into a round bottom flask at the end of the tube furnace where it made contact with the hydrochar. A PT100 thermo-element, connected with a temperature controller, measured the temperature in the middle of the furnace. After the reaction, the furnace was cooled down to 80 $^{\circ}$ C and the obtained activated hydrochar was washed with 500 mL deionized water and finally dried for 12 h at 120 $^{\circ}$ C.

Subsequent physical activation of the WT and HTC hydrochars was performed at higher temperatures (450 and 750 °C) in a steam medium. This process was completed in order to obtain activated hydrochars with high pore volumes and specific surface areas, with their potential application as adsorbents in industrial and environmental processes.

The elemental analysis was performed with a EuroEA 3000 Elemental Analyzer, and ash contents were determined according to ASTM D3174-02. Surface characterization was done by infrared spectroscopy and solid-state NMR. IR spectra were measured under ATR conditions (diamond ATR crystal) with a Tensor 27 (Bruker, USA) spectrometer and OPUS software for spectra interpretation. After background recording, each sample was measured with 25 scans. Next, 13C and 1H solid state NMR spectra were obtained using an Avance III HD NMR spectrometer (Bruker BioSpin) with a cryomagnet having a nominal field of 11.75 T. Resonance frequencies were 125.86 MHz for 13C and 500.46 MHz for 1H. A standard (double resonance) 4.0 mm WVT probe (Bruker), which can be operated at spinning speed up to 15 kHz, was used for the experiments. At 13C, for 1H, MAS NMR spectra were referenced to solid 1,1,1,3,3,3-hexamethyl-2,2-bis (trimethylsilyl) trisilane, with resonance signals occurring at 6.9 ppm (13C) and 0.1 ppm (1H) relative to the primary reference TMS (0 ppm).

Solid-state spectra were recorded using a rotor-synchronized Hahn echo pulse sequence (p/2–t–p–t–acquire; t: rotor period) and standard cross-polarization (CP) pulse sequence, respectively. A total of 13C {1H} CP MAS NMR was used with a flip-back pulse on the 1H spins following the data acquisition process. Recycling delays amounted to 15–120 s for the Hahn echo experiments and 15–60 s for the CP experiment, respectively. For the 1H NMR and 13C Hahn echo experiments, 16, and up to 2112 scans, were collected. The sample was recorded within 1 day. The studied samples were operated to set the Hartman–Hahn match under the MAS conditions with the match optimized for a spinning speed of 14 kHz. Data processing was performed with TopSpin 3.5 software (Bruker).

The specific surface areas were evaluated by nitrogen sorption with a TRISTAR II 3020 (Micromeritics GmbH, Germany) followed by BET analysis. After drying the samples for 12 h under a vacuum at 150 °C, the free space was measured with helium prior to the recording of the isotherms. Adsorption and desorption data were collected at a pressure range of p/p0 = 0.005-0.95 (36 points, randomly chosen by measurement mode) and p/p0 = 0.95-0.1 (19 points, linear distribution), respectively. Specific surface areas were calculated using the BET formalism in accordance with the Rouquerol criteria.

Thermal stability was evaluated via thermogravimetric analysis (TGA) with a TGA 4000 (Perkin Elmer Corporation, USA) setup, including a furnace with balance, temperature sensor, heating/cooling elements, AI_2O_3 crucible, and Pyris software. Before the measurement, the apparatus was purged with nitrogen for 30 min at a rate of 40 mL min⁻¹, and the crucible was baked out. Measurements were carried out under a nitrogen atmosphere. The temperature was varied as follows: hold for 1 min at 30 °C, ramp from 30 °C to 750 °C with 10 °C min⁻¹, and, finally, hold for 60 min at 750 °C.

The separated liquid products of HTC were stored in a fridge before undergoing further analyses. The determination of 5-HMF amounts were completed using the Merck quick test.

3. Results and Discussion

The analysis results of the HTC and WT hydrochar after activation are presented in Table 1.

