



Supercritical water gasification of sewage sludge: Gas production and phosphorus recovery



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HIGHLIGHTS

- SCWG can be used for converting sewage sludge into energy and P-recovery.
- The efficiency of uncatalyzed gasification of sewage sludge was 52% at 600 °C.
- Phosphorus content went up from 3% in sewage sludge to 9% in the solid after SCWG.
- The highest release of phosphate from SCWG residue was 95.5%.
- Oxalic acid had better performance than sulfuric acid in P leaching (95%).

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ABSTRACT

In this study, the feasibility of the gasification of dewatered sewage sludge in supercritical water (SCW) for energy recovery combined with P-recovery from the solid residue generated in this process was investigated. SCWG temperature (400 °C, 500 °C, 600 °C) and residence time (15 min, 30 min, 60 min) were varied to investigate their effects on gas production and the P recovery by acid leaching. The results show that the dry gas composition for this uncatalyzed gasification of sewage sludge in SCW mainly comprised of CO₂, CO, CH₄, H₂, and some C2–C3 compounds. Higher temperatures and longer residence times favored the production of H₂ and CH₄. After SCWG, more than 95% of the P could be recovered from the solid residue by leaching with acids. SCWG combined with acid leaching seems an effective method for both energy recovery and high P recovery from sewage sludge.

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1. Introduction

Phosphorus is an important nutrient for all living organisms. It is part of a large number of biological systems and is a main constituent of fertilizers used in agriculture (Acelas et al., 2014). Almost all phosphorus used in fertilizers is derived from phosphate rock, and due to the decreasing availability and quality of mineral phosphate resources and the increase of the phosphate rock price, alternative resources for the production of phosphate fertilizers for agriculture must be found (Acelas et al., 2013; Cordell et al., 2009). Sewage sludge represents an important secondary phosphorus source for two main reasons. Firstly, during wastewater treatment with chemical and/or enhanced biological P-removal (EBPR) most

of the phosphate ends up in a concentrated form in the sewage sludge (Blöcher et al., 2012). Secondly, wastewater treatment facilities usually operate at an industrial scale, resulting in large amounts of the phosphorus-rich sludge. Technologies that reduce mass and volume of sewage sludge and at the same time produce a usable sludge product have gained more and more interest in recent years. Sludge combustion is widely practiced on a full-scale basis in many highly populated urban areas, e.g. in Japan, Germany, and the Netherlands (Lederer and Rechberger, 2010).

One way to recover phosphorus is from sewage sludge ashes produced in thermal processes, such as pyrolysis, combustion or gasification (Azura et al., 2013; Petzet et al., 2012; Yanagida et al., 2009). However, sewage sludge contains a high percentage of water, which causes high drying costs when these thermal processes are used. Therefore, supercritical water gasification (SCWG) has been identified as a promising technology for the conversion of wet biomass streams with a low overall heating value to high

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heating value product gases (Chakinala et al., 2013), while still having the possibility of phosphorus recovery.

In supercritical water (SCW), the pressure and temperature have been increased to or over the critical point (22.1 MPa and 374 °C). As a reaction medium, supercritical water is featured as having a high diffusivity, a low viscosity, and a high solvating ability for organic compounds (Youssef et al., 2010). In addition, water is an active reactant in steam reforming and water-gas shift reactions in the supercritical region. Therefore, supercritical water is able to transform sewage sludge into fuel gases such as H₂, CO, and CH₄ with reduced tar and coke formation compared to the traditional thermal processes (Zhang et al., 2010). The selectivity of the gas production toward H₂, syngas (H₂ + CO), or CH₄ can be steered by tuning the process conditions and by the use of catalysts (Chakinala et al., 2009).

From the perspective of cost-effective disposal of bio-based wastes, the costs associated with SCWG of sewage sludge may be high, but when the solid residue generated during SCWG is used to recover and recycle phosphorus, this makes the process more economical. In most SCWG studies however, most of the attention has been paid on energy recovery (Azadi et al., 2013; Wilkinson et al., 2012; Xu et al., 2013) and on catalysts to promote production yield of hydrogen from different types of biomass, among which sewage sludge (Fiori et al., 2012; Gasafi et al., 2008; Zhang et al., 2010), manure (Youssef et al., 2010) and wood (Yong and Matsumura, 2012).

To date, very few studies have reported on phosphorus recovery from solid residues generated during SCWG of sewage sludge. In a related study, (Zhu et al., 2011) showed that during SCWG of sewage sludge the majority of phosphorus ends up into the solid residue (20 mg P/g). This is in agreement with (Yanagida et al., 2008), who evaluated the behavior of some inorganic elements in poultry manure after SCWG and found that the liquid phase contained nearly all the N, K and Cl, while nearly all Ca, P and Si ended up in the solid phase. S was divided more or less evenly between both phases. Clearly, the solid residue after SCWG is a promising source of P.

