PERFORMANCE OF DRY SEPARATION PROCESSES IN CENOSPHERES RECOVERY FROM COAL FLY ASH

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ABSTRACT

Many researchers have recently been very interested in the recovery of valuable materials from fly ash. One of them is cenospheres, which are beneficial for light weighted material components due to their physical and chemical characteristics. Cenospheres recovery technologies are mostly based on wet separation process due to their density, which is lower than water density. Meanwhile the possibility of dry separation process has not yet been fully explored. In contrast to wet separation, a dry separation process provides substantial benefits. These benefits include: much less land consumption and reduced levels of pollution related to the leaching of toxic materials from coal fly ash.

In this study, experimental work has been conducted using two types of dry separation processes: a pneumatic separator closed type and a micron separator. Comparing the two alternative techniques, the micron separator obtained Newton’s efficiency higher than that of pneumatic separator closed type and showed a better degree of separation. Cenospheres recovery, particle distribution and visualisation of both underflow and overflow product using Scanning Electron Microscopy (SEM) were also performed to comprehensively depict the separation performance of both dry separation processes. It was found that micron separator’s Newton’s efficiency tendency is in line with the estimate, showing that dry separation process achieves a degree of separation as high as the commonly used technology which is wet separation process.

Keywords: cenospheres, coal fly ash type IV, pneumatic separator closed type, micron separator, Newton’s efficiency

INTRODUCTION

In recent times, cenospheres recovered from coal fly ash as one of the coal combustion residues (CCRs) has become a focus of interest due to their superior physical and chemical properties. These include light weight, enhanced insulation, less water absorption, excellent mechanical strength, chemical inertness, and good thermal resistance (< 1250-1300°C) (Barbieri et al, 1999). Cenospheres are formed from the combustion of coal in a molten state. Flowing from the combustion gas stream, the temperature of the molten particles is rapidly quenched, and freezes into a spherical shape. Any gases presented within the molten particles are trapped inside the spheres, usually in the form of bubbles. These bubbles cause the production of cenospheres. Bubbles may occur in multiple forms within the frozen particles, or as single, concentric form that is nearly as great as the diameter of the particle itself. The thickness of cenosphere wall may be very small – eg < 10 per cent of the particle diameter – and, if so, the resultant densities are typically less than 1 g/cm³ (Raask, 1968). Based on this definition, most of technologies designed to recover cenospheres from coal fly ash are related to their relative density using water as separating fluid (this is referred to as a wet separation process) (Hirajima et al, 2008).

Torey (1978) found that although cenospheres have similar composition to fly ashes, their particle size is generally bigger. Therefore, a reasonable hypothesis can be formed that recovery of cenospheres based on particle size difference may be able to be conducted. While this has not yet been fully explored, the application of a dry separation process in cenospheres recovery appears to have potential. There are other potential benefits related to the land availability and the reduction of toxic material released by coal fly ash during wet separation processes.

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In this paper, separation performance of pneumatic separator closed type and micron separator will be studied in order to recover cenospheres from coal fly ash.

**Theoretical background**

Newton’s efficiency is a parameter to determine whether a separation process provides higher degree of separation. The formulation of Newton’s efficiency is shown in (1) (Wills, 1997).

\[ \eta_N = R_c - (1 - R_{fa}) \]  

where:

- \( \eta_N \) = Newton’s efficiency
- \( R_c \) = Recovery of cenospheres in underflow product (wt %)
- \( R_{fa} \) = Recovery of coal fly ash type IV in overflow product (wt %)

Recovery of cenospheres and coal fly ash type IV are further formulated from material balance, based on cenospheres and coal fly ash type IV concentration, respectively in feed, overflow, and underflow product, as shown in (2) and (3) (Wills, 1997).

\[ R_c = \frac{c_f W_c}{c_j W_f} \left( \frac{c_j - c_m}{c_j - c_c} \right) c_c c_f \]  

\[ \eta_{fa} = \frac{W_{fa} \left( 1 - c_m \right)}{W_f \left( 1 - c_f \right)} \left( \frac{c_j - c_m}{c_j - c_c} \right) \left( \frac{c_{fa} - c_c}{1 - c_f} \right) \]  

where:

