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Elutriation of Carbon Black Powder from a Powder-Particle Fluidized Bed for the Process Gas Heat Recovery

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Energy saving has been recently conducted at the chemical industry by various kinds of technology to conquer Kyoto protocol COP3 as well as to reduce running cost of the production process. High temperature gas heat recovery leads to a big energy saving potential, which usually includes solids. Traditional shell and tube heat exchanger is not applied to the heat recovery because of fouling on the surface area of the tube. Heat recovery technology by the fluidized bed heat exchanger has been investigated for carbon black process gas as well as submerged type incinerator flue gas already presented at PRES'06 in Prague.

To explore the possibility of the process gas heat recovery in carbon black manufacturing process, the behavior of carbon black (CB) in the powder-particle fluidized bed (PPFB) was investigated. Alumina balls of 500 µm or 750 µm and silicon nitride balls of 500 µm were used as coarse particles. The bench-scale cold model experiments were carried out to investigate the final hold-up of CB in the bed and the amount of impurity in the elutriated carbon powder from the bed. The results clarified that covered layers of CB were hardly formed on the surface of 500-µm silicon nitride balls and 750-µm alumina balls. In such cases, the amount of impurity was hardly detected. Therefore, PPFB technology can basically be applied to the heat transfer equipment in a big energy-consuming carbon black manufacturing process.

1. Introduction

The carbon black (CB) production process consumes a tremendous amount of energy. Oil furnace process, which has been adopted for over 95% of CB manufacturing process worldwide (Gardiner et al., 1992), continuously sprays heavy aromatic tar oil into the reaction furnace having a high-temperature zone of over 1673 K. The tar oil particles are burned there, and are cooled down by water to inhibit the complete combustion. The incompletely burned tar oil particles are transformed into CB particles in the reaction furnace. Since the temperature is still high, the aerosols of CB are again cooled down by water to about 500 K to facilitate the separation of CB and gas by bag filters.

The CB suppliers have been struggling to recover energy loss in an effective way. If we realize further energy recovery at the upstream of the bag filters, we can convert it to steam generation with high temperature and pressure. Finned tube heat exchangers may suffer from a fouling problem leading to a reduction of heat exchanger performance (Herranz et al., 2002).

In this paper, we proposed a so-called Powder-Particle Fluidized Bed (PPFB) (Kato et al., 1994) for the heat recovery at the upstream of the bag filters. Installing fluidized bed technologies for the heat recovery can aim at not only decreasing fouling on the surface area of the tube but also expecting bigger heat transfer coefficient (Rasouli et al., 2005) than traditional shell and tube heat exchanger. Heat exchanger tubes and coarse particles in the PPFB provide the surface sites on which CB powder can adhere and deposit. The excess deposition of CB is avoided by the movement of coarse particles in the PPFB, making help CB entrain with the gas flow. This kind of heat exchanger should be operated under the steady state, where the feeding rate of CB balances the elutriation rate of CB. In case of considering the design of PPFB heat exchanger, it is important to evaluate the hold-up of fine particles in the bed. Li et al. (2001a) describes for the elutriation rate of fine powders (groups C or A particles) and their hold-up in the bed, which would not be applied to CB (extremely fine than groups C or A particles). Another important point on applying a PPFB to the CB process gas heat recovery is powder impurity due to contacting with coarse particles. As the first step we need to clarify the behavior of CB powder in the PPFB and quantify the amount of impurity in the entrained CB powder due to the possible attrition of coarse particles.

In the present work, the cold model experiments were carried out using a bench-scale PPFB to investigate the elutriation behavior of CB powder. The stable hold-up of CB in the bed were measured in the continuous operation. The amount of impurity in the collected CB powder was also quantified by chemical analysis.

2. Experimental

2.1 Materials

Alumina balls of 500 μm (HC type S) and 750 μm (HD-11, Nikkato Co.), and silicon nitride balls of 500 μm (SUN-11, Nikkato Co.) were used as coarse particles. Carbon black of 0.024 μm in primary particle size (#44, Mitsubishi Chemical Co.) was used as fine powder. Table 1 shows the properties of coarse particles (CP) and fine powder (FP).