The research aim of this work is to study the feasibility of the SCWG of sewage sludge in combination with P-recovery from the solid residue generated in this process. The effects of SCWG temperature and residence time are evaluated using a high-pressure autoclave and a heated fluidized bed. Acid leaching of phosphate from SCWG residue is studied with oxalic and sulfuric acid, and compared to ash resulting from combustion.

2. Methods

2.1. Raw material

The dewatered sewage sludge (DSS) used as feedstock for supercritical water gasification was collected from the wastewater treatment plant of the Water Board Regge and Dinkel located in Hengelo, the Netherlands (lat. 52.27957°, long. 6.77086°). The sludge was dried (105 °C, until constant weight), pulverized in a mortar and then sieved into two fractions of different particle size (the first: 600 μm ≤ Φ ≤ 1 mm, the second: Φ ≤ 600 μm), and stored in airtight containers. The material with particle size ≤ 600 μm was used in the SCWG experiments, because it was the largest fraction (by weight). The water content, as well as the proximate and the ultimate analysis of the dried dewatered sewage sludge (DDSS) are shown in Table 1. The C, H, and N content of the DDSS (dry ash free, daf) were measured in triplicate using an Elemental Analyzer (Flash 2000-Interscience), with oxygen calculated by difference. The higher heating value (HHV) was calculated according to the Dulong equation (Xu et al., 2012), using

the results of the elemental analysis. The mineral composition of the DDSS was determined by X-ray fluorescence spectrometry (XRF) and is shown in Table 2. For the XRF measurements samples were dissolved in lithiumtetraborate (Li₂B₄O₇, flux) to make fused beads. These were measured together with standards with known composition containing the same elements in the same range of concentration on a P analytical PW 1480 using the SuperQ software. Results were not normalized for loss on ignition.

2.2. Super-critical water gasification (SCWG) of sewage sludge

SCWG experiments were carried out in a batch autoclave reactor (Inconel alloy: ~72% Ni, 15% Cr and ~8% Fe) with an internal volume of 45 mL. A diagram of the set-up is shown in Fig. 1. For safety reasons, the reactor set-up was placed in a safety chamber and was controlled from outside the chamber during the experiments. The inner temperature of the reactor was measured by a thermocouple inserted through an orifice in the bottom cap, and the pressure was measured via a gas connection from the top lid to a pressure transmitter. The pressure and the temperature of the reactor were monitored and recorded using Pico Log software. With the help of a pneumatic arm, the autoclave was immersed in and removed from the fluidized sand bed. This sand bed was heated by an electric oven and preheated air which was also used for the fluidization of the bed.

In a typical run, the autoclave was loaded with 10 g of a mixture of 15 wt% of DDSS and 85 wt% of deionized water, after which the autoclave was tightly closed and connected to the pneumatic arm of the set-up. Prior to each experiment, a leakage test was performed by pressurizing the autoclave with 100 bar of nitrogen and monitoring pressure after closing the gate valve. This also served to remove any air from the reactor. Finally, after restoring ambient pressure in the reactor, the rest of the experiment was controlled from outside the safety chamber via the control panel. Using the pneumatic arm, the reaction was initiated by immersing the autoclave into the preheated fluidized sand bed. After the determined residence time, starting from when the set temperature was reached, the reactor was lifted and quenched in the cold water bath. Triplicate runs were conducted for each combination of temperature and reaction time.

2.3. Separation and analysis of the reaction products

The procedure for separating gasification products is shown in Fig. 2. Once the reactor was cooled down to room temperature and before depressurizing the system, gas samples were taken with a syringe and analyzed with an off-line gas chromatograph (Varian Micro GC CP-4900) containing two analytical columns (Molsieve 5A Ar, 10 m and PPQ He, 10 m) and equipped with a thermal conductivity detector to determine H₂, CO₂, CO, CH₄ and other light organic gases (C₂H₄, C₂H₆, etc).

The remaining product (liquid and solids in suspension; Product 1) was then collected in a polypropylene bottle. Then, the reactor was rinsed with de-ionized water to remove any solid deposits from the autoclave. The resulting suspension (Product 2) was also collected. The suspensions were separated into solid and liquid phases by vacuum filtration using a 1.0 μm pore size membrane filter (Glass microfiber filters. Whatman. GF/B). The filtrate from Product 1 was collected in a sampling vial to determine phosphorus concentration whereas the solid residue on the filter was collected and named Solid 1. The solid residue after the filtration of Product 2 was also collected and named Solid 2. Both solids were rinsed with demineralized water while on the filter, then the filter was dried at 105 °C until constant weight and then the solids were collected and mixed together in a sampling tube for analysis.

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