



Full Length Article

Improving the processability of coke water slurries for entrained flow gasification



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ABSTRACT

A new stabilization concept for coke water slurries based on the capillary suspension phenomenon (Koo and Willenbacher, 2011) is presented. Adding a small amount of an immiscible, secondary fluid to the slurry results in the formation of a sample-spanning particle network controlled by capillary forces. This is accompanied by a strong increase in low shear viscosity controlling sedimentation, whereas the viscosity at high shear rates, relevant for twin-fluid atomization, remains unchanged. Wheat straw and beech wood coke, from fast pyrolysis, suspended in water (mass fraction $\phi_m = 20\%$) have been used as model systems and octanol-1 was added as secondary fluid ($\phi_{sf} = 0\text{--}3.1$ vol.%) to proof this concept. Visual inspection, centrifugation experiments as well as nuclear magnetic resonance imaging confirm the drastically increased sedimentation stability offering new opportunities for storage and transport of such slurries.

Atomization of the stabilized slurries was investigated using an external mixing atomizer varying gas-to-liquid ratio (GLR) between 0.5 and 1.5 at a constant liquid mass flow of 10 kg h^{-1} . Sauter mean diameter (SMD) of the created droplets decreases substantially with increasing secondary fluid content, especially at low GLR. SMD reduction is more pronounced for wheat straw than for beech wood coke slurries. These findings are attributed to the reduction of surface tension induced by the added octanol. Furthermore, the spray angle decreases with increasing octanol content at low GLR. The reported results provide valuable insight for suitable design of the gasification process.

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1. Introduction

Biomass is the only renewable carbon source and is supposed to become a major raw material for organic chemistry. Beside this, biomass can be used as fuel in common power plants by torrefaction pretreatment [2], to receive bio-gas (mixture of CH_4 and CO_2) from fermentation [3] or for synthesis gas production (mixture of CO , CO_2 , H_2 and H_2O) via gasification [4]. The synthesis gas can subsequently serve as fuel for a gas turbine, e.g. in an IGCC (Integrated Gasification Combined Cycle) to produce heat and power or chemicals through synthesis steps [5]. The bioliq[®]-process, developed at the Karlsruhe Institute of Technology, provides a new way to use residual biomass (e.g. wheat straw or beech wood). This is done in a two-step procedure. In a first step the biomass is converted by pyrolysis into a high energy density biogenic fluid

that is subsequently used as fuel for a high pressure entrained flow gasifier (EFG) [6]. The obtained synthesis gas can be converted by gas cleaning into high value products, such as methanol, liquefied petroleum gas (LPG) or synthetic natural gas (SNG) [7]. Flow behavior of the pyrolysis products is utterly important for almost all process steps. While storage stability (e.g. against sedimentation) is controlled by the viscosity at low shear rates, the intermediate shear rate regime is relevant for unit operations like pumping and mixing, finally the flow behavior at high shear rates is decisive for the atomization in the entrained flow gasifier. Rheology of coal water slurries and its effect on their stability and atomization behavior has been investigated previously. Strong non-Newtonian flow behavior at high particle loading was observed and the dependency of viscosity on particle volume fraction for different types of coal was described [8].

Mansour and Chigier showed experimentally that spray quality is mostly determined by the high shear limiting value of the apparent viscosity [9]. Working with an internal mixing twin-fluid jet

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atomizer Tsai and Vu confirmed that slurry viscosity has a strong impact on airblast atomization quality but also emphasize the relevance of particle size distribution [10]. Zhao et al. investigated break-up regimes and jet break-up length in highly viscous coal water slurries. Rayleigh type break-up is dominant for these systems and jet break-up length as well as droplet size increase with increasing viscosity [11]. Furthermore, slurry viscoelasticity can also have an impact on the spray structures. Shear wave structure is common for low viscoelasticity while jet oscillation is predominant for high viscoelasticity and additionally the occurrence of periodic spray structures depending on slurry viscosity is reported [12]. Atomization at increased reactor pressure was also examined using an external mixing twin-fluid atomizer because most EFG operate at increased ambient pressure to account for the demand of subsequent process steps. At constant aerodynamic We number an increase of droplet size with increasing ambient pressure was observed [13].

Preventing particle sedimentation in coal water slurries generally requires high energy input (e.g. permanent stirring of storage tanks) and accordingly significant operating costs. Turian et al. investigated the sedimentation of coal particles in water regarding particle size and sphericity. They report a delayed settling rate for smaller and non-spherical particles [14]. Particle settling can be reduced increasing the continuous phase viscosity e.g. using polymeric thickeners (e.g. CMC, rhamsan gum or polymethacrylate) [15,16]. Low molecular weight amphiphilic additives can be used to control the flocculation and network structure of coal water slurries. Tudor et al. discuss how the settling rate, as well as concentration profile and compression strength of the sediment can be controlled changing the coverage of the particle surface with non-ionic surfactant and upon addition of non-adsorbing polymer to the continuous phase [17].

