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Study of material agglomeration during nozzle injection of multiviscosity liquid into a fluidized bed reactor by the electric conductance method

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ABSTRACT

Fluidized bed agglomeration is an important and challenging problem for thermal cracking in fluid cokers. A low coker temperature can be problematic because the bitumen is injected into the fluidized bed with a different viscosity, resulting in formation of agglomerates of varying sizes, which slows the cracking reactions. In the present study, the bed material agglomeration process during nozzle injection of multiviscosity liquid was investigated in a fluidized bed operated at different mass ratios of the atomization gas to the liquid jets (GLR = 1%-3.5%) and gas velocities ($3.9U_{mf}$ and $5.9U_{mf}$) based on a conductance method using a water–sand system to simulate the hot bitumen–coke system at room temperature. During the tests of liquid-jet dispersion throughout the bed, different agglomeration stages are observed at both gas velocities. The critical amount of *tert*-butanol in the liquid jets that could lead to severe agglomeration of the bed materials (poor fluidization) at GLR = 1% is about 10 wt% at the low fluidizing gas velocity ($3.9U_{mf}$) and 18 wt% at the high gas velocity ($5.9U_{mf}$). This study provides a new approach for on–line monitoring of bed agglomeration during liquid injection to guarantee perfect contact between the atomized liquid and the bed particles.

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Introduction

In the fluid coking process, which operates at about 500 °C, bitumen is spray atomized with steam and injected into a fluidized bed of hot coke particles (Kondoh, Tanaka, Nakasaka, Tago, & Masuda, 2016). Although the presence of liquid will generally have an adverse effect on fluidization, because it might increase the cohesivity of particles and defluidize part or the entire bed, there are often strong incentives to operate with high liquid loading. In fluid cokers, the heavy feedstock is injected onto hot coke particles and undergoes thermal cracking to give lighter hydrocarbons and solid coke. Ideally, the injected liquid should contact the largest possible amount of fluidized particles by forming a thin liquid film on the surface of each particle (Hussain, Kumar, & Tsotsas, 2015; Zhao, O'Rourke, & Snider, 2009). Despite the rapid and intense solid mixing achieved in commercial fluid cokers, the distribution of injected bitumen on the fluidized coke particles

* Corresponding author. Fax: +86 0432 64807281. E-mail address: yangning@nedu.edu.cn (N. Yang). is relatively poor (Knapper, Gray, Chan, & Mikula, 2003; Gómez-Hernández, Soria-Verdugo, Briongos, & Santana, 2012). Therefore, identifying the operating conditions that enhance the liquid distribution over individual free-flowing solid particles is essential to minimize agglomerate formation.

Several techniques have been used to investigate the contact between the liquid jets and the solid in the fluidized bed. Pore et al. (2012) used magnetic resonance imaging to produce maps of the particle velocities around jets to investigate particle movement between the jets and to detect any dead zones in the fluidized bed. Kolkman, van Sint Annaland, and Kuipers (2016) developed a noninvasive experimental technique and obtained whole-field data of liquid-solid mixing in a gas-fluidized bed with liquid injection. Berruti, Dawe, and Briens (2009) mapped the boundaries of gas-liquid jets to characterize the jet angle, jet penetration, and the overall jet cavity during liquid-jet injection into a gas-solid fluidized bed using a thermal tracer method. Li et al. (2016) used thermal conductivity detectors to measure the local concentrations of a gaseous species as it evolves from a continuously sprayed liguid into the fluidized bed to investigate the mass transfer between the gas and the solid. Leach, Portoghese, Briens, and Berruti (2008)

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described a passive conductance technique where the conductance of the bed was measured from triboelectrification of the particles. The results indicated that as the liquid spreads through the bed, Portoghese, Berruti, and Briens (2008) refined the technique using triboelectric probes to monitor the changes in the wetted surface area of the fluidized bed produced by liquid jets by applying a sinusoidal current to the fluidized bed and measuring the real-time conductance across the fluidized bed. This technique is adopted in the current work.

Operating data from Syncrude fluid cokers have shown that reducing the coker temperature has two major benefits (Castañed, Muñoz, & Ancheyt, 2012): the yields of the valuable liquids increase and sulfur oxide emissions decrease, because sulfur is concentrated in more refractory coke fractions that will no longer be combusted. However, feed injection is the main stage that determines the reaction rate, and a major drawback is that a lower temperature reduces the reaction rate and unconverted feed can thus remain on the coke surface, making the particles sticky. Moreover, the unconverted feed exhibits different viscosity in the process of temperature reduction, which results in formation of agglomerates of varying sizes that hinder heat transfer to the reacting liquid, slowing the cracking reactions and causing fouling of the coker internals. However, the change in the liquid viscosity during decrease of the temperature has not been taken into account in previous studies. Almost all studies have assumed that the viscosity of the feed injection is constant. This cannot provide an effective reference for practical operation.

