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Vacuum powder feeding and dispersion analysis for a solar thermochemical drop-tube reactor

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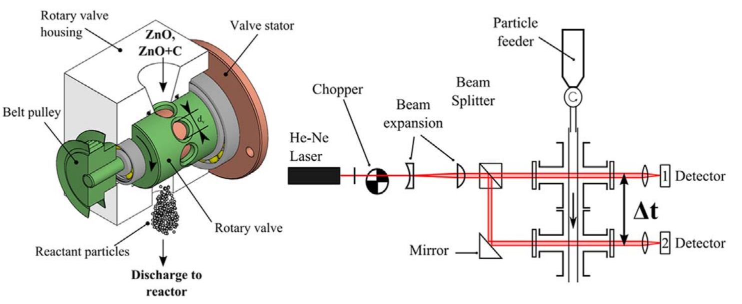
HIGHLIGHTS

- Particle feeding at low pressure with rotary valve and ultrasonic vibratory feeders.
- Particle residence time, axial and radial dispersion have been assessed.
- Residence time at 1 mbar is reduced by 70% compared to ambient pressure.
- Axial and radial particle dispersion under vacuum is reduced by 87% and 67%.

GRAPHICAL ABSTRACT



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ABSTRACT

Ultrasonic vibratory and rotary valve particle feeders have been designed, constructed, and investigated for application to feeding reactant powder to a solar thermochemical drop-tube reactor. Zinc oxide and carbon particles are fed continuously to the drop-tube under vacuum pressures as low as 1 mbar. The particles are probed in situ by laser transmission measurements with the aim to characterize particle residence time, axial and radial dispersion as a function of operating pressure. The ultrasonic feeder disperses particles well and can be operated at mass flow rates in the range of 57–288 mg min^{-1}. The rotary valve feeder operates in the mass flow range of 3.46–41.96 g min^{-1} and exhibits reduced particle dispersion due to discrete pulsing mass flow created from the rotating valve. The time resolved transmission signals reflect characteristic changes under different experimental vacuum conditions. Particles traveling through the measurement zone at 1 mbar exhibit residence and clearance times of 0.05 s and 0.52 s, respectively. At 960 mbar, residence and clearance times are increased to as much as 0.16 s and 3.98 s, respectively. Particles falling at 1 mbar show radial dispersion three times less than those falling under ambient pressure. A critical result of the functional characterization of powder feeding under vacuum is a potential reaction capacity limitation at low vacuum pressures due to short particle residence time and narrow axial dispersion.

1. Introduction

Renewable fuel production harnessing the power of the sun is a visionary pathway to substitute fossil fuels in the transportation sector while cutting CO_2 emissions and preserving the environment. High temperature thermochemical cycles, driven by concentrated solar power, offer the potential for renewable hydrogen and synthetic fuel production (Herron et al., 2015; Steinfeld, 2005). The Zn/ZnO redox pair has been identified as a promising candidate to complete the thermochemical cycle in two reaction steps (Bilgen et al., 1977; Loutzenhiser and Steinfeld, 2011; Palumbo

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Nomenclature

A	Peak area [dimensionless]
d_p	Particle diameter [μm]
d_v	Diameter of metering valve [mm]
$d_{10,50,90}$	Characteristic particle diameter on cumulative distribution
δ	Background attenuation [dimensionless]
h	Peak height [dimensionless]
$I_{1,2}$	Signal of detector 1 or 2 [V]
I_b	Detector signal baseline [V]
m	Cumulative fed mass [g]
$\dot{m}$	Reactant feed rate [g min^{-1}]
p	System pressure [mbar]
T	Transmission of signal [dimensionless]
T_f	Transmission during feeding [dimensionless]
t	Experimental time [min]
τ_{10}	Particle residence time at 10% peak height [s]
τ_{50}	Particle residence time at 50% peak height [s]
$t_{c,10}$	Clearance time at 10% peak height [s]
$t_{c,50}$	Clearance time at 50% peak height [s]
Δt	Time lag between detector signals [s]
w	Width of particle feed pulse [mm]
ω_S	Ultrasonic sieve stirrer rotational speed [min^{-1}]

Acronyms

DAQ	Data acquisition
DSLR	Digital single-lens reflex camera
IQR	Interquartile range, 2nd and 3rd quartiles around the median of a dataset
PSI	Paul Scherrer Institute
PSD	Particle size distribution
LE	Leading edge

et al., 1998). Zinc is produced by the thermal dissociation of ZnO, or with the addition of a reducing agent such as carbon. If carbon is used, it must be supplied by waste material or biomass to maintain sustainability (Osinga et al., 2004; Wieckert et al., 2007). Solar radiation, concentrated to several thousand times the intensity of normally incident sunlight, is used to deliver the necessary process heat for the endothermic reaction (Barlev et al., 2011). Recently, a solar drop-tube vacuum reactor was characterized for the carbothermal reduction of ZnO to metallic Zn (Brkic et al., 2016). The reactor concept can also be utilized for the production of other commodity metals such as Mg or Al (Brooks et al., 2006; Vishnevetsky and Epstein, 2015).