The addition of an immiscible secondary fluid can have a strong impact on the flow behavior of suspensions. Under certain conditions adding the secondary fluid can result in a viscosity reduction [18,19]. In this work a novel stabilization concept for coke water slurries based on the capillary suspension phenomenon [1] is presented. Adding a small amount (~1–3 vol.%) of a second immiscible fluid to a suspension results in a sample-spanning and stable particle network controlled by capillary forces. This results in a drastic increase in low shear viscosity and pronounced shear thinning. Two different types of capillary suspensions are observed depending on the three-phase wetting angle $\theta_{(S,B)}$ the secondary fluid forms against the particle surface surrounded by the primary or bulk fluid.

In the pendular state the secondary fluid preferentially wets the particles ($\theta_{(S,B)} < 90^\circ$) and forms pendular bridges connecting the particles finally resulting in a percolating network. In the so-called capillary state ($\theta_{(S,B)} > 90^\circ$) clusters of particles are formed around small droplets of secondary fluid and these clusters are the building blocks of the sample-spanning network [20]. According to this structure formation capillary suspensions are highly resistant to sedimentation and exhibit unique flow properties. Their flow behavior can be easily adjusted to the demands of different manufacturing or application processes in a wide range [21] selecting the appropriate type and amount of secondary fluid.

The capillary suspension concept has been established as a generic formulation platform for a broad range of innovative materials including novel food formulations [1], capillary suspension based foams [22,23] or pastes for printed electronics providing unique shape accuracy and surface uniformity [24]. Capillary suspensions are even used as precursors for highly porous, high mechanical strength membranes [25,26]. The concept has also been used to control structure formation in particle-laden polymer blends [27]

or to assemble metals and nanoparticles into novel nanocomposites superstructures [28].

Here we describe the application of the capillary suspension concept to coke water slurries and we thoroughly discuss its impact on flow behavior, sedimentation stability, i.e. storage and transport, as well as atomization in entrained flow gasification.

2. Materials and methods

2.1. Materials

Coke particles from beech wood and wheat straw were used in this study. Equivalent sphere particle size distribution was determined by Fraunhofer diffraction (HELOS H0309, sympatec GmbH, Clausthal-Zellerfeld, Germany) using an ultrasonic wet dispersing unit (QUIXEL, sympatec GmbH) for dispersing the particles in deionized water. Density was determined using a pycnometer after Gay-Lussac (Carl-Roth GmbH, Karlsruhe, Germany). Porosity was measured with mercury intrusion porosimetry (AutoPore IV, micromeritics, Norcross, USA). Furthermore, ash content, carbon content and higher heating value have been determined according to DIN 51719, DIN 51732 and DIN 51900, respectively. Corresponding results are summarized in Table 1, equivalent sphere particle size distributions and respective scanning electron microscopy (SEM) images are shown in Fig. 1.

The SEM images indicate a rough but fairly isometric particle shape with visible pores. The coke was stored under N_2 atmosphere in barrels to minimize surface oxidation. Deionized water was used as the bulk phase for the slurries and acted as a model fluid for the aqueous pyrolysis condensate. Octanol-1 (Alfa Aesar, Karlsruhe, Germany) with a density of 0.83 g cm^{-3} and a dynamic viscosity of 9 mPa s was used as secondary phase for the formation of capillary suspensions. Both coke sorts are highly hydrophobic. The three-phase contact angle for beech wood coke in water with added octanol is $\theta_{(S,B)} = 67 \pm 6^\circ$ and for the wheat straw coke with the same conditions it is $\theta_{(S,B)} = 64 \pm 12^\circ$, i.e. both coke types have similar wetting behavior and both systems are supposed to form pendular state capillary suspensions when octanol is added to the aqueous coal slurry.

2.2. Sample preparation

The coke particles were slowly mixed into the bulk fluid using a turbulent beater blade until a homogeneous, air bubble free slurry was achieved. An increase of the angular mixing speed from 400 to 1000 rpm during an additional stirring period of 20 min was performed after all particles had been added to the sample to break remaining agglomerates. Then the secondary fluid was added to the suspension and thoroughly mixed again using a dispensing stirrer at $\sim 1000 \text{ rpm}$ for 10 min. This procedure worked for the lab scale samples with a volume of ca. 50 ml and also for the bigger volume of 40 l needed for the atomization experiments. All rheo-

Table 1
Characteristic physico-chemical specifications of investigated coal particles.

	Wheat straw coke	Beech wood coke
Volumetric mean equivalent sphere diameter	20 μm	12 μm
Particle density	1.85 g cm^{-3}	1.53 g cm^{-3}
Particle porosity	73%	57%
Ash content (DIN 51719)	29.2%	4.5%
Carbon content (DIN 51732)	49.5%	78.5%
Higher heating value (DIN 51900)	20 MJ/kg	30 MJ/kg

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