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Original Research Paper

Bed collapse and expansion characteristics of multi-walled carbon nanotubes in fluidized beds

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ABSTRACT

The objective of this study was to investigate bed collapse and expansion characteristics of different types of multi-walled carbon nanotubes (MWCNTs) in fluidized bed with a 0.14 m-ID × 2.4 m-height Plexiglas column. Three types of MWCNTs were used as bed materials: (i) N, NC7000™ prepared by Nanocyl®, (ii) S_f, fine entangled MWCNTs agglomerated by strong cohesive force such as van der Waals force, (iii) S_c, coarse entangled MWCNTs with a single particle. Similarity between MWCNTs and Geldart group particles was investigated based on bed collapsing process. Results showed that bed collapsing processes of N, S_f, and S_c were similar to those of Geldart groups A, C, and B particles, respectively. Based on bed collapse and expansion characteristics, dense phase voidages of N and S_f were 0.795 and 0.921, respectively, in bubbling fluidization at superficial gas velocity of 0.19 m/s.

1. Introduction

Nano-materials have been synthesized in fluidized bed reactor recently [1–3]. Various techniques have been described [4–7]. Among them, bed collapsing method has been used to characterize particles and investigate void fraction in dense phase in bubbling fluidized bed [8–13]. The bed collapsing process generally involves three stages: bubble escape stage, hindered sedimentation stage, and solids consolidation stage. For Geldart group A particles, the bed collapsing process involves all three stages. For Geldart group B particles, this process only involves the bubble escape stage. For Geldart group C particles, the bed collapsing process involves both the short hindered sedimentation stage and the long solid consolidation stage [13]. Although results for the bed collapsing process of groups A and B particles are similar in most of the literature, there are some differences in the interpretation of bed collapse characteristics for group C particles. According to a previous study [10], gases can get out of the bed in the form of channeling with time because cavities can occur in the bed instead of bubbles during fluidization of group C particles. This process, similar to the solid consolidation stage for Geldart group A particles, proceeds at a slow rate [12]. According to previous literature [14], entangled MWCNTs considered as primary agglomerates have three-dimensional (3-D) network structures. Studies on bed collapse characteristics of MWCNTs are limited.

Therefore, the objective of this study was to investigate bed collapse characteristics and dense phase properties of MWCNTs under bubbling fluidization condition.

2. Experimental

2.1. Materials

MWNCTs used in this study had entangled structure formed during the growth of MWCNTs because MWCNT strands had nanoscale diameter and microscale length. These entangled MWCNTs were considered as primary agglomerates of MWCNTs. For fine primary agglomerate of MWCNTs, multi-agglomerate structure was formed with cohesive force in fluidized beds. In this study, three types of MWCNTs were used as bed materials: (i) N, NC7000™ prepared by Nanocyl® with morphology of curled up ball, (ii) S_f, agglomerates of fine entangled MWCNTs with irregular shapes such as elongated shapes and curled up ball, (iii) S_c, coarse entangled MWCNTs with a single particle. Scanning electron microscopy (SEM) was performed as described in a previous study [14]. Properties and minimum fluidizing velocity of MWCNTs are summarized in Table 1. Particle density was measured by mercury porosimeter while particle size was analyzed by sieving method. For S_f, size analysis was impossible because of its strong cohesive-

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Nomenclature

d_{sv}	Sauter mean diameter, µm
d_t	column diameter, m
f_{ic}	fraction of irregular cavities in the bubbling fluidized bed, –
H_0	static height of initial bed at the ambient condition, m
H_1	bed height in the bubble escape stage, m
H_2	bed height in the hindered sedimentation stage, m
H_3	bed height in the solid consolidation stage, m
H_b	bed height calculated by measuring pressure drop, m
H_c	bed height at critical point, m
H_e	bed height in dense phase, m
H_f	initial bed height for bed collapsing process, m
H_t	column height, m
H_∞	bed height at t = ∞, m
$-\Delta p_b$	pressure drop across the bed, Pa
t	elapsed time for bed collapsing process, s
t_b	time when all bubble have escaped, s
t_c	time at critical point, s
U_f	superficial gas velocity at fluidization, m/s
U_{mf}	minimum fluidizing velocity, m/s
U_{mb}	minimum bubbling velocity, m/s

Greek letters

ε_e	void fraction in the dense phase of a fluidized bed, –
ε_f	void fraction in a fluidized bed as a whole, –
ε_s	solid volume fraction, –
ρ_b	bulk density, kg/m^3
ρ_p	particle density, kg/m^3

Subscripts

0	initial
1	bubble escape stage
2	hindered sedimentation stage
3	solid consolidation stage

Table 1
Properties and minimum fluidizing velocity of different types of MWCNTs.