2.2 Experimental setup and procedure

Fig. 1 shows the schematic diagram of a bench-scale cold model apparatus. The column of a PPFB is a transparent vinyl chloride column having 52 mm i.d. and 1.16 m height from a gas distributor. The top of the column was connected to a cylindrical bag filter through a flexible hose. Dry air was used as a fluidizing gas and an assisting gas for conveying CB powder that was dropped from a powder feeder (Microfeeder GMD-60, Hosokawa/Gerike) to the PPFB column at 0.10 m from the gas distributor.

Coarse particles or coarse particles mixed with 2.5wt% CB powder (Run 13) were packed in the PPFB column at static bed heights of 0.155-0.195 m. Before being packed in the column, the coarse particles were sometimes pretreated by water washing and/or blowing off at high superficial gas velocities to remove any dusts included in the coarse particles. Table 2 shows the pretreatment conditions.

Table 1. Properties of coarse particles (CP) and fine powder (FP).

	Coarse particles			Fine powder
Code	AL500	AL750	SN500	CB
Material	Al_2O_3	Al_2O_3	Si_3N_4	Carbon black
Type	HC type S	HD-11	SUN-12	#44
Particle size, d_{cp} *or* d_{fp} [μm]	500	750	500	0.024 (0.3-5)*
Particle density, ρ_p [kg/m^3]	3600	3600	3220	-
BET specific surface area, S [m^2/g]	-	-	-	110
Minimum Fluidization velocity, U_{mf} [m/s]	0.297	0.489	0.265	-
Terminal Velocity, U_t [m/s]	5.03	7.55	4.67	-
Chemical Composition [wt%]				
Al_2O_3	92	93	-	0.0013-0.0021
Si_3N_4	-	-	92	
SiO_2	7	5	-	0.0015-0.0021

* agglomerated particle size

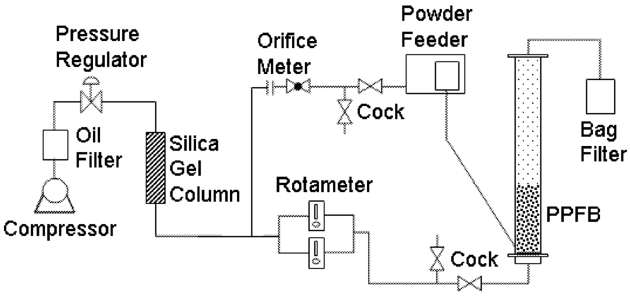


Fig. 1. Schematic diagram of experimental apparatus.

Table 2. Conditions of pretreatment of coarse particles and elutriation tests.

Run No.	CP (Pretreatment*)	Weight of CP, W_{cp} [kg]	Static bed height, L [m]	Gas velocity, U [m/s]	Initial FP hold-up, X_o [-]	FP feeding rate, F_{av} [g/min]
11	AL500 (N-1015t55)	0.8990	0.195	1.0	-	(1.4)
12	AL500 (6-1215t245)	0.7150	0.155	1.1	-	3.5
13	AL500 (N-C2.5)	0.7150	0.155	0.9	0.025	3.1
14	SN500 (N-N)	0.6322	0.155	0.9	-	4.2
15	AL750 (N-14t220)	0.7909	0.155	1.2	-	1.4-4.3
16	AL750 (N-R15)	0.7926	0.155	1.4	0.0021	2.0-3.6
17	AL750 (N-R16)	0.7912	0.155	1.0	0.0005	0.5-2.1

* N-1015t55: no water washing, blowing-off at 1.0-1.5 m/s for 55 min; 6-1215t245: 6 times water washing, blowing-off at 1.2-1.5 m/s for 245 min; N-C2.5: no water washing, 2.5wt% CB added; N-N: no water washing, no blowing-off; N-14t220: no water washing, blowing-off at 1.4 m/s for 220 min; N-R15: no water washing, direct use after Run 15; N-R16: no water washing, direct use after Run 16.