The high operating temperatures required for thermal ZnO reduction (>2300 K) impose stringent constraints for available reactor construction materials. In order to reduce the onset temperature of reaction it is proposed to carry out the reduction in vacuum (Chambon et al., 2010; Levêque and Abanades, 2015; Vishnevetsky and Epstein, 2015). Typical reactor operation for the solar reduction of metal oxides can be performed under direct irradiation (Chambon et al., 2011; Furler and Steinfeld, 2015; Koepf et al., 2012, 2015; Müller et al., 2006; Villasmil et al., 2013) and indirect irradiation (Levêque and Abanades, 2015; Osinga et al., 2004; Wieckert et al., 2007, 2004) of the reactants. Most of these reactors are operated in batch or semi-batch mode which requires frequent evacuation and purging during operation. Alternatively, a tubular reactor featuring vertical orientation and continuous reactant feeding offers several advantages (Steinfeld and Jovanovic, 2014; Takacs, 2013), such as scalable and modular design, low thermal inertia, robustness against thermal shock, and the ability to operate continuously at reduced pressure. Previously, rapid carbothermal reduction processes were reported in literature for a drop-tube arrangement (Weimer et al., 1993, 1991), where the tube itself is directly heated, resulting in indirect radiation heat transfer to the falling reactant particles. Ideally, the reactants are dispersed into a fine particle cloud and act as a volumetric absorber with quick heat up times and fast reaction kinetics (Johnson et al., 2002; Perkins et al., 2008).

Homogenous dispersion of untreated ZnO powder is difficult due to its cohesive flow properties (Koepf et al., 2014). The particles (<1 μm) tend to form tightly bound agglomerates as a consequence of van der Waals forces or due to the high surface tension of adsorbed water. Typical dispersion methods imply the use of shear forces provided by high velocity gas jets (Calvert et al., 2009), or the use of free-flow additives such as fumed silica. The agglomerates are entrained in the gas flow and are either broken down by shear forces, particle-particle collisions, or upon impact against the containment wall. If working under vacuum, the use of excessive gas flows is limited by the pumping capacity of the vacuum setup. In order to work efficiently, the pumping power must be reserved for product gas removal and not wasted on auxiliary gas flows. Standard powder feeding technologies at the laboratory scale are typically comprised of screw (Barati Dalenjan et al., 2012), vibratory tray (Tardos and Lu, 1996), and rotary valve feeders (Al-Din and Gunn, 1983; Francis et al., 2006; Koepf et al., 2014; Reed et al., 2000). For low particle feed rates, fluidized bed and brush feeders are reported to be effective (Francis et al., 2010; Lind et al., 2010; Woodruff et al., 2012), but are not suitable for vacuum due to the high gas flows required for particle dispersion. An alternative and less common method to feed fine powders is to use ultrasonic excitation and sieving (Hidaka and Miwa, 1979; Lu et al., 2006; Takano and Tomikawa, 1998).

On-line control of feeding behavior and knowledge of reactant residence time at low pressure is necessary for effective operation of a solar drop-tube reactor. It is common to use laser techniques to achieve online characterization of particulate flows (Lee Black et al., 1996). Dry laser diffraction (Ma et al., 2000) and particle imaging (Adrian, 1991) are common techniques used to obtain information on the in-situ particle size distribution of moving particle streams. Characterization of the generic particle concentration can be obtained by an optical laser transmission measurement, in which light is attenuated according to Beer's law (Raghunathan et al., 1993; Wang et al., 2010). Implementation of a laser transmission measurement setup with two beam lines also allows for measuring particle residence time, which is essential information to design and characterize a solar drop-tube reactor.

In this work, two different feeding concepts are investigated and characterized for application to a solar thermochemical drop-tube reactor that is operated at reduced pressure. The first feeding concept pursues the excitation of ZnO particles with ultrasound in a metallic sieve, with the aim to create a highly dispersed and steady flow of reactant powder. A piezoelectric transducer is brought to oscillation by a high frequency signal (>30 kHz). The vibrations are transmitted to a particle-containing sieve, thus generating a stream of aerosolized particles. The second feeding concept consists of a hopper with a conical outlet and a rotary valve to control the particle mass flow. The rotary valve feeder allows mass flow variation in a wider range at the expense of reduced particle dispersion and a characteristic pulsing mass flow.