



Poultry Manure Derived Biochars – The Impact of Pyrolysis Temperature on Selected Properties and Potentials for Further Modifications

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ABSTRACT

The overall goal of this work was to investigate the potential of poultry manure for thermal conversion into biochar and the impact of temperature on selected biochar properties. Biochar is a solid carbonized material that demonstrates a number of interesting properties such as high microporosity and surface area, presence of surface functional groups and micro and macroelements. Therefore, it can be applied as a sorbent to remove organic and inorganic substances from liquid and gaseous phases, as an amendment in composting and anaerobic fermentation, a component of fertilizers and soil improver or as a filler in production of biocomposites. The scope of this work included: collection and analysis of poultry manure samples from an organic poultry farm (a), laboratory pyrolysis of poultry manure in selected temperatures (400-700 °C) (b), the analysis of biochar properties (chemical composition, surface area, functional groups, etc.) produced at different temperatures (c) and discussion of potential applications for the produced poultry derived biochars (d). The efficiency of conversion of poultry manure to biochar was about 62% for 400 °C and about 55% for 700 °C. The obtained results demonstrated that biochars produced from poultry manure showed a complex chemical composition. The following elements were present: carbon, oxygen, nitrogen, sodium, magnesium, aluminium, silicon, phosphorus, sulphur, chlorine, potassium, calcium, iron. Surface area of the obtained biochars was very low (about 5 m²g⁻¹). Gradual aromatization of the investigated biochar and the formation of wide range of oxygen functionalities were observed. In view to the obtained results poultry manure derived biochars due to chemical composition could be potentially applied as a component of fertilizers, soil improvers and composts. However, other applications such as removal of various contaminants, e.g., from wastewater or exhaust air would require additional modification through thermal and/or chemical treatment.

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KEYWORDS

Poultry manure, Biochar, Pyrolysis, Temperature, Physical properties.

INTRODUCTION

Poultry manure is a type of waste generated in significant quantities primarily in Poland, United Kingdom and France being the leaders among the EU countries in the production of poultry [1]. One of the most recently investigated methods for managing this type of waste is conversion of poultry manure through pyrolysis to biochar. Biochar is a carbonaceous material obtained through pyrolysis of plant and animal derived biomass such as plant residues, agricultural and food processing waste, sewage sludge or animal or poultry manure. It is not a new material but with modern analytical techniques and modifications the properties of biochars are now being rediscovered and investigated in detail [2]. Some of these biochar properties include chemical composition, surface area, microporosity and the presence of surface functional groups, and thus play an important role in the process of sorption. It has been observed that the efficiency of sorption depends on surface area, porosity and cation exchange capacity of biochars, and also thermal stability [3]. Other studies indicated that the presence of non carbonized organic matter, hydrophobicity and aromaticity of biochars can affect sorption of selected contaminants [4]. The presence of lignocellulose and various elements in substrates can have an effect on the process of sorption of organic contaminants by biochars [5]. It has been reported that the presence of surface functional groups in biochars could indicate their potential towards selected applications, primarily the removal of different contaminants from the environment such as heavy metals from water or wastewater [6]. For example, biochars with surface functional groups that contain oxygen, demonstrate ability to immobilize heavy metals such as lead (Pb^{2+}), copper (Cu^{2+}), nickel (Ni^{2+}) and cadmium (Cd^{2+}) [7].

