



Immobilization of phosphorus in cow manure during hydrothermal carbonization



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ABSTRACT

The surplus of manure phosphorus (P) with increasing livestock production might pose a risk of P loss to the environment due to the high mobility of P in manure. Thus, there is an increasing need to mitigate P loss from manure. This study aimed to investigate the effect of hydrothermal carbonization (HTC) on the immobilization of P in cow manure. The results demonstrated that the P content in cow manure was increased substantially by ~20% after HTC, while the water-extractable P (WEP) and Mehlich-3-extractable P (MEP) in manure was reduced significantly by >80% and 50%, respectively. The decrease in P solubility might result from the increased apatite P (increased by >85%) and decreased soluble Ca (decreased by ~50%) after HTC. These results suggested that HTC could be an efficient strategy to immobilize P in cow manure, thereby potentially mitigating the P loss problem from cow manure.

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1. Introduction

With increasing livestock production over recent decades, enormous amounts of phosphorus (P) are accumulated in manure (Bouwman et al., 2013). Manure storage-related issues (Brands, 2014; Vadas and Powell, 2013) and the soil application of manure (Liu et al., 2014) might pose a potential risk of P loss as the nonpoint source to environment because of the high solubility of P in manure (Glaesner et al., 2012; Liang et al., 2014). For example, McDowell et al. (2001) reported that water-soluble P accounted for 50% of the inorganic P in dairy manure, and that 95% of the water-soluble P was released during rainfall. Thus, regulations for manure management (Ribaud, 2007) and methods for P immobilization in manure, e.g., mineral amendments using aluminum- and calcium-containing chemicals (Armstrong et al., 2012; Wagner et al., 2008; Wilson et al., 2008), were developed to minimize P loss from manure.

The P in waste biomass after thermal treatment is much less soluble than is that in untreated waste biomass (Liang et al., 2014; Qian and Jiang, 2014), suggesting that the thermal treatment of manure could be an emerging and promising strategy to control the

P loss problem associated with waste biomass such as manure. Moreover, the process of thermal treatment could simultaneously destroy pathogens, remove odor compounds, and eliminate soluble toxic metals in the manure (Reza et al., 2013). The land applications of the biochar derived from thermal treatment of biomass have several implications in many fields, including manure treatment, crop production (Asai et al., 2009), soil amendment (Githinji, 2014), remediation (Cao et al., 2011), and carbon sequestration (Lee et al., 2010). However, although some attention has been paid to the P-release characteristics of biochar (Angst and Sohi, 2013; Liang et al., 2014; Wei et al., 2013), little is known about the speciation and solubility of P in manure-derived biochar from hydrothermal carbonization (HTC) (Qian and Jiang, 2014; Shi et al., 2014). Therefore, few studies have analyzed the management of P loss from manure by converting manure into biochar using HTC.

HTC is a thermal–chemical treatment that uses facile processing temperatures (130–250 °C) and water as the carbonization medium under self-generated pressures (Titirici et al., 2012), and has been used as a cost-effective technique for converting biomass into valuable functional carbonaceous materials (Dai et al., 2014; Titirici et al., 2012), converting organic wastes into bio-coal (Oliveira et al., 2013), or extracting biooil from organic waste (Dong et al., 2009). The feedstock for HTC could avoid drying because the carbonization takes place in water. Thus, compared with traditional thermal treatment (pyrolysis), which is conducted at a higher temperature

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(300–700 °C), HTC is more competitive for the thermal–chemical treatment of manure with a high level of moisture. However, HTC is not applied widely in the thermal–chemical treatment of manure, and little is known about the change in P speciation and solubility in manure after HTC.

In the current study, cow manure with a high moisture content was treated directly under hydrothermal conditions. The overarching objective of this study was to explore the possibility of using HTC in the management of P loss in manure. Specifically, this study aimed to explore the P speciation and solubility in untreated manure and manure-derived biochar from HTC to elucidate the effects of HTC on the immobilization of P in cow manure.

2. Materials and methods

2.1. Materials

Cow manure (one bucket, 20 L) was collected from a dairy farm in Chengdu, China, and kept at 4 °C until use. A polytetrafluoroethylene-lined autoclave (50 mL) was purchased from Dongtai Zhongkaiya Stainless Steel Co., Ltd (in Dongtai, Jiangsu Province).

2.2. HTC of cow manure

Wet cow manure (20 g) with an 85% moisture content was added to each of the autoclaves, which were then heated to 200 °C for 4, 6, 8, 10, 12, and 24 h. After treatment the autoclaves were removed from the oven and cooled in ambient air. The hydrothermal-treated cow manure in the autoclave was dried for 12 h at 105 °C, and then ground and sieved through 80 meshes for further analysis.

2.3. Chemical analysis

The ash content was measured using a proximate analyzer (TE-IA810). The carbon content was measured using Shimadzu TOC-V_{Series} SSM-5000A. The Fe, Al, Ca, and Mg content was measured using ICP-OES (Optima 5300V, Perkin Elmer) after fusing 0.5 g of sample with 0.2 g of LiBO₂, and then extracted with 4% HNO₃. All tests were conducted in duplicate.