After the coarse particles were packed in PPFB, the cock to feed the air was quickly turned on to keep the desired superficial gas velocity. Immediately after that, the CB elutriation test was started by working the powder feeder. The elutriated CB powder was captured by a cylindrical bag filter. At a certain time interval (usually 2 min), the dry air supply, CB feeding and vibrations were simultaneously stopped. Total amount of elutriated CB was measured by weighing the bag filter. After that, the bag filter was installed again, and the next test was started by following the same procedure described above. Table 2 shows the experimental conditions of the elutriation test.

2.4 Analysis

The final hold-up of fine powder (CB) in the bed, $X_{i,\mathrm{fin}}$, is defined as

$$X_{i,\mathrm{fin}} = W_{\mathrm{fp}} / (W_{\mathrm{fp}} + W_{\mathrm{cp}}) = (W_{\mathrm{t}} - W_{\mathrm{cp}}) / W_{\mathrm{t}} \qquad (1)$$

where W_{fp} is the weight of fine powder in the bed, W_{cp} is the weight of coarse particles in the bed, and W_{t} is the total weight of bed particles after the final test in the run.

The impurity elements, Al and Si, were quantified by ICP-OES analysis for the residues after ignition of the CB samples.

3. Results and Discussion

3.1 Steady elutriation of CB

Fig. 2 shows the time scope of total amount of CB elutriated from the PPFB for Runs 11 to 17. Because of the strong cohesiveness of CB, the feeding of CB was sometimes not stable in the first half of the operation. However, after consolidating bulk CB powder in the hopper, the elutriation quantity of CB became stable for a necessary run time. The results in the last half of the operation suggested that the steady state was achieved. However, the elutriation rates of CB were much higher than the value calculated from the equations proposed by Li and Kato (2001a, 2001b), suggesting that CB powder entrained in the form of large agglomerates increased the gravitational force, as compared to the adhesion force.

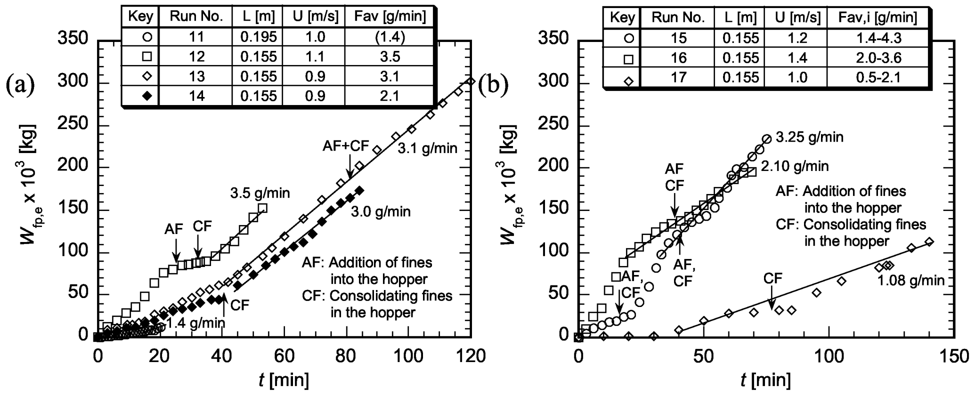


Fig. 2. Time scope of total amount of CB elutriated from the PPFB using (a) 500-μm coarse particles, (b) 750-μm coarse particles.

3.2 Final hold-up of CB in the bed

Fig. 3 shows the final hold-up of fine powder in the bed as a function of superficial gas velocity. The hold-up of fine powder decreased exponentially with the increase of the superficial gas velocity.

When the superficial gas velocity was lower than 1 m/s, larger coarse particles gave much smaller hold-up of fine powder in the bed than smaller coarse particles. This results agreed to the previous work by Nakazato et al. (2006), who had used 0.5-µm $Al(OH)_3$ as fine powder in the PPFB. However, the elutriation mechanism of CB would be different from that of sub-micron powders as 0.5-µm $Al(OH)_3$ since a covered layers by CB were hardly recognized on the surface of 750-µm alumina balls as well as 500-µm silicon nitride balls.