Process	Feedstock Mixture	Processing Conditions	Activation Conditions	Ash Content %	Carbon Content %	SSA, m²/g	Pore Volume, cm ³ /g	Pore Diameter, nm
		10	-	41	34	13	0.035	11
WT	PL/5D (2.1)	40 min/	1 h/450 °C	53	41	13	0.015	8
	(2:1)	300 C	1 h/750 °C	58	37	460	0.258	2
		40 min /	-	29	50	5	0.017	12
WT	(1,1)	40 mm/	1 h/450 °C	37	46	10	0.021	8
	(1.1)	300 C	1 h/750 °C	53	44	300	0.185	2
		40 min /	-	20	60	7	0.027	10
WT	(1.2)	40 mm/	1 h/450 °C	28	60	29	0.032	4
	(1:2)	300 C	1 h/750 °C	34	56	447	0.229	2
		20 min /	-	39	37	14	0.044	13
WT	rL/5D (1.0)	30 mm/	1 h/450 °C	51	41	15	0.001	6
	(1:0)	350 C	1 h/750 °C	57	34	226	0.148	3
		20 min /	-	31	49	10	0.031	12
WT	(1,1)	30 mm/	1 h/450 °C	44	43	13	0.018	6
	(1:1)	330 C	1 h/750 °C	52	46	298	0.159	2
		20 min /	-	19	75	5	0.014	11
WT	(2.1)	250 °C	1 h/450 °C	25	61	35	0.013	7
	(2.1)	330 C	1 h/750 °C	40	55	470	0.252	2
	PI /SD	30 min /	-	19	59	5	0.020	8
WT	(1.2)	350 °C	1 h/450 °C	24	67	7	0.021	2
	(1.2)	550 C	1 h/750 °C	35	55	491	0.261	2
			1 h/450 °C	49	44	14	0.003	9
HTC	PL/SD	1 h/220 °C	1 h/750 °C	54	38	118	0.048	5
me	(1:0)	4 h/220 °C	1 h/450 °C	51	47	36	0.017	5
			1 h/750 °C	60	35	125	0.030	5
			1 h/450 °C	24	58	125	0.017	27
HTC	PL/SD	1 h/220 °C	1 h/750 °C	19	72	325	0.101	4
me	(1:1)	4 h/220 °C	1 h/450 °C	28	63	167	0.003	2
			1 h/750 °C	21	68	445	0.033	3

Table 1. Characteristics of activated hydrochars obtained after WT or HTC followed by physical steam activation.

According to data shown in Table 1, the characteristics of activated the hydrochar (AH) obtained by WT were influenced by feedstock composition and the processing temperature. Thus, with an increase in the proportion of sawdust (SD) in the mixture, from 33% to 66%, the carbon content in AH increased from 34% to 60% at a process temperature of 300 °C. At a process temperature of 350 °C, the composition of the initial feedstock had a lower effect on the characteristics of AH, whose carbon contents ranged from 49% to 75%, with the maximum carbon content observed in AH obtained from a mixture containing 67% of poultry litter and 33% sawdust (ratio 2:1).

Physical activation in a steam environment at 750 °C significantly increased the pore volumes and specific surface areas (SSA) of AH, while reducing the pore sizes. Moreover, AH characteristics were affected by the composition of the initial biomass, with higher proportions of PL in the feedstock mixture resulting in high pore volumes and specific surface areas of 0.258–0.252 cm³/g and 460–470 m²/g, operating at temperatures of 300 and 350 °C, respectively. However, the application of PL alone resulted in lower values, revealing interactions caused by the addition of SD in the feedstock, which resulted in higher porosities and specific surface areas.

The characteristics of the AH obtained by HTC were influenced by both the composition of initial feedstock, and the activation temperature. Furthermore, considering the higher activation temperature of 750 °C, the characteristics of AH for the investigated parameters (pore volume, specific surface area and pore diameter) were similar to AH obtained using the innovative WT process. These results show that HTC could be replaced with WT processing under mild conditions (lower overpressure), obtaining AH of similar characteristics after subsequent physical activation.