The literature reports growing interest in investigating the properties of poultry manure derived biochars and their potentials for removal of various contaminants. Most of the reported studies focuses on the effect of process parameters such as temperature on the properties of the produced biochars [8]. Novak *et al.* [9] investigated how biochars with diverse specific surface area, the content of carbon, hydrogen and oxygen, and also ash contributed to the improvement of chemical and physical properties of soil. Other researchers observed that changing pyrolysis temperature allowed obtaining biochars with wide range of alkalinity which in turn could determine their application to different types of soil [10]. Also, many studies focused on investigating mechanisms for binding organic contaminants [11]. Not only mechanisms for binding organic contaminants but also inorganic contaminants have been studied extensively. Qi *et al.* [12] studied how selected biochar properties can facilitate the process of sorption with special reference to inorganic contaminants. Based on the reported research, poultry derived biochars can demonstrate similar potential as sorbents for removal of selected volatile organic compounds [4]. It was observed that biochars obtained from different kinds of manure demonstrated potential for sorption of selected organic micropollutants from water and wastewater [13]. For example, biochars can be efficient in removal of salicylic acid [5]. Other researchers investigated the application of biochars for removal of phthalate acid esters from water [14]. It is worth to emphasize that biochars are investigated as sorbents for removal of inorganic compounds such as heavy metals (e.g., lead) [15]. The literature provides a number of examples where biochars demonstrated potentials for removal of nitrogen compounds. For example, Tang *et al.* [16] used biochars produced from sewage sludge to remove ammonium from wastewater. These properties of biochars allow potentially a wide range of applications in order to mitigate the environmental pressures. Undoubtedly, the greatest potential for biochar is to be used as an additive for improving soil properties [17]. However, many researchers point out the potential of biochar for removal of various contaminants from water and wastewater [18]. Therefore, biochars obtained from pyrolysis of poultry manure

are the subject of many investigations. This is mostly due to the fact that poultry manure derived biochars demonstrate higher ash contents, and thus higher sorption efficiencies for organic contaminants. It was pointed out that the content of mineral ash provides additional space for binding cations and/or anions [19].

However, the literature does not provide sufficient information on the properties of poultry derived biochars, in particular chemical composition, surface area and the presence of functional groups and how these properties are affected by different pyrolysis temperatures. Therefore, there is a need for better understanding how pyrolysis temperature can be used to modify, and thus tailor the properties of poultry derived biochars to be used as sorbents. In addition, knowledge about the key properties of poultry derived biochars – being crucial in the process of sorption – could be a starting point for further modifications of biochar properties, e.g. by physical or chemical methods.

The overall goal of this work was to investigate the properties of biochars obtained from poultry manure through pyrolysis and the effect of pyrolysis temperatures on biochar properties such as chemical composition, surface area and the presence of functional groups. The scope of this work included:

- Collection and pretreatment of poultry manure sampled from an organic poultry farm;
- The analysis of air-dried poultry manure;
- Pyrolysis of poultry manure into biochar in a laboratory pyrolysis reactor in the range of temperatures from 400 to 700 °C;
- The analysis of the selected properties of poultry manure derived biochars, with special reference to chemical composition, surface area and surface functional groups.

MATERIALS AND METHODS

The presented work was performed at the Department of Environmental Engineering, Częstochowa University of Technology, Poland.

Poultry manure and pyrolysis parameters

Poultry manure – already semi-dry – was sampled from an organic poultry farm (Poland), let to further air dry and subjected to pyrolysis in a continuous pyrolysis reactor (PRW-S100x780/11). The heating temperatures of the poultry manure samples were: 400 °C, 500 °C, 600 °C and 700 °C. The heating and retention times were 120 min and 60 min, respectively. After the process of pyrolysis was completed, the samples were left in the reactor until they reached room temperature [20].

Physicochemical and physical analysis

Poultry manure and poultry derived biochars (referred here as BPK) were analyzed for moisture content (by oven drying in 105 °C), organic matter (by incineration in a muffle furnace at 550 °C for 5 h), total carbon (by MultiN/C, Analytkjena, in 4 replications), pH (1:10 v/v, in 2 replications) and also total and bioavailable phosphorous. BPK at selected temperatures were analyzed for chemical composition, surface area, morphology, presence of surface functional groups. The analytical techniques included Scanning Electron Microscopy (SEM) and Fourier Transmission Infrared spectroscopy (FTIR). The carbon, hydrogen, nitrogen, sulfur (CHNS) elementary analysis was performed with the Thermo Scientific™ FLASH 2000.

RESULTS AND DISCUSSION

Selected physicochemical properties of poultry manure

Poultry manure (Figure 1a) demonstrated Moisture Content (MC) of 8%, Organic Matter (OM) of 55% (dry basis), Total Organic Carbon (TOC) of 15% (dry basis) and pH of 6.77.