Bed material	N	S_f	S_c
Bulk density, ρ_b [kg/m^3]	53	20	81
Particle density, ρ_p [kg/m^3]	150	151	138
Sauter mean diameter, d_{sv} [µm]	242	N/A	1203
U_{mf} [m/s]	0.004	0.018	0.074

ness. More specific information on the minimum fluidizing velocity of MWCNTs can be obtained from a previous literature [14].

2.2. Experimental set-up

Schematic diagram of experimental apparatus is shown in Fig. 1. A Plexiglas column was used to investigate bed collapse and expansion characteristics of MWCNTs. This apparatus has been used in a previous study [14]. Inside diameter and total height of the column were 0.14 m and 2.4 m, respectively. An expanded column of 0.3 m was placed at the top of the column to reduce elutriation of particles. Elutriated particles were collected in a cyclone and returned to the column through the standpipe. A porous plate made by sintered metal was used as gas distributor. Fluidizing gas was introduced into the column using a mass flow controller (MFC). To measure pressure drop, ports were installed with an axial height.

2.3. Measurement techniques

Bed height was obtained by measuring pressure drop along the column with decreasing superficial gas velocity. A differential pressure transducer (Setra, model 264) was used. The reference point was 0.05 m above the distributor. Points were positioned at intervals of 0.05 m up to 0.55 m and 0.10 m above 0.55 m. After steady state, bed pressure drop was recorded at each point. Bed height and pressure drop across the bed in fluidized beds were determined from measured pressure drop with axial height. Bed voidage in fluidized beds was then calculated with the following equation.

$$-\Delta p_b = H_b(1-\varepsilon_f)(\rho_p-\rho_g)g \quad (1)$$

Bed collapse technique was used to study bed collapse characteristics and void fraction in the dense phase of MWCNTs. Before bed collapsing process, MWCNTs were fluidized at steady-state. Bed collapse was initiated by interrupting the gas supply. Variation of bed height was measured by image method.

3. Results and discussion

Typical photographs showing variation in bed during the bed collapsing process for N are presented in Fig. 2. As shown in Fig. 2a, the behavior of bed was bubbling fluidization at superficial gas velocity of 0.190 m/s. However, bubbles were not observed in this regime. For Geldart group A particles, bubbles formed near the distributor. They moved to bed surface during bubbling flu-

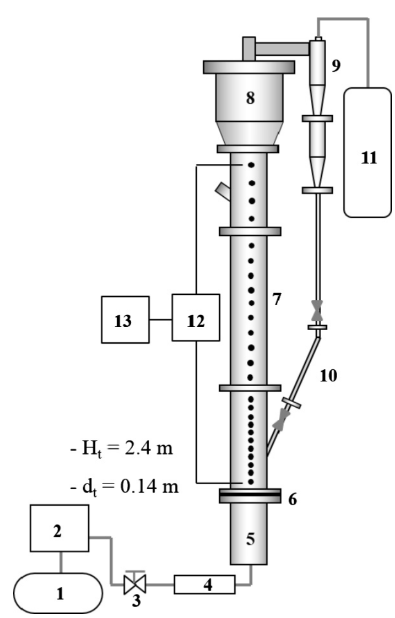

Fig. 1. Schematic diagram of experimental apparatus. 1, air compressor; 2, air dryer; 3, pressure regulator; 4, mass flow controller; 5, plenum chamber; 6, distributor; 7, column; 8, expansion column; 9, cyclone; 10, standpipe; 11, bag filter; 12, differential pressure transducer; 13, recorder.

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