Scale-up the production of swine manure hydrochar in a continuous pilot plant

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In the last decades, a variety of thermochemical processes have been developed to improve the characteristics of biomass for its use as a suitable industrial biofuel. Most of these processes require the use of dry biomass, which leads to high energy consumption. Hydrothermal carbonization (HTC) is a thermochemical process able to treat wet biomass (without pre-drying step), at mild temperatures (180 – 250 °C), low residence time (5 – 120 min) and autogenous pressure. The main HTC product is a carbonaceous solid called hydrochar, with suitable properties to be used as a biofuel and a liquid fraction, rich in soluble organic compounds and nutrients (Ipiales et al., 2021). One of the current gaps in HTC studies is the lack of reported data for pilot- and full-scale processes, because most existing research focuses on batch scale carried out in batch reactor (Zijlstra et al., 2022). In this study, swine manure (SM) was valorized by HTC under batch and continuous mode, using bench and pilot scale reactors, to obtain a hydrochar with suitable characteristics to be considered as an industrial biofuel according to ISO/TS 17225-8 (2016).

HTC experiments were carried out at 180, 210 and 230 ºC: i) in an electrically heated 4 L ZipperClave® pressure vessel (bench scale) operated in discontinuous mode, ii) in a continuous reactor developed by ARQUIMEA (pilot plant scale). The reaction time in batch test was 45 min, and the residence time in the continuous reactor were 20 min and 45 min. Dehydrated SM was mixed with tap water up to 7.5% total solids (TS). The HTC products were separated by filtration. The hydrochar was dried at 105 ºC and process water was stored at 4 ºC. The hydrochar (HC) and process water (PW) were labelled according to HTC operational conditions (temperature and reaction/residence time) and operation mode (batch (B) or continuous (C)), i.e., HC180-45-B or HC180-45-C; PW180-45-B or PW180-45-C).

Table 1 shows the main characteristics of SM and hydrochars. In batch tests, the mass yield of hydrochar (Y_{HC}) decreased with increasing temperature from 43% for HC180-45-B to 35% for HC230-45-B, because of the increase in temperature favored the hydrolysis of proteins, carbohydrates, and lipids. The C content of the hydrochar increased by 5 – 8 percentage points and the higher heating value (HHV) by 8 – 13 percentage points compared to the feedstock. The energy yield (E_{yield}) was in the range 40 – 47%. In continuous tests, Y_{HC} decreased significantly with increasing residence time. In addition, a smaller increase in carbon content and HHV was observed compared to the batch test. At the lowest temperature and residence time (180 °C and 20 min) a negligible increase in C content and HHV was observed. Increasing the reaction time to 45 min resulted in an increase of around 2 percentage points in C content and HHV. Increasing the reaction temperature increased the carbon content by 3 – 7 percentage points and around 10% the HHV. E_{yield} was significantly higher in the continuous than in the batch tests. Reaction temperature showed a greater effect on C content, HHV, Y_{HC} and E_{yield} than reaction/residence time. According to the standard on biofuels from biomass for use at industrial level (ISO/TS 17225-8), the hydrochar obtained in the batch and continuous tests comply the requirements for HHV (> 17 MJ/kg), N (< 3%), S (< 0.5%) and ash content (< 20%).

Table 1. Main characteristics of swine manure and hydrochars.

