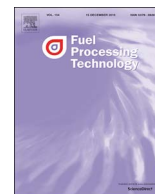




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Research article

Ash and slag properties for co-gasification of sewage sludge and coal: An experimentally validated modeling approach

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ABSTRACT

Regulatory changes in the EU and Germany, and environmental aspects direct the attention of the agricultural and energy sector towards sewage sludge as a new resource. The present study evaluates the co-gasification of sewage sludge with brown coal in a slagging gasifier. Currently, a lack of experimental data on phosphorous containing ashes and slags under reducing atmospheres limits our understanding of gasification of this fuel. To address this gap, in this study the ash and slag properties of one brown coal and its mixtures with sewage sludge were experimentally investigated under a reducing atmosphere and predicted with FactSage™. An ASPENplus™ simulation of the co-gasification is conducted to evaluate the impact of the changing feedstock characteristics on major process parameters when different mixtures of sewage sludge and brown coal are fed into a Siemens gasifier.

The flow temperature of the coal ash can noticeably be reduced if the sewage sludge ash content is increased up to 50 wt%. Thermochemical calculations using FactSage™ are in good agreement with the experimental ash fusion behavior. This paper describes a method to improve the calculation to better fit the experimental values. The elemental liberation of the main ash components are modeled using FactSage™ and experimentally validated. Except for potassium, no significant release into the gas phase was detected in the experiment. This is in contrast to the predicted evaporation of phosphorous. The addition of sewage sludge results in a lower viscosity of the slags of the mixtures in this study. As a result, the studied entrained-flow gasifier can be operated at lower temperatures while staying at an optimal viscosity for slag tapping. On the other hand, it decreases the temperature working range inside a slagging gasifier, where the slag viscosity lies between 2 and 25 Pa s. The resulting implications on the process parameters of entrained flow gasification have been modeled with ASPENplus™. Even low sewage sludge additions to the brown coal allow for lower gasifier temperatures, decreasing the specific oxygen consumption. Moreover, the cold gas efficiency and the H₂/CO ratio are increased.

1. Introduction

Current means of handling sewage sludge (landfills, fertilizer, incineration) are becoming less attractive due to changing regulations and concerns about pollution [1–3]. Moreover, sewage sludge disposal has a high cost [4] (from 50 to 120 € per ton [4]). Since it is a “renewable” feedstock and has a calorific value comparable to fossil fuels such as coal when dried [1], it can be used as an alternative feedstock and as an addition to fossil fuels. Processes such as combustion, pyrolysis, partial oxidation and gasification can serve this purpose. The latter three allow for the production of high value chemicals alongside heat and electricity generation (polygeneration).

This study focuses on the utilization of sewage sludge by gasification. The advantages of gasification are the generation of a very clean syngas. Moreover, the reduction in volume and thermal sludge disinfection during gasification enable disposal according to EU regulations at a reduced cost of disposal [1–3]. Furthermore, the often reported catalytic effect of sewage sludge [2,5,6] supports the heterogeneous gasification reactions. Any phosphorous enclosed in the glassy slag is conserved for later recovery. This conservation has the advantage that the vitrified resource is not quickly dispersed into the environment. This is also a disadvantage, because the enclosed phosphorous is also harder to recover. Both aspects have to be regarded. While there are studies on the utilization of sewage sludge in gasification, there are no

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studies on slagging gasification conditions. Namely, there is limited to no data on the viscosity of sewage sludge slags under a reducing environment. This study is the first published research on this topic and assesses the impact of sewage sludge in an entrained-flow gasification.

