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Hydrodynamic behavior of silicon particles with a wide size distribution in a draft tube spout-fluid bed

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HIGHLIGHTS

- Effects of operation parameters on.ΔP_m, u_{ms} and W_s in DTSFB were examined systematically.
- Silicon particles with a wide size distribution were used as bed materials.
- A novel sampler was designed for measuring solids circulation rate.
- A correlation was put forward to predict the maximum spouting pressure drop.
- A correlation was put forward to predict the minimum stable spouting velocity.

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ABSTRACT

An experimental study on the hydrodynamic behavior of silicon particles with a wide size distribution in a draft tube spout-fluid bed was performed in a 182 diameter cylindrical column with a flat gas distributor. Effects of spouting and fluidizing gas velocity, static bed height, entrainment zone height and draft tube diameter on the maximum spouting pressure drop, minimum stable spouting velocity and solids circulation rate were investigated. The results show that the maximum spouting pressure drop increases with static bed height, entrainment zone height, draft tube diameter and fluidizing gas velocity. The minimum spouting velocity increases with entrainment zone height, while it decreases with static bed height, draft tube diameter and fluidizing gas velocity. Two correlations on the maximum pressure drop and minimum spouting velocity are proposed respectively based on the experimental data, which are in good agreement with the present experiments. In addition, the solids circulation rate increases with static bed height, draft tube diameter, fluidizing and spouting gas velocity. It also increases with entrainment zone height at a lower fluidizing gas velocity, however it decreases with entrainment zone height at a higher fluidizing gas velocity.

1. Introduction

With low energy consumption and high conversion rate, hydrogenation reduction of silicon tetrachloride [1,2] has been proved to be an effective technology to address the main issue (i.e., extensive generation of silicon tetrachloride as a byproduct) of the Siemens Process, which is currently the main industrial production technology of ultra-pure polysilicon accounting for 75% of the total production [3–6]. In the hydrogenation reduction process, a gas-solid reaction is involved as follows:

$$3SiCl_4(g) + Si(s) + 2H_2(g) \rightarrow 4SiHCl_3(g) \quad (1)$$

The process has been mostly carried out in conventional fluidized bed [7–9] using sub-millimeter grade silicon particles with a wide size distribution (10–1400) as raw materials. However, two significant limiting problems exist [10]: insufficient gas-particle contact due to particles agglomeration and stratification; pipe blockage caused by particles entrainment from the reactor. By contrast, the draft tube spout-fluid bed (DTSFB) combines the favorable properties of both spouted and fluidized beds. It provides a better gas-particle mixing and contacting process by introducing fluidizing gas and draft tube. The introduction of a fluidizing gas into the annular region increases the contact area of gas-particles and makes it possible to eliminate the dead zone in the bottom of the bed. The insertion of the draft tube reduces the interaction of particle flow between spout region and annular region, enabling the formation of a good inner particle circulation. It has also been reported that particles drain from the reactor can be reduced in a

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Nomenclature

D_c	bed diameter (m)	u_ms	minimum spouting velocity (m/s)
D_i	spout nozzle diameter (m)	W_s	solids circulation rate (g/s)
D_D	draft tube diameter (m)	m_{samp}	sampling mass (g)
L_D	length of draft tube (m)	t_samp	sampling time (s)
H_t	entrainment zone height (m)	ΔP_m	maximum pressure drop (Pa)
H_0	static bed height (m)	ΔP_s	pressure drop of spout region (Pa)
d_p	average diameter of silicon particle (m)	σ_s	standard deviation in the spout region (Pa)
A_A	cross sectional area of annular region (m^2)	ρ_b	bulk density of silicon particles (kg/m^3)
A_S	cross sectional area of sampler (m^2)	ρ_ρ	real density of silicon particles (kg/m^3)
u_f	superficial velocity of fluidizing gas (m/s)	ϵ	bulk voidage of silicon particles
u_mf	minimum fluidizing velocity (m/s)		
u_s	superficial velocity of spouting gas (m/s)		

DTSFB due to lower gas flow [11]. In addition, there are several other potential advantages of DTSFB [12–14]: greater operating flexibility, lower pressure drop, lower requirement of particle size, and better control of solid circulation. In this work, a DTSFB was proposed as a novel reactor for the hydrogenation of silicon tetrachloride with the aim to improve the flexibility and efficiency of the operation. A schematic diagram of the system is shown in Fig. 1(a).

