



Process conditions of pyrolysis and hydrothermal carbonization affect the potential of sewage sludge for soil carbon sequestration and amelioration



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ABSTRACT

Anaerobic thermochemical carbonization can be used to treat sewage sludge and facilitate its reuse. The aim of this study was to compare and evaluate chars produced by pyrolysis and hydrothermal carbonization (HTC) of sewage sludge, so-called *sewchars*, under varying process conditions in terms of their soil amelioration potential. Three different sewage sludges, two pyrolysis and four HTC *sewchars* were evaluated with respect to their stability in soil, perspective for nutrient recycling and potential use as soil amendments. The *sewchars* were produced at different temperatures (pyrolytic low temperature conversion LTC 400 °C; PYREG pyrolysis 700 °C; HTC 180–200 °C) and for varying treatment times (LTC and PYREG 1 h; HTC: 4–8 h). O/C and H/C ratios of about 0.7 and 1.8 respectively, indicated that HTC *sewchars* will tend to be less recalcitrant in soils than pyrolysis *sewchars*. The duration and temperature of HTC correlated positively with the chars' contents of adsorbed labile carbon and nitrogen compounds. However, HTC failed to increase the chars' resin-extractable phosphorus contents. The cation exchange capacity of sewage sludge was reduced by HTC but stayed on a higher level than after pyrolysis. With increasing treatment temperature, the pH, ash (content) as well as macro- and micronutrient contents increased. Pyrolysis *sewchars* also accumulated heavy metals; however their contents were lower than those reported previously. An enrichment of heavy metals was also observed for the HTC *sewchars*, albeit to a much lesser extent.

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1. Introduction

Most soils in excessively managed agricultural ecosystems have lost significant amounts of soil organic carbon (SOC) in the course of their cultivation [1]. This loss may adversely affect important soil chemical and physical properties such as nutrient storage, soil aggregate formation, and organic matter stability in the long term [2]. A promising way to mitigate anthropogenic climate change, restore organic carbon to soils and promoting carbon sequestration is to establish a carbon sink by applying biochar as soil amendment [3]. According to Lehmann and Joseph [4] the term *biochar* means charred organic matter produced by pyrolysis. It is resistant to degradation and recognized as a useful additive for increasing

soil fertility [5]. The properties of biochar and, hence, their effects on soils and plants can vary widely depending on the conditions used in their production and the feedstocks from which they are derived.

While pyrolysis is one way of producing chars, they can also be generated by hydrothermal carbonization (HTC). The advantage of HTC is that wet feedstocks can be converted into carbonaceous solids without the need for an energy-intensive pre-drying process [6]. The exothermic dehydration reactions take place in an aqueous environment in a pressure reactor under slightly acidic conditions at temperatures of around 200 °C [7]. The compatibility with moisture makes HTC a promising method for producing chars from feedstocks such as sewage sludge, animal manure and human waste [6,8].

Globally, there is an urgent need for sustainable ways of treating and recycling large quantities of carbon- and plant nutrient-rich sewage sludge. Converting this sludge into carbonaceous material for treating soils could be economically and ecologically benefi-

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cial [9]. Pyrolysis and HTC remove pathogens and can potentially degrade thermally labile pollutants of these materials, enabling the sustainable recovery of plant mineral nutrients that are enriched in sewage sludge [6,10]. The generation of so-called *sewchars* could provide environmental benefits by helping to mitigate climate change and ameliorate soils as well as being beneficial for human health and environmental protection [11]. Moreover, the thermochemical treatment of sewage sludge could improve wastewater and sludge treatment processes at sewage plants. Specifically, HTC causes the cytolysis of microbial cells and the hydrolysis of water-binding organic molecules, thereby increasing the mechanical dewaterability of sewage sludge and facilitating improvements in the energetic and economic balance of sludge treatment processes [12,13].

Gasco et al. [14] produced biochar from sewage sludge for the preparation of potential carbon based adsorbents. However, using the carbonaceous material as a soil amendment has barely been considered yet. Similar to chars produced from other biomass, the properties of *sewchars* (e.g. elemental composition, molecular structure, particle size) and therefore their effect within the soil-plant-system can vary widely depending on the thermochemical carbonization procedure (e.g. pyrolysis, HTC) and its process conditions (e.g. maximum temperature, heating rate, and residence time). Although basic information on the characteristics of pyrolysis derived biochar from sewage sludge is available [15–19] information on their potential agronomic effects is scarce [20–24]. Linking the properties of hydrochar from sewage sludge to their agronomic properties in terms of their carbon sequestration and soil amelioration potential has not been assessed in detail so far. Especially a comprehensive evaluation of the potential of pyrolytic and hydrothermal conversion technologies for a sustainable recovery of wastewater derived plant nutrients (e.g. N, P and K) are often not taken into consideration. In general, the recycling of nutrients during the HTC has been evaluated mainly for other feedstocks such as wheat straw [25], digestate [26] or animal manure [27].