- \( W_f \) = weight of feed (kg)
- \( W_c \) = weight of cenospheres concentrated underflow product (kg)
- \( W_{fa} \) = weight of coal fly ash concentrated overflow product (kg)
- \( c_f \) = concentration of cenospheres in the feed (wt %)
- \( c_c \) = concentration of cenospheres in concentrated cenospheres underflow product (wt %)
- \( c_{fa} \) = concentration of cenospheres in coal fly ash concentrated overflow product (wt %)

**MATERIALS AND METHODS**

**Materials**

Industrial cenospheres and coal fly ash type IV, both supplied by the Idemitsu Co. Ltd., were chosen as model particles. For all the experiments, 20 wt per cent of industrial cenospheres were mixed with coal fly ash type IV as feed sample. In this study, samples were prepared using model particles in order to obtain more homogenous particles, so that the inconsistency of the separation influenced by particle diameter can be minimised. In addition, basic estimation separation has been conducted employing these model particles for comparison.

**Pneumatic separator closed type**

Figure 1 depicts the pneumatic separator closed type utilised in this experiment which was developed by Matsuoka Engineering Co Ltd. In this study, different yields (wt per cent) of overflow or underflow product were obtained by adjusting fan speeds in the range of 1.2 m/s - 3.7 m/s. Feed rate was maintained at 100 g/min. The yield of underflow and overflow product at each air flow rate were collected and analysed. Further float and sink testing was undertaken to determine the separation efficiency represented by Newton’s efficiency. Finally, a particle distribution of each overflow and underflow product was determined using a laser scattering particle size distribution analyser (LA-950).

**Micron separator**

The micron separator by Hosokawa Micron Co., was utilised to observe the possibility of dry separator on cenospheres recovery from coal fly ash. In addition, the study should verify the separation estimate. Figure 2 depicts the outline of the apparatus. Separation was varied by setting up the rotor speed in a range between 600 rpm to 2200 rpm. The feed carried by the main air flow was maintained in the feed rate range of 160 kg/h to 180 kg/h. Total weight of feed for all experiments was 17.5 kg with
20 wt per cent of industrial cenospheres in the feed. Similarly the pneumatic separator closed type, float-sink test and particle distribution analysis were conducted for each overflow and underflow product.

**Particle distribution**
The particle distribution of each overflow and underflow product was determined using a laser particle scattering particle size distribution analyser (LA-950). A one per cent slurry was made of each overflow and underflow product. To ensure the stability of each particle, disperse media was added into the solution. Sodium hexametaphosphate (NaPO₃)₆ was used with concentration of 1 g/L. A sufficient amount of surfactant was then added prior to five minutes of ultrasonic vibration before the particle distribution measurement.

**Scanning electron microscopy (SEM)**
SEM studies were carried out to analyse the existence of cenospheres in overflow or underflow product, as well as qualitative examination of the particle distribution. After the use of both pneumatic separator closed type and micron separator, some part of the overflow or underflow product were affixed onto SEM sample plate with carbon tape. Ensuring the homogenous sample is important and was obtained by shaking each overflow or underflow product well. In order to ensure a single...
layer of particles on the SEM sample plate as well as the cleanness of the plate, nitrogen gas was introduced for about one minute. Images were then acquired using a Keyence VE 9800 scanning electron microscope.

**Float and sink test**

A float and sink test was conducted to quantify the concentration of cenospheres in each overflow and underflow product. Suspension of 5 wt per cent using 1 l of water was used for float and sink test. Ultrasonic vibration was applied for 15 minutes to ensure the stability of single particle. The suspension was then poured into 2 l of graduated cylinder, and this was placed in a holder vertical pole and remained at ambient temperature for eight hours. Following this, float and tail products were collected, drained through a 0.2 μm filter, dried at 100°C and weighed. The float product is cenospheres, while the tail product is coal fly ash particles. Having this analytical data, separation efficiency represented by Newton’s efficiency was then calculated.