The objective of this study is to investigate and determine the effect of the liquid viscosity on material agglomeration in a gas-solid fluidized bed during nozzle injecting liquid using a capacitance method by changing the mass ratio of the atomization gas to the liquid jets (GLR = 1%-3.5%) and the fluidization velocity (3.9U_{mf} and 5.9U_{mf}). According to Aminu, Elliott, McCaffrey, and Gray (2004), the initial viscosity of the bitumen used in commercial fluid cokers is between 1 and 2 mPas when the temperature varies from 300 to 530 °C. Liquids with different amounts of tertbutanol are used to match the different viscosities of bitumen at the temperatures used in industry. The results of this research will aid in development of new strategies to prevent bed agglomeration in fluid cokers.

Experimental

Apparatus

The experiments were performed in a 2.10 m high fluidized bed with a $0.288 \,\text{m} \times 1.54 \,\text{m}$ rectangular cross-sectional area, as shown in Fig. 1. To uniformly fluidize the bed, a porous plate distributor was used. The fluidization air flow rate was controlled and measured with a calibrated 1.4 mm sonic orifice. The pressure transducers were positioned on the side wall at heights of 0.15 m above the gas distributor. The thermocouple at the top of the bed helped to maintain the bed temperature at 22 °C at the start of each injection, and the thermocouples were penetrated 2 cm into the equipment to avoid significant heat loss from stem. The liquid jets were atomized into the bed using a nozzle about 0.4 m above the distributor. Two types of spray nozzles were used in this study, as shown in Fig. 2. The first type of nozzle used in the calibration experiment was a "high GLR nozzle" (Fig. 2(a)) consisting of a hollow cylinder and operated with a GLR of 36%. Using such a high GLR is impractical from an industrial viewpoint because of high costs and flow restrictions. In other experiments, we used the second type of nozzle, which is called TEB (named after the inventor of this

Table 1

Binary solutions of *tert*-butanol in water and their properties.

Liquid concentration (wt%)	Viscosity (mPa s)	Contact angle (°)	Surface tension (mN/m)
18	2.0	64	24.7
10	1.39	67	42
0	1.2	75	68.0

patented nozzle, T.E. Base), as shown in Fig. 2(b). It is a scaled down version of the spray nozzles used in the coking process with a realistic GLR (Base, Chan, Kennett, & Emberley, 1999), which typically ranges from 1% to 3.5%. Air as an atomization gas was mixed with water in a premixer before reaching the nozzle to atomize the liquid into small droplets. The liquid flow rate for these experiments was about 10 g/s.

To measure the local bed conductance, three electrodes were installed at different heights on one side wall of the bed, as shown in Fig. 1. The electrodes consisted of a stainless-steel hollow tube with an outer diameter of 7 mm soldered at both extremities. A common electrode was arranged on the opposite wall. The electrical circuit is shown in Fig. 3. A signal generator connected to the electrodes provided a sinusoidal 100 Hz current with a constant voltage (V_S) of 7.74 V. Along the electrical circuit, a 100 k Ω resistor (R) was inserted in series. The intensity of the current flowing through the circuit can be obtained by measuring the voltage drop across this resistor (V_R) :

$$I = V_{\rm S}/(R + R_{\rm bed}) = V_{\rm R}/R.$$
(1)

Eq. (1) can be rearranged as

$$R_{\rm bed} = R(V_{\rm S}/V_{\rm R} - 1).$$
 (2)

The bed conductance can be obtained from the inverse of Eq. (2):

$$C_{\text{bed}} = 1/R_{\text{bed}}.\tag{3}$$

The instantaneous bed conductance can then be measured from the two voltages $V_{\rm S}$ and $V_{\rm R}$. The voltages and transducer signals were recorded with a data acquisition device (Model 34901A, Agilent, USA), which was equipped with self-programmed software.

The bed material used in this study was Barco 71 silica sand (99.88% SiO₂) (Opta Minerals Inc., USA) with a Sauter mean diameter of 204 μ m and density of 2650 kg/m³. The U_{mf} of the material was 0.072 m/s. In each experiment, 100 kg of dry sand was placed inside the bed through the top access window shown in Fig. 1 and then the access window was sealed. McDougall, Saberian, Briens, Berruti, and Chan (2004) showed that the water-sand system is a good simulator of the hot bitumen-coke system at room temperature because there is nearly perfect wettability of the solids by the liquids in both cases. To investigate material agglomeration during nozzle injection of the multiviscosity liquid into the fluidized bed, tert-butanol in water solutions with different concentrations were prepared, as shown in Table 1. The properties of these mixtures were experimentally determined using methods described in the literature (Mohagheghi, Hamidi, Briens, Berruti, & McMillan, 2014). Liquids containing 0, 10, or 18 wt% tert-butanol with viscosities of 1.2 to 2.0 mPas at room temperature were used to match the viscosities of bitumen at the injection temperature used in industry. The *tert*-butanol would act as a binder and maintain the integrity of the granule aggregates formed during the initial contact of the liquid jets with the sand particles. Therefore, the system at room temperature behaves like in the industrial conditions.

Samples of the bed materials were collected from three sampling points on the side wall at heights of 0.2, 0.8, and 1.3 m above the gas distributor. Approximately 30 g samples were scooped out of the bed at regular intervals (every 30s), and the total sam-

a larger wetted area of the bed leads to higher electrical conductance of the bed. Portoghese, Berruti, Briens, and Chan (2007) and

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