Due to specific mobilization and immobilization reactions, the effects of pyrolysis and HTC processes on the amount and availability of plant nutrients can differ. In addition to direct fertilizing effects, carbonized biomass can increase the cation exchange capacity of soils and, thus, the capacity to retain cationic nutrient elements in exchangeable (i.e. plant available) forms for extended periods of time [28]. Further indirect effects of treatment with carbonized biomass can include changes in the soil pH and modifications of plant–microbe symbioses such as mycorrhizal and external associated symbiosis. Due to the binding of organic molecules and the precipitation of Fe- and Al-phosphates in tertiary wastewater treatment processes, sewage sludge is a significant P sink. The thermochemical treatment of sewage sludge may facilitate the liberation of precipitated phosphates and their conversion into forms that are directly available to plants as mentioned by Huang and Tang [29]. Zhang et al. [30] focused only on the effects of the temperature and reaction time on the elemental composition of hydrochar from sewage sludge.

A further important factor to consider when contemplating the treatment of soils with materials derived from sewage sludge is that wastewater treatment tends to concentrate heavy metals in the sludge fraction, which may limit its potential use as a soil amendment. The potential enrichment or depletion of heavy metals during pyrolysis or HTC of sewage sludge must therefore be taken into account if the resulting charred material is to be used for soil treatment.

It has been shown that hydrochar have a lower content of aromatic compounds and a higher proportion of labile carbon compounds than pyrolysis products, suggesting that they may be less stable and less suitable for long-term carbon sequestration [31]. However, there is evidence that on decadal scale, hydrochar might

lead to similar increases in soil C storage [32,33]. One way of assessing the potential short-term stability of *sewchars* in soils is to analyze their contents of labile carbon (C) and nitrogen (N).

Upon drying, non-volatile compounds present in the liquid phase will remain on the char [21], however this specific fraction has not been analyzed qualitatively and quantitatively so far. The hot and cold water extractabilities of soils have been used as sensitive measures of the sizes of their labile C and N fractions [34]; high extractability indicates the presence of readily degradable carbon (C) and nitrogen (N) fractions whereas low extractabilities indicate the presence of more stable C and N fractions that are more resistant to degradation.

Given the above context, the overall aim was to compare and evaluate *sewchars* in terms of their carbon sequestration and soil amelioration potential. Therefore, the detailed objectives of the study presented herein were (1) to investigate the basic physicochemical characteristics of *sewchars* produced from different sewage sludges by pyrolysis and HTC processes with different operating temperatures and durations; (2) to quantify the stability of these *sewchars*; (3) to evaluate the potential of pyrolytic and hydrothermal conversion technologies for nutrient recycling; and (4) to assess the accumulation or depletion of heavy metals.

2. Material and methods

2.1. Char production and sample preparation

Nine materials were included in the analysis – three feedstocks, two pyrolysis *sewchars*, and four HTC *sewchars*. The feedstocks were primary sludge (PS), activated sludge, unstabilized (ASU) and activated sludge, stabilized (ASS). The two pyrolysis *sewchars* were produced within 1 h at temperatures of 400 °C by thermocatalytic low temperature conversion (LTC, [35]) and 700 °C by the PYREG fast pyrolysis process [36], respectively. The four HTC *sewchars* were prepared by heating at temperatures (180–200 °C) for different length of time (4–8 h) and are referred to as HTC I, HTC II, HTC III and HTC IV. The detailed conditions and feedstocks are listed in Table 1. Unfortunately, samples of the activated sludge used to produce the PYREG *sewchar* were not available for analysis. The materials were provided by the following companies: PS and HTC I–III by ARTEC Biotechnologie GmbH (Bad Königshofen, Germany), ASU and HTC IV by TerraNova Energy GmbH (Düsseldorf, Germany), ASS and LTC by AZV Pfattertal (Mintraching, Germany) and fast pyrolysis *sewchar* by PYREG GmbH (Dörth, Germany). All materials were homogenized, milled, dried at 70 °C and stored in closed plastic cups at room temperature before analysis.

2.2. General analysis

The pH of the materials was measured using dried samples in H₂O (actual acidity) and in a 0.01 M solution of CaCl₂ (potential acidity). Samples extracted with 0.01 M CaCl₂ were shaken horizontally for 2 h and then filtered (Schleicher & Schuell 595 1/2, Dassel, Germany). After pH measurement, the resulting extracts were filtered and analyzed for their ammonium–N (NH₄⁺–N) and nitrate–N (NO₃[–]–N) concentrations with a flow injection analyzer (FIAsstar 5000, Foss GmbH, Rellingen, Germany).

Pore surface areas were determined by using a Brunauer–Emmett–Teller Surface Area (BET) analyzer (Belsorp II, MicrotracBEL, Japan) with nitrogen as the adsorbate [37]. The surface areas were determined based on the physical adsorption of nitrogen.

The samples' overall elemental composition, including heavy metals, was determined by microwave digestion and analyzed by ICP–OES (Spectro Ciros CCD, Spectro Analytical Instruments, Kleve;

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