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# Development of a new type of thermogravimetric analyser with a mini-tapered fluidized bed. Effect of fluidization of particles on the stability of the system

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#### Abstract

A new type of thermogravimetric analyser (tapered fluidized bed thermal gravimetric (TFBTG) analyser) has been developed, consisting of a balance, tapered fluidized bed and separate gas nozzle. The relations between the gas velocity and pressure drop and between the gas velocity and weight change were studied experimentally. The effect of the fluidization of the particles on the balance is discussed on the basis of a cold model experimental system. The following results were obtained: (1) there are five regions in the experimental system, i.e. (a) fixed bed, (b) fluidized core region with outward fixed bed, (c) fluidized core region with outward moving bed, (d) complete fluidized bed and (e) fluidized bed with emergent particles; (2) regions (c) and (d) can be used for thermal gravimetry, since the particles fluidize very well and both the pressure drop and weight change are almost constant in these regions. © 1997 Published by Elsevier Science S.A.

Keywords: Tapered fluidized bed; Thermal gravimetry; Fluidization behaviour

# 1. Introduction

The thermogravimetric analyser (TGA) is a useful tool of wide applicability in various fields, such as chemistry, physics, biochemistry, pharmacy, energy, environment, etc. Recently, a TGA/gas chromatography/mass spectrometry system has been developed for analytical purposes. However, the TGA can measure only small amounts of samples (20 mg), leading to problems in the analysis of the composition of the gaseous component released from a complex mixture such as coal, or refuse-derived fuel (RDF). Fig. 1 shows a schematic diagram of the commercial TGA, including the sample holder. If a large amount of sample is packed into the holder, an inhomogeneous temperature distribution may be generated in the bed when it is heated.

To solve this problem, a new type of TGA, called a tapered fluidized bed thermal gravimetric (TFBTG) analyser, has been developed in this study, which has good characteristics of heat and mass transfer in the bed.

So far, several mathematical models have been proposed to describe the relationship between the pressure drop and gas velocity in a tapered fluidized bed [1-3]. Toyohara and



Fig. 1. Schematic diagram of the commercial TGA.

Kawamura [4] studied fluidization characteristics, such as the pressure drop and particle movement behaviour, at various apex angles and operating conditions. Maruyama and Koyanagi [5] reported that the bed height and pressure drop of a tapered fluidized bed could be predicted by a model based on measurements in the fast bubble regime of slugging fluidized beds. Toyohara and Kawamura [6] carried out experiments to study particle circulation in the peripheral regions of a tapered fluidized bed, with a bottom (fluid inlet)

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cross-section of  $4 \times 4$  cm and apex angles of  $30^{\circ}$  and  $45^{\circ}$ , and indicated that the volumetric rate of circulation of the particles is proportional to the excess gas rate  $(u_i - u_b)$ . Recently, a map of the gas interstitial velocity vectors for conical spouted beds was proposed by Jose et al. [7] using the gas flow model.

However, the tapered fluidized bed used in the TFBTG system is different from the original tapered fluidized bed. The stability of the TFBTG system may be affected by the fluidization of particles shaking the balance. Therefore, to determine the suitable operating conditions for the TFBTG system, the relations between gas velocity and pressure drop and between gas velocity and weight change were studied experimentally using a cold model system. The effect of the fluidization of the particles on the balance was discussed using error bars for the weight change.

# 2. Experimental apparatus

Fig. 2 shows a schematic diagram of the new type of TGA. It consists of a balance, a mini-tapered fluidized bed and a gas nozzle. The balance is an electron balance (Chyo JL-180). The weight change was continuously recorded with a computer printer (YMC P-70). To observe the movement of the solid particles, the tapered bed was made of glass. The distributor set at the bottom of the tapered bed was a stainless steel screen of 400 mesh. The gas nozzle was located at a distance of about 0.5 mm from the bottom of the bed. The pressure drop in the bed was measured by a pressure tap located at the gas nozzle.