Ashes of sewage sludge are mainly dominated by the system $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-CaO-P}_2\text{O}_5\text{-Fe}_2\text{O}_3$ [3,7–14]. In some cases additional species or substitutions, like Fe_2O_3 is displaced by Na_2O , occur [10]. Nevertheless, distinct differences are found in the ash fusion behavior of various sewage sludge ashes (cf. refs. [7–10]). Their ash fusion interval from deformation temperature (DT) to flow temperature (FT) can range from 50 up to 400 K but for most of the sewage sludge ashes, it is found in the range between 80 and 200 K [3,7–9,13,14]. Their ash fusion behavior can be affected by $\text{Al}_2(\text{SO}_4)_3$ - or $\text{Fe}_2(\text{SO}_4)_3$ -based material if it is added for phosphorous capture [7]. Blending of sewage sludge and coal can have a strong impact on ash fusion temperatures (AFT). In the case of high AFTs of the pure fuel ashes, their blends may show lower temperatures. For low/intermediate temperatures of the individual ashes, the blends can end up with higher ash fusion temperatures or certain plateaus for extended mixture ranges [9,13,14]. The impact of blending is low if coal and sewage sludge have sufficiently similar ash compositions (cf. ref. [10]). For the particular role of P_2O_5 , especially its low-temperature eutectics and high temperature mineral phases formed with other oxides, the attention is directed towards refs. [9,15] (also to the references therein). Other positive effects when co-utilizing sewage sludge comprise an improvement of the ash fusion behavior and related slag formation at low temperatures of biomasses, and a retention of alkalis (fouling risk on heat exchangers) in the ash [11,15]. The effect on the ash fusion temperatures has to be understood prior to the prediction of the behavior of those complex multi-component systems.

In the present study, the ash fusion behavior, elemental liberation, and the slag viscosity of sewage sludge and its blends with coal are investigated. Additionally, it is required to understand the process performance of the different fuels and their mixtures. To this end, FactSage™ (ash fusion temperatures and release of oxides) and ASPENplus™ (process performance) are the chosen software for modeling the processes in this study.

2. Materials and methods

2.1. Materials

A brown coal and a representative mixture of several sewage sludge samples are analyzed in this study. The sewage sludge mixture was prepared by mixing 58 different sewage sludge samples and predominantly originates from municipalities. These samples were taken by RWE Power AG, Bergheim, Germany, from various wastewater treatment plants in Germany, the Netherlands, Belgium, and Luxembourg in the period between October 2014 and February 2015.

2.2. Experimental methods and modeling

2.2.1. Fuel preparation and ashing method

All fuels were dried to a constant weight at 105 °C and ground to particle sizes $\leq 200 \mu\text{m}$ prior to the fuel analyses and ashing. The ground samples were ashed according to DIN 51719 at 815 °C. Afterwards, the ashes were ground to particle sizes $\leq 63 \mu\text{m}$ for all experimental analyses. Besides the pure fuel ashes, ash blends with 10, 20, and 50 wt% sewage sludge ash addition were produced and investigated in this study. These ash-based blends have corresponded to contents of sewage sludge (moisture 78.5 wt%) of approximately 5.8, 12.2, and 35.7 wt% in a mixture with the raw brown coal (moisture 52.95 wt%). Despite the best efforts to homogenize all samples, there are slight differences between the ashes used for the different parts of the investigation. For example, a fused tablet was produced from all pure ashes and the blends for the determination of the ash composition by X-ray fluorescence analysis. This tablet was produced at

temperatures of around 1150 °C, which can result in a loss of certain amounts of volatile alkali oxides and sulfur due to the sulfate decomposition. This study aims to provide the necessary information to accurately connect the composition with the experimental results. Therefore, the compositions of the used individual ash samples were measured prior to each part of this study and are reported in this work.

2.2.2. Proximate and ultimate analysis

The proximate and ultimate analyses of both fuels were determined by using a muffle furnace and a vario MACRO from Elementar Analysensysteme GmbH according to the standards DIN 51718–51724.