In recent years, a number of experimental and theoretical studies have been carried out to investigate the hydrodynamic characteristics of spouted bed [eg: [15–19]] and spout-fluid bed [eg: [20–27]] with or without a draft tube. San José et al. [28] studied the bed stability of conical spouted bed with a draft tube and compared the results with those obtained without internal devices. Link et al. [29] combined experimental and simulation data and mapped the spout-fluid flow regimes in spout-fluid bed. Zhong et al. [30] studied the maximum pressure drop and minimum spout-fluidizing velocity in a spout-fluid bed using six kinds of Geldart D particles. Xu et al. [31] researched the minimum spouting velocity in a cylindrical DTSFB and a correlation of the minimum spouting velocity was proposed based on the experimental data. Berruti et al. [32] and Muir et al. [33] investigated the solids circulation rate in DTSFB with a semi-cylindrical unit and concluded that the solids circulation rate was strongly affected by the operating conditions and the geometric configuration of the reactor.

These earlier works were mainly focused on single particle size or narrow particle size distribution, and their distributor mostly were conical. The results of Luo et al. [34] showed that silicon particles with wide size distribution (belonging to Geldart B) behaved as Geldart A particles in a fluidized bed. Gauthier et al. [35] studied the influence of particle size distribution of powders on the minimum fluidization velocity and found that the addition of finer particles will greatly affect the hydrodynamic behavior of gas-solid fluidized beds. What is the character of DTSFB with a flat bottom when using silicon particles with wide size distribution as bed materials, similar to those using particles with single particle size or narrow particle size distribution? Reports on the above question are scarce so far.Su et al. [5] established a flow regime map in

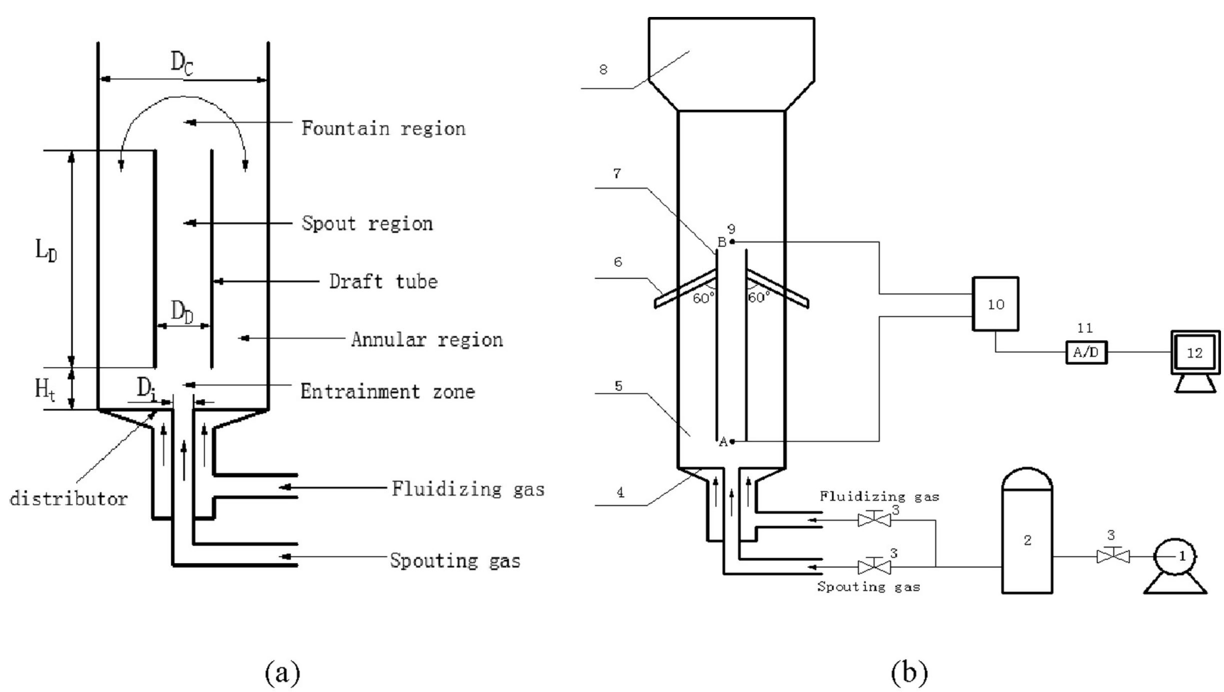

Fig. 1. (a) Schematic diagram of a DTSFB; (b) Schematic diagram of the experimental system. 1: air blower; 2: buffer tank; 3: control valve; 4: gas distributor; 5: spout-fluid bed; 6: sampler; 7: draft tube; 8: expanding section; 9: pressure port; 10: capacitive differential pressure transducer; 11: A/D converter; 12: computer.