The fluidized particles used were silica sand, whose size distribution was measured by a Cilas-Granulometer (HR 850B) as shown in Fig. 3. The sample weight was maintained



Fig. 2. Schematic diagram of the experimental apparatus.



Fig. 3. Particle size distribution.

at about 1 g. The mean particle size was about 150  $\mu$ m. The density was 2650 kg m<sup>-3</sup>, the minimum fluidization velocity 0.019 m s<sup>-1</sup> and the terminal velocity 1.79 m s<sup>-1</sup>. The superficial gas velocity was based on the outlet gas velocity of the gas nozzle.

#### 3. Results and discussion

#### 3.1. Relationship between pressure drop and gas velocity

Fig. 4 shows the relationship between the pressure drop profile and the gas velocity. The squares and circles show the relationships for increasing and decreasing gas velocities respectively. When the gas velocity was increased, the pressure drop through the tapered bed increased. At a certain velocity, point B, the pressure drop reached a maximum value, and then sharply decreased to point B'. When the gas velocity was increased continuously from point B' to point D, the pressure drop eventually approached a constant value. On decreasing the gas velocity from point D to point H, the pressure drop remained almost constant, but with a further decrease in the gas velocity, the pressure drop began to decrease. The pressure drop for decreasing gas velocity remained constant over a wider region than that observed for increasing gas velocity.

#### 3.2. Relationship between weight change and gas velocity

Fig. 5 shows a typical relationship between the weight change and the gas velocity. When the gas velocity was



Fig. 4. Relationship between gas velocity and pressure drop.



increased, the bed was supported by the atmospheric buoyancy force, and so the weight decreased. At a certain value of the gas velocity, the weight decreased and reached a minimum with destruction of the static bed. As the gas velocity was increased from point B' to point C, the weight remained constant. However, a further increase in the gas velocity from point C to point D caused the weight to decrease again. On decreasing the gas velocity, after the weight had increased from point D to point E, it remained constant until point H, and then increased again. This constant region was wider than that observed for increasing gas velocity. The shape of the curve of gas velocity vs. weight change is different from the reverse curve of gas velocity vs. pressure drop shown in Fig. 4 only in the region between point C and point D (D and E). This difference is probably due to the fact that, when the gas velocity exceeds a certain value, particles emerge from the bed.

# 3.3. Change in the flow pattern as a function of gas velocity

Fig. 6 shows the flow patterns of the particles from visual observations. Five flow patterns were observed in the bed when the gas velocity was decreased from point D through points E, F, G and H to point A. Each region corresponds to that shown in Fig. 5. In the region from point D to point E, a state of fluidization was reached and particles began to emerge from the bed. As the gas velocity was decreased from point E to point F, this phenomenon disappeared. Between point F and point G, the fluidized core region with outward moving bed was formed; particles circulated between the core and the outward moving bed. Between point G and point H,





only the core zone was in a state of fluidization, with an outward fixed bed. When the gas velocity was decreased from point H to point A, the bed became static. Accordingly, it is concluded that the region between point E and point G, called the validity region, can be used for thermal gravimetry.

### 3.4. Fluctuation in weight in the validity region

To discuss the fluctuation in the weight caused by particle fluidization, error bars of the weight change were used in the validity region. Fig. 7 shows the weight fluctuation at various flow rates. It can be seen that the maximum fluctuation of the weight ranged from -0.005 to +0.007, i.e. -0.5% to +0.7%, based on the initial sample weight. The fluidization of particles in the bed shakes the balance very slightly in the validity region.

# 4. Conclusions

The following conclusions can be drawn from this experimental study: (1) there are five regions in the experimental system, i.e. (a) fixed bed, (b) fluidized core region with outward fixed bed, (c) fluidized core region with outward moving bed, (d) complete fluidized bed and (e) fluidized bed with emergent particles; (2) regions (c) and (d) can be used for thermal gravimetry, since the particles fluidize very well and both the pressure drop and weight change are almost constant in these regions.

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