**RESULTS AND DISCUSSION**

**Yield of product**

Yield of overflow and underflow product in correlation with the operating condition of both pneumatic separator closed type and micron separator are shown in Figure 3. In pneumatic separator closed type, variable of the operating condition is the fan speed. Fan speed regulation produces different yields of underflow and overflow product. As depicted in Figure 3a, the higher air flow rate, the higher yield of overflow product yield, and vise versa in underflow product. This is due to the difference of terminal velocity of particles. The higher the air flow rate the more particles will be in the overflow product, because more terminal velocity of particles are exceeded. Therefore, it was found that the concentration of cenospheres in underflow product is higher than coal fly ash as shown in Figure 3c. However, this tendency was observed only below 2.7 m/s of air flow rate, a vise versa concentration of cenospheres was obtained over this air flow rate. It is proposed that the phenomenon occurred as

![Graph](image-url)
a result of re-concentration of fine particles from overflow to underflow product (further explanation see Particle distribution).

Similarly, micron separator performance is affected by rotor speed, the difference with pneumatic separator closed type is the mechanism of separation. In micron separator, the mechanism is affected by the centrifugal force created by the rotor. From Figure 3b, it can be seen that the higher the rotor speed, the higher the yield of underflow product. From float and sink testing, it was observed that cenospheres are concentrated in underflow product, as shown in Figure 3d. Although the mechanism is different, somehow the separation principal is influenced by the particle size, similar to the pneumatic separator closed type. Raask (1968) suggested that particle size of cenospheres are bigger than coal fly ash, and this appears to be supported by Torey (1978), admittedly with different sample of coal fly ash. Finding the concentration of cenospheres in underflow product in both pneumatic separator close type and micron separator, is understood as a result of different particle size resembled with the previous study on estimate separation using dry separation process (Hirajima et al, 2009).

**Particle distribution**

Particle distribution describes the change of particles concentration based on particle size. Figure 4 depicts the particle distribution of pneumatic separator closed type. Figure 4a and 4b show particle distribution of overflow and underflow product of pneumatic separator closed type. In the overflow

![Particle distribution of pneumatic separator closed type; (a) overflow (OF), (b) underflow (UF).](image-url)
product, shown in Figure 4a, it can be seen that the concentration of coarse particles with a particle diameter of 100 μm was increasing in response to an increase of overflow yield product. Gradually, coarse particles, which are mostly cenospheres, were brought into the overflow product once their terminal velocity is exceeded. As a consequence, the percentage weight of fine particles in overflow product was found to be the opposite. Initially, fine particles where coal fly ash concentrated is brought to the overflow product and accumulated until a remaining amount at the stage where the terminal velocity of coal fly ash particles exceeded, regardless to particles interaction during the separation.

In term of underflow product, depicted in Figure 4b, the highest yield rate of 99 per cent was obtained at the lowest air flow rate of 1.2 m/s. In this condition, the weight percentage of both fine and coarse particles was approximately the same. Once the air flow rate increased, more fine particles were transferred to the overflow product resulting in a decrease of fine particles weight percentage and an opposite tendency of coarse particles in the underflow product where the cenospheres are concentrated. However, over 45 per cent of underflow yield at 2.2 m/s of air flow rate, the opposite phenomenon was observed, the weight percentage of fine particles was increased with an increase of air flow rate. It is suggested that this might be due to the re-concentration of fine particles from

![Graph](https://via.placeholder.com/150)

**FIG 5** - Particle distribution of micron separator; (a) overflow (OF), (b) underflow (UF).
overflow to underflow product. As it can be seen from the schematic outline of the apparatus in Figure 1, there is a possibility of the particles in the overflow chamber to be recovered in the underflow product. This is a result of the improper isolation of overflow and underflow product chamber.