The carbon content in AH obtained by WT and HTC of PL, after physical activation in a steam environment at 450–750 °C, was 1.7–1.8 and 1.7–2.2-fold higher than in the biochar obtained by another research group who investigated the pyrolysis of PL at 400–600 °C, respectively [26].

SSA of AH produced by WT and HTC, and activated with steam at 450 $^{\circ}$ C, was 3.5–6.7-fold higher than in biochar obtained by the pyrolysis of PL at 400–600 $^{\circ}$ C [26].

An increase of the hydrochar activation temperature to 750 °C, obtained by WT or HTC from PL as sole feedstock without sawdust, results in SSA of AH up to 125–236 m²/g. Furthermore, the addition of a share of SD in the feedstock results in significantly higher SSA of up to 447–491 m²/g. Nevertheless, the physical steam activation at 450–750 °C does not yield high SSA values above 3000 m²/g, which can be obtained by chemical activation processes [24].

In a previous study [19], we have shown that hydrochar obtained from a mixture of PL and SD before and after ac7tivation by pyrolysis, contains large amount of nutrients that make activated biochar a valuable fertilizer: potassium 30.7–57.7 mg/g, magnesium 9.7–19.2 mg/g, phosphorus 20.3–39.7 mg/g. Furthermore, in addition to nutrient supply as a biofertilizer, hydrochar may have additional benefits as a soil improver, due to its porosity and nutrient sorption properties, increasing water and nutrient retention [24,26].

An additional benefit of converting PL into raw or activated hydrochar is the removal of the pathogenic microflora which is contained in large amounts in raw PL. Hence, pyrolysis processes result in the efficient disinfection of PL [27]. Following pyrolysis, the pH of PL decreased to neutral values (pH—6.2–6.8), which are favorable for plant development. The total nitrogen content in poultry litter amounts to 8–9%, of which 10–12% is in an easily degradable water-soluble form. The presence of large amounts of readily available nitrogen creates a danger to the development of plants. Pyrolysis leads to a sharp decrease in the content of water-soluble and easily decomposable forms of nitrogen, decreased by 0.67% in weight, and the removal of unpleasant odors.

Table 2 shows the results of the analysis of the liquid products of WT and HTC.

According to the results shown in Table 2, the method of thermal feedstock treatment (WT or HTC) does not significantly affect the content of total organic carbon in the liquid phase.

The content of 5-HMF in the liquid phase after WT decreases along with increased proportions of poultry litter in the mixture, from 0.74-0.8 g/L at a PL share of 33%, down to 0.32-0.42 g/L at a PL share of 66%.

An increase in WT process temperature resulted in decreased 5-HMF concentrations. However, the concentrations of 5-HMF in the liquid products after WT were 13–27-fold higher than after HTC. The higher 5-HMF concentrations in the liquid products of WT might be caused by very short contact times of biomass particles with superheated steam, not exceeding 2 s. These short contact times might prevent the decomposition of 5-HMF. This might occur in the HTC process as the liquid phase remains in contact with biomass during the entire reaction time of 4 h.

The results also show that sawdust (SD) is more suitable than poultry litter (PL) for the production of 5-HMF by wet torrefaction (WT) in a fluidized bed in a superheated steam environment.

Method	Substrate	Reaction Condition	TOC, g/L	5-HMF, g/L
WT	PL/SD (2:1)	300 °C	14	0.42
WT	PL/SD (1:1)	300 °C	10	0.54
WT	PL/SD (1:2)	300 °C	13	0.8
WT	PL/SD (1:0)	350 °C	14	0.3
WT	PL/SD (1:1)	350 °C	9	0.19
WT	PL/SD (2:1)	350 °C	13	0.32
WT	PL/SD (1:2)	350 °C	12	0.74
HTC	PL/SD (1:0)	4 h/220 °C	12	0.02
HTC	PL/SD (1:1)	4 h/220 °C	9	0.02
HTC	PL/SD (2:1)	4 h/220 °C	10	0.08
HTC	PL/SD (1:2)	4 h/220 °C	8	0.06
HTC	PL/SD (0:1)	4 h/220 °C	7	6.15

Table 2. Composition of the liquid products of WT and HTC.