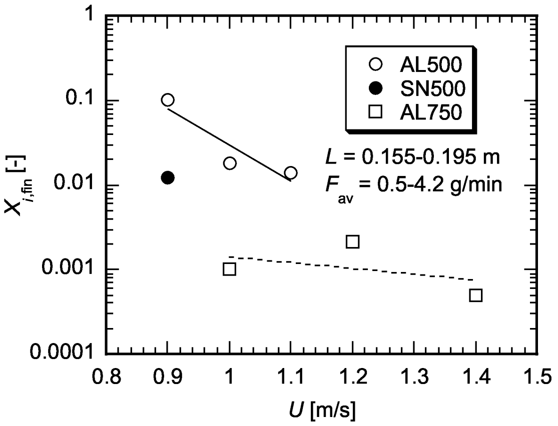
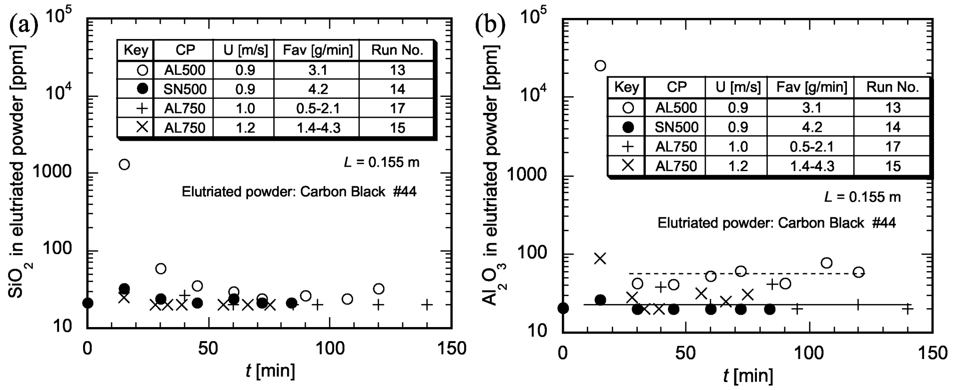


Fig. 3. Final hold-up of fine powder in the bed as a function of superficial gas velocity.



Key	CP	U [m/s]	Fav [g/min]	Run No.
○	AL500	0.9	3.1	13
●	SN500	0.9	4.2	14
+	AL750	1.0	0.5-2.1	17
×	AL750	1.2	1.4-4.3	15

Key	CP	U [m/s]	Fav [g/min]	Run No.
○	AL500	0.9	3.1	13
●	SN500	0.9	4.2	14
+	AL750	1.0	0.5-2.1	17
×	AL750	1.2	1.4-4.3	15

Fig. 4. Amount of impurity counted as (a) SiO_2 and (b) Al_2O_3 in elutriated powder at each time interval during continuous operation.

3.3 Amount of impurity in elutriated powder

Fig. 4 shows the amounts of impurity counted as SiO_2 and Al_2O_3 in the elutriated powder at arbitrary time intervals. Regardless of the pretreatment condition of coarse particles, large amount of impurity was shown at the initial stage of operation. This may be due to the contamination from outside of the apparatus before the start of the first test in the run. However, the amount of impurity soon became low and arrived at almost constant amount. At superficial gas velocity of 0.9 m/s, using AL500 gave higher amount of impurity, while SN500 gave no contamination during the run. On the other hand, using AL750 lowered the amount of impurity as compared to AL500 even at superficial gas velocities higher than 0.9 m/s. Lower U/U_{mf} was effective for lowering the amount of impurity, keeping the final hold-up of fine powder in the bed very low (Fig. 4). The intensity of fluidization, U/U_{mf}, can controls the elutriation behavior of CB.

4. Conclusions

As a first step to develop a PPFB heat exchanger for further heat recovery in the CB manufacturing process, cold model experiments were carried out to investigate the elutriation behaviour of CB and the amount of impurity in the elutriated powder. Use of large coarse particles in the PPFB was effective to reduce the impurity content in the elutriated CB powder, lowering the final hold-up of fine powder in the bed.

5. Acknowledgment

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