Water-extractable P (WEP) and water-soluble Ca were extracted by mixing 0.06 g of sample with 20 mL of ddH₂O, shaken for 24 h at 200 rpm, and then centrifuged at 4500 × g for 10 min. Mehlich-3-extractable P (MEP) was extracted by mixing 0.05 g of sample with 1 mL of Mehlich-3 solution at 200 rpm for 5 min, and the mixture was then centrifuged at 4500 × g for 10 min. The supernatant was filtered through a 0.22-μm filter and P was measured using a methylene blue colorimetric method (Wei et al., 2002). For P release kinetics, 0.1 g of each sample was mixed with 40 mL solution (ddH₂O or 40 mg P L⁻¹ KH₂PO₄ solution) at an initial pH of 7.5 ± 0.05 in 50 mL polyethylene tubes. The mixture was then stirred at 200 rpm and 25 °C. The pH of the mixture was measured, and 1-mL samples were collected after 2, 4, 8, 12, 24, 36, and 48 h, respectively. The sampled mixture was filtered through a 0.22-μm syringe filter, and P was measured. All these tests were conducted in duplicate.

The amount of P in each samples was analyzed using the standard measurement and test (SMT) procedure (Ruban et al., 1999). According to SMT procedure there are five P fractions: total P (TP), inorganic P (IP), organic P (OP), non-apatite inorganic P (NaOH–P), and apatite P (HCl–P). All tests were conducted in duplicate.

3. Results and discussion

3.1. The changes in C, ash and Ca content

The C content in cow manure was increased from 46.31% to 53.89% in the biochar after 4 h HTC (Tables 1 and 2), and peaked in the biochar obtained from 12 h HTC (60.32%; Table 2). The increase of C content was attributed to the dehydration and decarboxylation of lignocellulosic biomass under HTC conditions (Falco et al., 2011). The accumulation of C in gas phase (such as CO₂) during HTC might lead to the slightly decline of C in the resulting biochar obtained from 24 h HTC (Lu et al., 2012).

The content of Ca in cow manure was much higher than the content of Mg, Fe, and Al (Table 1), so only the change of Ca after HTC treatment was analyzed. The content of ash and Ca in cow manure increased gradually with increasing times of HTC. Specifically, they increased by 3.81% and 24.24% in the biochar obtained from 4 h HTC, respectively, and by 19.00% and 47.19% in the biochar obtained from 24 h HTC (Tables 1 and 2). The increase in ash and Ca might be attributed to the fact that the solid and liquid products after HTC were not separated before drying. Actually, inorganics in biomass could be removed to liquid phase after HTC, leading to the reduction of ash and mineral elements in solid products of HTC (Reza et al., 2013).

3.2. Water-soluble Ca

The amount of water-soluble Ca in cow manure was 12.02 mg g⁻¹ (Fig. 1), which was reduced remarkably to 6.02 mg g⁻¹ in the biochar after 4 h HTC (Fig. 1). The percentage of water-soluble Ca among total Ca in cow manure was reduced remarkably from 52.02% to ~20% after HTC (Fig. 1). Ca precipitates could be formed under hydrothermal conditions when there were enough anions (such as phosphate, carbonate), which can result in the decrease of the solubility of Ca. The results suggest that HTC could reduce the solubility of Ca substantially, although the HTC time had little effect on the percentage of water-soluble Ca among total Ca, which might be due to the fact that the precipitation of Ca was occurred in a short time (Mekmene et al., 2009). These results were also observed after the pyrolysis of dairy manure (Liang et al., 2014).

3.3. Phosphorus speciation

The environmental fate of P in manure is controlled partly by its P composition and solubility. Sequential fractionation involves extracting P from manure using specific chemical solutions to quantify P species with varying solubilities. NaOH–P, which measures P bound to Al, Fe, and Mn (hydr)oxides, is assumed to be labile P. HCl–P is generally referred to as apatite–P or Ca-bound P. Organic–P (OP) is a complex fraction that includes phospholipids, DNA, and simple phosphate monoesters, which are mobile in soil. The amount of NaOH–P and OP in cow manure was 2.43 and 3.40 mg g⁻¹ (Fig. 2a), respectively, and accounted for 17.78% and 24.88% of the TP in manure (Fig. 2b). This was reduced significantly to <1.05 and 0.79 mg g⁻¹ (Fig. 2a), respectively, which accounted for <6.41% and 4.83% (Fig. 2b) of the TP after HTC. In contrast, there was 7.84 mg g⁻¹ apatite–P in cow manure (Fig. 2a), accounting for 57.35% of the TP (Fig. 2b), which was increased significantly to >14.51 mg g⁻¹ (Fig. 2a), or >88.76% of the TP (Fig. 2b), after HTC. The amount of NaOH–P and OP in the resulting biochar decreased gradually with increasing HTC time, whereas the amount of HCl–P increased gradually with increasing HTC time. This suggests that P in cow manure is immobilized as more stable forms of P during HTC (Fig. 2). These results are consistent with the change in P species in sewage sludge during thermal treatment (pyrolysis) reported

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