The yield of pyrolysis of poultry manure to biochar decreased with the increase in temperature and amounted to 62%, 55%, 57% and 55% for temperatures of 400 °C, 500 °C, 600 °C and 700 °C, respectively. This is in line with the other results described in the literature. Song and Guo [8] reported significant decrease in the yield of poultry litter pyrolysis with the increase in the process temperatures (from 60.13% to 45.71%). Wystalska *et al.* [20], who investigated the efficiency of pyrolysis of various substrates such as sunflower husks observed similar relationship. This was also confirmed by other researchers [21]. Conversion of biomass through pyrolysis at higher temperatures results in lower yields of solid products [22].

Total and bioavailable phosphorous of poultry manure was of 15.36 and 2.514 g kg⁻¹, respectively.



Figure 1. Poultry manure (a) and poultry manure derived biochar (b)

Basic biochar characteristics

The basic characteristics of the obtained biochars (Table 1) included MC, OM, pH, TOC and BET. The moisture contents of BPKs was below 1%, organic matter ranged from 37 to 25% , total organic carbon ranged from 15 to 27%, pH ranged from 10 to 12 (depending on the temperature) and surface area was very low.

Table 1. Physicochemical properties of the obtained biochars obtained at selected temperatures

Sample	Temperature [°C]	MC [%]	OM [%]	pH (H ₂ O) _{H2O}	BET [%]	TOC [%]
BPK400	400	0.72 ±0.053	37.99 ±1.735	10.08	4.5375 ±0.1055	14.9 ±3.9
BPK500	500	0.42 ±0.111	28.62 ±1.050	11.16	5.0108 ±0.0779	28.3 ±7.4
BPK600	600	0.48 ±0.095	25.88 ±0.140	11.90	2.9517 ±0.0531	26.3 ±6.8
BPK700	700	0.13 ±0.021	23.80 ±0.831	12.38	5.6459 ±0.1028	27.3 ±7.1

The CNHS analysis (Table 2) indicated that carbon (C) was of 32-35% for tested temperatures. Hydrogen (H) and nitrogen (N) decreased with increasing temperatures whereas sulfur (S) was not detected.

Table 2. The CNHS elemental analysis of the obtained biochars obtained at selected temperatures (% , dry weigh basis)

Sample	Temperature [°C]	N [%]	C [%]	H [%]	S [%]
BPK400	400	3.35 ±0.53	34.58 ±8.58	2.28 ±0.45	-
BPK500	500	2.65 ±0.89	35.02 ±2.67	1.61 ±0.28	-
BPK600	600	2.34 ±0.36	34.93 ±2.89	1.13 ±0.22	-
BPK700	700	1.75 ±1.18	32.52 ±8.47	0.97 ±0.46	-

The produced biochars demonstrated typical properties of biochars produced from poultry manure [23]. These properties are strongly affected by the applied pyrolysis temperatures [24]. The impact of pyrolysis temperature on selected biochar properties was observed for chemical composition and pH. With the increase in temperature chemical composition changed – decrease in the contents of OM, N, C and H was reported.

Selected physicochemical properties of biochars

The surface structure of poultry derived biochars obtained at different pyrolysis temperatures is presented in Scanning Electron Microscope (SEM) photos in Figure 2.