For micron separator, the particle distribution of overflow and underflow is shown in Figure 5a and 5b. It can be seen from Figure 5a, the tendency of course particles and fine particle concentrations in overflow product was similar to that of pneumatic separator closed type as a function of overflow product yield. The higher yield of overflow product the lower weight percentage of coal fly ash, while coarse particles was in the opposite correlation. In term of underflow product, as it can be seen in Figure 5b, the concentration of fine particles was found decreasing in lower underflow product yield, while coarse particles were more concentrated. A different phenomenon is compared with pneumatic separator close type where a shifting concentration was occurred as previously explained. These findings verify the estimate separation study based on the terminal velocity conception that the separation is affected by particle size than density in dry separation process. From the calculation, it was found that the cenospheres concentration is in the underflow product where coarse particles are concentrated (Hirajima et al., 2009).

**Newton’s efficiency (efficiency of separation)**

Newton’s efficiency is a parameter to determine the efficiency of a separation process. Figure 6 depicts the correlation of underflow product where cenospheres concentrated, and the Newton’s efficiency. In comparison, an estimate Newton’s efficiency of dry separation process is also depicted (Hirajima et al., 2009). The trend of Newton’s efficiency of both apparatus types was found similar to the trend of estimate of Newton’s efficiency. The Newton’s efficiency was increased, reached the optimum value, and then decreased gradually along with increase of underflow product yield.

In pneumatic separator closed type, optimum Newton’s efficiency of 0.26 was obtained at 45 wt per cent of underflow product. At underflow product lower than 25 wt per cent, it was observed that the Newton’s efficiency acquired a negative value. As explained in section Particle distribution, this negative value of Newton’s efficiency was a result of the re-concentration of coal fly ash (fine particles) in underflow product, since theoretically coal fly ash should be concentrated in overflow product.

Observing the process using the micron separator, the separation performance revealed that the micron separator had better performance than pneumatic separator closed type. As Figure 6 demonstrates that micron separator obtained overall Newton’s efficiency higher than that of pneumatic separator closed type and similar as the estimate Newton’s efficiency. It reached 0.44 of optimum Newton’s efficiency at 29 wt per cent of underflow product.

Further observations were then conducted by SEM image. Figure 7 shows the SEM images of overflow and underflow product of both apparatus at optimum Newton’s efficiency level. From
the images, it can be clearly observed that the micron separator has performed a better separation process. Semi-quantitatively, it was found that in overflow product, there were less cenospheres occurring in the micron separator than in pneumatic separator closed type.

CONCLUSIONS

Dry separation processes are interesting separation methods to recover cenospheres from coal fly ash. This has special importance given the existing sustainable development related issues that occur in the application of wet separation processes. Among these issues are land availability and the release of toxic material by coal fly ash during the separation process. In this study, the separation performance of pneumatic separator closed type and micron separator were compared, as well as compared with the separation estimate conducted in previous study (Hirajima et al., 2009). Both apparatus provided similar tendencies of cenosphere concentration in underflow product. This is due to the bigger particle size of cenospheres than coal fly ash (Raask, 1968; Torey, 1978). Theoretically, the separation mechanism in dry separation process for cenospheres recovery from coal fly ash is determined by the terminal velocity of particles that are mainly affected by the particle size.

Newton’s efficiency obtained from estimation and experiments showed similar trends. However, micron separation performed better than pneumatic separator closed type. As high as about 80 per cent of the estimate Newton’s efficiency has been reached which was 0.44. The lower separation performance of pneumatic separator close type was resulted by the re-concentration of coal fly ash particles in the underflow product, shown by shifting concentration of coal fly ash and cenospheres in underflow product and negative Newton’s efficiency. Regardless to the lower or even negative Newton’s efficiency obtained in pneumatic separator closed type, dry separation process, in this experiment was the micron separator, which provided high efficiency of separation for cenospheres recovery from coal fly ash. It can be applied for main equipment or intermediate equipment in order to eliminate the disadvantages of wet separation process as previously mentioned.

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