		Y_{HC} (%)	**FC (%)**	**VM (%)**	**Ash (%)**	**C (%)**	**N (%)**	**S (%)**	**HHV (MJ/kg)**	**E_{yield} (%)**
	SM	–	13.7±0.1	75.8±0.1	10.5±0.1	45.0±0.4	1.4±0.0	0.5±0.0	18.5±0.2	–
HC180	**45-B**	42.7±0.1	23.9±0.1	68.3±0.2	7.8±0.2	50.0±0.6	1.4±0.0	0.3±0.0	19.9±0.2	46.7
	45-C	64.2±2.9	24.9±0.2	69.6±0.1	5.5±0.1	47.0±0.6	0.8±0.1	0.3±0.0	18.8±0.4	65.4
	20-C	73.0±3.9	29.9±0.1	64.3±0.1	5.8±0.1	46.2±0.5	1.0±0.2	0.3±0.0	18.6±0.1	73.3
HC210	**45-B**	40.0±0.2	25.4±0.2	66.7±0.1	7.9±0.2	52.2±0.6	1.4±0.1	0.3±0.0	20.5±0.4	45.7
	45-C	46.0±2.1	21.2±0.1	75.0±0.1	3.7±0.1	50.4±0.3	1.6±0.3	0.4±0.1	20.3±0.1	50.5
	20-C	61.2±3.4	21.9±0.2	73.4±0.0	4.7±0.1	47.5±0.6	1.1±0.0	0.2±0.0	19.0±0.3	62.9
HC230	**45-B**	34.7±0.3	25.9±0.1	65.9±0.1	8.2±0.2	53.0±0.1	1.5±0.0	0.3±0.0	21.0±0.1	39.9
	45-C	41.2±1.8	22.2±0.1	73.4±0.1	4.0±0.1	51.5±0.2	1.4±0.0	0.2±0.0	20.7±0.1	46.2
	20-C	60.8±2.8	21.0±0.2	74.5±0.1	4.1±0.1	49.8±0.6	1.5±0.0	0.2±0.0	19.7±0.3	66.1

Table 2 shows the main characteristics of the process water obtained in batch and continuous tests. The pH of the liquid fraction decreased with the reaction temperature and time, because of the increase of organic acid content, mainly acetic and propionic acids. The process water from batch test showed a high chemical oxygen demand (COD) in the range 16 – 22 g/L and total organic carbon (TOC) in the range 7 – 8 g/L, while the process water from continuous test presented a less organic carbon content (5 – 13 g/L COD and 2 – 7 g/L TOC). The higher organic matter content in process water in the batch test was a result of the longer time at high temperature of the SM because of the heating ramp up to the target temperature, which favored the hydrolysis of organic matter to the liquid fraction. The TS and volatile solids (VS) content showed minimal variation with temperature and time in the batch and continuous tests (4 – 5 g/L TS and 2 – 4 g/L VS), while the total nitrogen (TN) content hardly increased under the studied operating conditions. Phosphorus content as orthophosphate decreased with reaction temperature, being around 13 – 26% of the phosphorus present in the feedstock transferred to the process water.

Table 2. Main characteristics of process waters.

		pH	TS (g/L)	VS (g/L)	COD (g/L)	TOC (g/L)	TN (g/L)	P-PO$_4$ (mg/L)
PW180	**45-B**	4.3±0.1	5.0±0.8	3.6±0.8	16.3±0.1	8.1±0.2	0.3±0.0	423±2.3
	45-C	6.0±0.2	3.7±0.5	3.3±0.5	6.5±0.2	3.1±0.1	0.2±0.0	447±1.2
	20-C	6.8±0.2	3.9±0.2	3.1±0.2	4.9±0.6	1.8±0.1	0.2±0.0	430±1.4
PW210	**45-B**	4.0±0.1	4.0±0.6	2.2±0.3	19.8±1.9	6.5±0.4	0.3±0.0	275±2.6
	45-C	4.4±0.4	4.8±1.0	4.0±0.7	10.2±0.6	5.4±0.5	0.2±0.0	263±1.4
	20-C	5.1±0.2	3.1±0.4	2.1±0.4	12.4±1.1	5.7±0.3	0.2±0.0	264±1.6
PW230	**45-B**	3.8±0.1	3.5±0.2	2.9±0.1	22.1±1.3	6.9±0.1	0.4±0.0	224±1.1
	45-C	4.0±0.1	3.7±0.2	2.8±0.3	11.0±0.5	6.4±0.2	0.3±0.0	236±3.5
	20-C	4.1±0.2	4.1±0.4	3.4±0.4	12.7±1.1	6.7±0.1	0.3±0.0	255±2.1

Conclusions

The HTC of SM under batch and continuous experiments allowed obtaining a hydrochar with suitable characteristics to be considered a biofuel at industrial level according to ISO/TS 17225-8. Reaction temperature showed a greater influence on the increase in carbon content, HHV, and E_{yield} compared to reaction time. The process water showed a high content of organic compounds (COD and TOC), and a considerable content of nutrients (nitrogen and phosphorus), so it could be used as a substrate for material and energy valorization, providing a competitive advantage compared to other technologies. Hence, continuous HTC operation allows the valorization of large volumes of waste, providing a new and interesting alternative for energy and material valorization of SM, as well as optimizing the operational conditions for an efficient and profitable process.

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