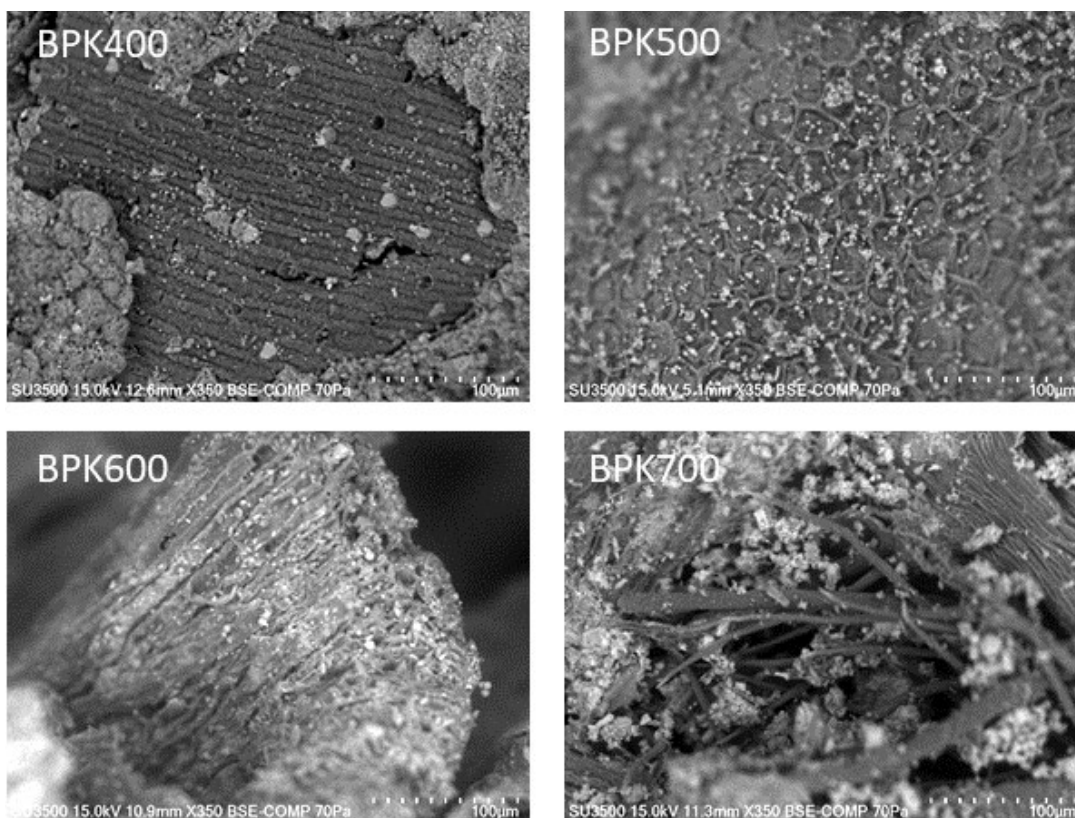


Figure 2. SEM images of the produced poultry derived biochars at selected temperatures (400 °C, 500 °C, 600 °C and 700 °C)

The structure of the PMBs produced at different pyrolysis temperatures are shown in SEM photos in Figure 2. They reveal a structurally differentiated material with heterogeneous surface morphology containing macropores, space voids, fibrous motifs as well as irregularly shaped particles on the surface. Macropores are often aligned into honeycomb-like patterns most likely reflecting the carbonaceous skeleton of the raw material. The irregular particles visible as brighter spots randomly distributed on the biochar surface are composed of inorganic salts excreted by poultry. This observation is confirmed by the Energy Dispersive Spectroscopy (EDS) analysis of the surface showing significant variations of the content of particular elements, particularly carbon and metals like silicon (Si), potassium (K), phosphorus (P), calcium (Ca) or iron (Fe) depending on the local morphology of the surface. In the case of the above-mentioned brighter spots, a higher content of inorganic phase is observed, marked by elevated level of potassium while the content of carbon is lower when compare to the area 1 (Figure 3). It should be kept in mind that the interpretation of the EDS data should be carried out with caution due to its local nature and wide range of light elements, for which accuracy of determination is low.

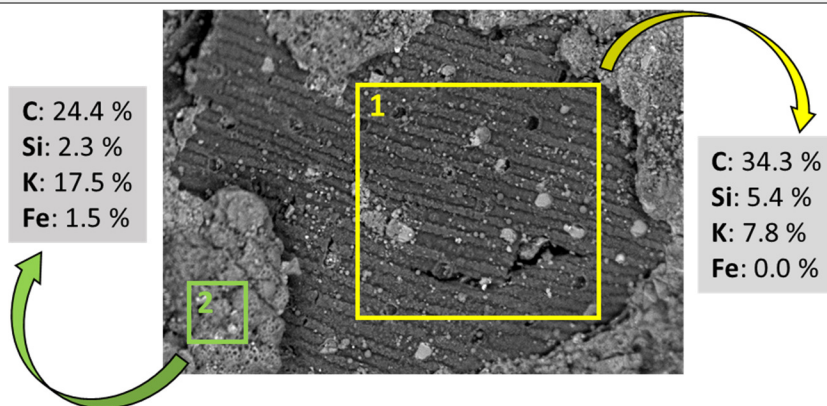


Figure 3. EDS analysis of the two selected regions of BPK400 biochar (the contents presented only for selected elements)