Cellulose is considered the main raw material for the production of HMF in the near future, since it is the most affordable non-food polysaccharide [28]. In [29], the process of obtaining 5-HMF from sugarcane bagasse by HTC was studied in a water medium at temperatures of 200–300 °C. Increased HMF yields were observed when the temperature increased from 200 °C to 270 °C. Above 270 °C, the yield of 5-HMF decreased, and the compound was undetected at 300 °C (>10 min reaction time). The maximum yield of 5-HMF obtained in the process reached 3.09 wt%. This 5-HMF yield is much higher than the yields obtained in our study, even while applying the innovative fluidized bed WT technology. This might be due to the fact that we used poultry litter biowaste as a main substrate, which contains lower amounts of cellulose. Indeed, HTC of sawdust (SD) as sole substrate yielded 76–307-old higher HMF contents than the HTC of the mixtures of poultry litter (PL) and sawdust (SD).

In large-scale industrial production of 5-HMF, reactor performance may be largely determined by the contact time of biomass and the water medium [28]. High-temperature (\geq 170 °C) coal-water dehydration methods imply shorter contact times between the biomass and the water medium (water or steam) in the reactor (amounting to seconds, or fractions of a second) and might be very promising for large-scale production. However, the possibility of using the process of wet torrefaction (WT) in a fluidized bed in a superheated water vapor environment for the production of 5-HMF, requires additional research. In particular, important prerequisites for future full-scale applications of the WT process were: the integration of a superheated steam generation process, a heat recovery and recycling system connected to the re-condensation of the liquid products and nitrogen and water recycling or a recovery system.

4. Conclusions

Studies have shown that both methods of heat treatment (WT and HTC) produce activated hydrochars with similar characteristics, following physical steam activation at 450–750 °C. Interestingly, in the innovative WT process operated as a fluidized bed in an

environment of superheated steam, biomass treatment can be completed within 30–40 min, while the conventional HTC process may last for 1–4 h.

The obtained activated hydrochars had specific pore surface areas (SSA) up to $491 \text{ m}^2/\text{g}$, yet higher SSA may be achieved by chemical activation methods. Further studies are needed to investigate the potential application of activated hydrochars as biofertilizers and soil improvers. Due to the low SSA, and the presence of nutrients, the obtained activated hydrochar may be more suitable for agricultural applications, rather than as industrial adsorbents. In this configuration, biomass treatment by WT/HTC, and the subsequent physical steam activation, alleviates the drawbacks arising from direct poultry litter application to the soil, since pathogens and excess nitrogen were removed, while the transportability, storability, and soil-improving properties were improved.

The mixture of poultry litter and sawdust is not the optimal feedstock for 5-HMF production due to low cellulose contents, as highlighted by the much higher 5-HMF yield obtained after processing sawdust as sole feedstock. Still, the valorization of the 5-HMF contained in the liquid products of WT/HTC might improve the economics of the biowaste treatment processes.

In this regard, the innovative fluidized bed WT process yielded 13–27-fold higher 5-HMF contents compared with the conventional HTC process. Hence, it would be interesting to further investigate the WT process in view of 5-HMF production from diverse feedstocks.

More research is required to prove that the WT process, in a fluidized bed in a superheated steam medium, can be considered a promising technology for biowaste treatment and 5-HMF production. This research would focus on the investigation of diverse feedstock and their mixtures, as well as the optimization of energy efficiency during the process through efficient insulation and the addition of heat exchangers, along with the efficient recovery or recycling of water and nitrogen flows.

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