2.2.3. X-ray fluorescence (XRF) analysis

The chemical composition of the ashes was determined by a S4 Pioneer XRF analyzer from Bruker AXS. For this purpose, fused tablets of the ash/lithium tetraborate and lithium metaborate were prepared. Herein, the ash was excited by Rh K_α radiation and finally element-specific X-rays are emitted, which were used for the determination of the contained element shares according to standard DIN 51729–10. The ash composition is given on an oxide basis. The relative experimental error is between 1 and 5 wt%.

2.2.4. Ash fusion behavior

The ash fusion behavior was studied using a Leitz heating microscope EM201 from Hesse Instruments according to the standard DIN 51730. For this purpose, small ash cylinders 3 mm in both height and diameter were formed from $\leq 63 \mu\text{m}$ ash powder. The four characteristic values of deformation, softening, hemispherical, and flow temperature (DT, ST, HT, and FT) were determined by a camera system and a related image analysis using the respective height-to-width ratio of the ash cylinder. Reducing conditions related to gasification were reproduced by an atmosphere composed of 65 vol% CO and 35 vol% CO_2 . The ash cylinder was continuously purged by this gas mixture during heat up at a rate of 10 K/min. The accuracy for the determination of the characteristic temperatures is 80 K (DT, FT) and 60 K (ST, HT) according to DIN 51730.

2.2.5. Thermochemical calculations

Thermochemical calculations were done using the Equilib module in the software package FactSage™ version 7.0 [16,17]. The chemical equilibrium was calculated by a minimization of the Gibbs free energy. Several practical limitations, like limited mass transport, unknown reactions and the formation of interfaces etc., are not or only partially considered in the calculations [18]. In principle, the software package has access to the most extensive thermochemical databases that are currently available. The databases FToxid, FTmisc, FTSalt and FactPS were used in that order. That is to say that in the case of database overlap for specific species, the order of priority for database selection was chosen like in the previous sentence. The selection of the solution species comprise: (1) A-Slag-liq, A-Olivine, $\text{M}_2\text{O}_3(\text{Corundum})$, Mullite (FToxid-Mull), $\text{Ca}(\text{Al,Fe})_2\text{O}_4$, $\text{K,Na//SO}_4,\text{CO}_3(\text{ss})$, and $\text{K,Na//CO}_3\text{-SO}_4(\text{ss})$ - each with two potential immiscible phases; (2) $[\text{Li}],\text{Na,K//SO}_4,\text{CO}_3(\text{ss})$ - with three potential immiscible phases, as well as (3) Fe-liq, A-Wollastonite, Cordierite, Nepheline, A-Melilite, $\text{NaAlO}_2\text{-HT}$, $\text{Ca}(\text{Al,Fe})_2\text{O}_9$, $\text{Ca}(\text{Al,Fe})_4\text{O}_7$, $\text{Ca}(\text{Al,Fe})_6\text{O}_{10}$, $\text{Ca}_2(\text{Al,Fe})_2\text{O}_5$, $\text{Ca}_3(\text{Al,Fe})_2\text{O}_6$, $\text{Ca}_2(\text{Al,Fe})_8\text{SiO}_{16}$, $(\text{Ca,Na}_2)\text{Ca}_8\text{Al}_6\text{O}_{18}$, $\text{Ca}_3\text{Ti}_2\text{O}_7\text{-Ca}_3\text{Ti}_2\text{O}_6$, $\text{Mg}_2\text{P}_2\text{O}_7\text{-HT}$, and $\text{K}_3\text{Na}(\text{SO}_4)_2$ - as single phases. A gas atmosphere consisting of 65 vol% CO and 35 vol% CO_2 was input into to the FactSage™ calculation, in addition to the ash composition, in order to account for the reducing atmosphere during the ash fusion test. The added mass of gas was 20-fold the mass of the total ash amount composed by the stoichiometry listed in Table 3. This ash-to-gas ratio can be varied in a broad range without a significant influence on the calculated mineral phases [18].

It is possible to determine the flow temperature via FactSage™ by calculating the temperature of the last solids melting or the first solid

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