In contrast to the EDS, the CHNS elemental analysis is a technique where averaged elemental composition can be precisely determined, however only for certain elements (Table 2). As it can be seen, the content of carbon is similar regardless the temperature applied during the pyrolysis of the poultry manure. This is due to the high content of volatile materials in the feedstock, that are rapidly volatilized at the initial stage of the pyrolysis process [25]. However, the nitrogen and hydrogen content decreases as the pyrolysis temperature increases due to the cleavage and breakage of weak bonds within the biochar structure. Sulfur was not detected in biochars, regardless the pyrolysis temperature.

To have more detailed view of the chemical changes provoked by the different pyrolysis temperatures, Fourier Transform Infrared spectroscopy/Attenuated Total Reflectance (FTIR/ATR) spectra of the biochars were recorded and are presented in Figure 4.

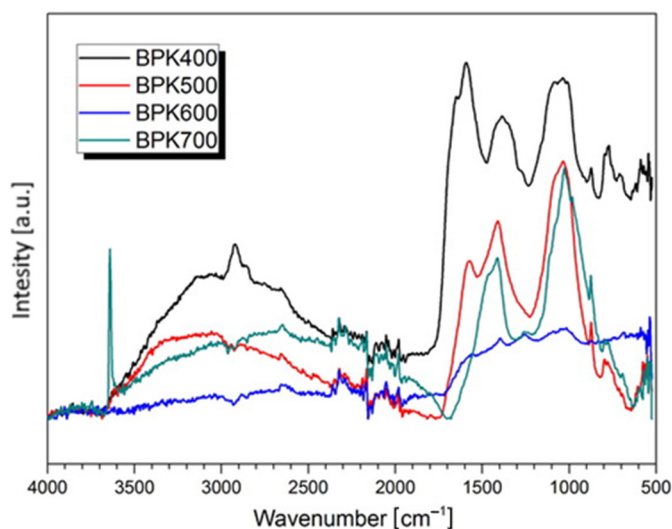


Figure 4. FTIR/ATR spectra of poultry derived biochars produced at selected temperatures (400 °C, 500 °C, 600 °C and 700 °C)

The broad band on the spectrum of the biochar BPK400 extending from $\sim 3,700\text{ cm}^{-1}$ to $\sim 2,400\text{ cm}^{-1}$ and centered at $\sim 3,100\text{ cm}^{-1}$ is assigned to the asymmetric and symmetric stretching modes of water (residual and crystallization). The intensity of this signal is significantly reduced in the case of remaining biochars what testify that the residual and possibly some part of crystallization water is released at temperatures higher than 400 °C. This signal has the highest intensity for BPK400.

In the region $\sim 2,930\text{-}2,850\text{ cm}^{-1}$ of FTIR spectrum of BPK400, there is a group of bands attributed to the stretching modes of C-H groups (i.e., CH_2 and CH_3) present in aliphatic compounds [26]. This group of bands is not so intense for biochars pyrolysed at temperatures $500\text{-}700\text{ }^\circ\text{C}$, most probably due to the breaking of relatively weak C-H bonds [10]. The presence of CH groups is additionally confirmed by the presence of the bands in the region $1,415\text{-}1,375\text{ cm}^{-1}$ attributed to the deformation modes of those groups [10].

Stretching modes of C-C bonds in aromatic rings are manifested by the presence of series of bands in the range $1,595\text{-}1,550\text{ cm}^{-1}$ for biochars pyrolysed at temperatures $400\text{-}600\text{ }^\circ\text{C}$. Their intensity decreases when pyrolysis temperature increases, however, in the case of BPK700 biochar pyrolysed at $700\text{ }^\circ\text{C}$ the intensities again increases. This phenomenon may be explained by the gradual decomposition of lignin, which is a polymer composed of cross-linked phenylpropane units often present in a large quantities in poultry manure [27]. Further increase of temperature to $700\text{ }^\circ\text{C}$ may result in remarkable aromatization resulting in creation of phenolic structures coming from dehydration of lignin combined with carbonization of carbohydrates [28]. This hypothesis is supported by the fact that on the FTIR spectrum of BPK700 biochar there is sharp and intensive signal at $3,641\text{ cm}^{-1}$, which can be attributed to the stretching modes of free hydroxyl groups [29]. This signal is not present in the case of the remaining samples pyrolysed at lower temperatures. It should be mentioned here that lignin is particularly difficult to biodegrade, and reduces the bioavailability of any biochar, thus its thermally-induced degradation on the course of the preparation of BPK700 biochar is of paramount importance.

The strong signal at $\sim 1,620\text{ cm}^{-1}$ on the spectrum of BPK400 biochar can be attributed to the C=O stretching modes indicating the presence of ketones, quinones, or/and carboxylic carbon [30]. However, its intensity significantly decreases for BPK500 and totally disappears for BPK700. The signal located at $\sim 1,420\text{-}1,400\text{ cm}^{-1}$ can be attributed to the C=C stretching modes present in olefins and aromatic rings. The relative intensity of this signal increases with increasing pyrolysis temperature indicating that formation of aromatic carbon takes place when the pyrolysis temperature increase. This hypothesis is additionally confirmed by the results of elemental analysis showing decreasing H/C ratio with increasing pyrolysis temperature (Table 1).

Stretching modes of C-O bonds are observed in the range of $\sim 1,100\text{-}1,020\text{ cm}^{-1}$ for all biochars [30]. The corresponding signals have the highest intensity among all the signals present on FTIR spectra. Their presence may also suggest formation of aromatic ethers via incorporation of oxygen atoms into cyclic carbon structures [31]. On the other hand, the absorption bands located at $\sim 1,050\text{-}850\text{ cm}^{-1}$ can be attributed to C-O stretching and O-H deformation modes in alcohols, phenols, ethers and esters [31]. The presence of oxygen-containing groups on the biochar surface is very important as they play a significant role in the immobilisation of heavy metals present in soil followed by formation of metalorganic complexes [32]. Wide range of metal ions have been reported to be bound with the oxygen functionalities and including aluminum (Al^{3+}), iron ($\text{Fe}^{3+}/\text{Fe}^{2+}$), calcium (Ca^{2+}) but also hazardous heavy metals ions like lead (Pb^{2+}), copper (Cu^{2+}), nickel (Ni^{2+}), and cadmium (Cd^{2+}) [2].

CONCLUSIONS

Poultry manure can be successfully converted through pyrolysis to biochars with the yield depending on the pyrolysis temperature as reported previously in the literature. This was also confirmed by our study. The temperature of pyrolysis had the effect on the selected biochar properties. Pyrolysis temperature altered the chemical composition (as evidenced by elemental analysis) and the type and number of functional groups (as evidenced by FTIR spectroscopy). While content of carbon did not change

significantly, the contents of nitrogen and hydrogen decreased with increasing pyrolysis temperature. Two most important chemical changes from the point of view of potential applications were: gradual aromatization of the investigated biochar and formation of wide range of oxygen functionalities. The specific surface areas (BET) of biochars were not affected by pyrolysis temperature and were similar to other manure-derived biochars reported in the literature.

Another conclusion from this study is that the properties of poultry manure derived biochars, in particular low surface area and the presence of surface functional groups, would require modifications by employing physical, chemical or thermal process to be applied, e.g., as sorbents/adsorbents to remove selected contaminants from the environment. Although, the chemical composition of produced biochars suggests that those biochars show some potential as soil improvers or composting amendments, this would require further studies.

The presented results constituted the preliminary study for the project Nutri2Cycle which has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 773682 and further work on converting poultry manure into biochars will be continued within this project. These results were obtained in order to continue work on designing biochar properties for various environmental applications. Future work will include selecting and optimizing the processes for modification of the properties of poultry derived biochars and testing them as sorbents/adsorbents for removal of heavy metals from aqueous media.

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