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Recovery and concentration of buoyant cenospheres using an Inverted Reflux Classifier

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Cenospheres are hollow, low-density particles found in power station fly ash. They have many commerciallyuseful properties which make them a valuable by-product. However, recovering cenospheres from fly ash is difficult due to their low concentration and fine size. Experiments were performed to test the novel approach of using an Inverted Reflux Classifier. In this configuration, the particles are fluidised by adding wash water from above which helps to wash any entrained dense material from the overhead product. Inclined channels are mounted at the base to minimise the loss of buoyant cenospheres in the waste underflow stream.

Experiments were performed at both laboratory scale (80 mm \times 100 mm cross-section) and pilot scale (300 mm \times 300 mm cross section) using mixtures of cenospheres and silica, all nominally less than 100 μ m in size. In batch tests, the bed expansion behaviour of the positively-buoyant cenospheres in the Inverted Reflux Classifier was found to be analogous to the behaviour of negatively-buoyant particles in the standard configuration. Continuous steady-state experiments were performed using feeds with suspension solids concentration varying from 0.3 to 9.5 wt.% solids and a buoyant cenosphere grade of 0.5 to 65 wt.%, with a range of fluidisation wash water rates, and degree of volume reduction (ratio of volumetric feed to product rate). Both units delivered high recoveries and product grades. An increase in volume reduction (decreasing overflow rate for a given feed rate), caused a drop in recovery and an improvement in grade. The throughput advantage compared to a conventional teetered (fluidised) bed separator was over 30 in some cases. Both laboratory and pilot-scale units displayed similar behaviour and the results were also consistent with existing correlations for negatively-buoyant particles in the standard Reflux Classifier. Hence this technology has clear potential for recovering and concentrating cenospheres from fly ash.

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1. Introduction

Cenospheres are hollow spherical alumina-silicate particles formed during coal combustion. The effective density of individual cenospheres can vary from that of silica (~2600 kg/m³) down to 200 kg/m³ or even lower, depending on the number and size of the gas inclusions in each particle [\[1\]](#page--1-0). However, in this work we use the term "cenosphere" to refer to only those particles with an effective density less than water i.e.

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specific gravity less than 1.0. Cenospheres are free flowing, strong, low in density and have low thermal and electrical conductivity. These properties make them a valuable by-product which is finding increasing use in a range of industries, such as aero-space, building and chemical, where they provide advantages such as low weight, increased heat resistance, better electrical insulation, low shrinkage, microwave shielding and reduced water absorption (e.g. [\[2](#page--1-0)–7]). Therefore, the recovery of cenospheres from power station fly ash is attracting growing interest, with various wet and dry processes having been trialled (e.g. [8-[12\]](#page--1-0)).

There are some major challenges in efficiently recovering cenospheres. Their surface, magnetic and electrical properties are similar to many of the other components found in fly ash. Thus methods such as flotation and electrostatic separation that are routinely used in mineral processing for separating fine particles, and have been successfully applied to remove combustible material from fly ash, cannot be effectively applied to recovering cenospheres. Gurupira et al. [\[10\]](#page--1-0) do report using a triboelectric separator to recover cenospheres from fly ash, but rely on a definition of cenosphere as particles of specific gravity less than 2.0, whereas most commercial interest is focussed on cenospheres with specific gravity less than 1.0. Hence, the only practical method available appears to be density-based separation. However, cenospheres are generally present at low concentrations, typically

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0.01 to 5 wt.%, and are small in size, typically in the range 50 to 250 μm diameter [\[13\]](#page--1-0). Gravity-based separation processes struggle to perform well down in this size range (e.g. [\[14\]](#page--1-0)).

It is in theory simple to separate positively buoyant cenospheres from the denser components of fly ash by float-sink separation in water. Indeed the usual approach to producing commercial grade product involves scooping the buoyant cenospheres from the surface of fly ash tailings ponds [\[9\].](#page--1-0) However, this requires large holding ponds, results in significant losses and the product includes a high level of contamination, so it needs further upgrade. Attempts to separate cenospheres via a conventional elutriation device inevitably also entrain a significant amount of high density silica slimes in the overhead product.

The objective of the present study is to evaluate the potential for an Inverted Reflux Classifier (IRC) to efficiently separate and recover cenospheres from fly ash. The standard Reflux Classifier (RC) is a separation device that consists of a fluidised bed with a set of parallel inclined channels mounted above (Fig. 1a). The inclined channels provide a much larger effective settling area than a standard teetered (fluidised) bed separator due to the Boycott effect [\[15\].](#page--1-0) The selective re-suspension of low-density particles that settle on the channel walls suppresses the effects of particle size on the separation, thus making the separation more sensitive to density difference [\[16\].](#page--1-0) Recent work has further shown that when narrow channels are used to promote laminar flow with high shear rates, the separation becomes almost entirely based on density difference [\[17,18\]](#page--1-0), especially in the intermediate range of particle Reynolds numbers $(1 < Re_t < 500)$.

The standard Reflux Classifier design has been in commercial use for about 8 years, initially in the coal industry but increasingly also in the mineral processing industry, where it is used to separate valuable material from gangue based on differences in their particle density [\[19\].](#page--1-0) In all the commercial applications to date, the particle species being separated have both been denser than the water used to fluidise the unit. However, cenospheres are buoyant in water, and only present at low concentrations. Thus their recovery and concentration from fly ash in a standard RC would result in an unacceptably high level of ultrafine dense silica being entrained in the overhead product stream. So instead, it was decided to trial an alternative approach of inverting the entire apparatus so that the fluidising water is added from above, and the inclined channels are located at the base (Fig. 1b). The downward flow of fluidising (or wash) water near the exit at the top should help

Fig. 1. Diagram of the (a) standard and (b) Inverted Reflux Classifier.

prevent the elutriation of dense silica particles in the overhead product, whilst the enhanced effective "settling" area of the inclined channels at the base should help prevent the buoyant cenospheres being lost in the underflow. This is a novel application of the Reflux Classifier concept. The focus of this paper is on preliminary experiments designed to test its potential.

The background theory of how standard Reflux Classifiers work is outlined in Section 2. [Section 3](#page--1-0) gives details of the experimental methodology of the present study. There were two stages in the work. Firstly, laboratory-scale experiments were performed in a unit with a horizontal cross-sectional area of 80 mm \times 100 mm. Then pilot-scale experiments were performed in a unit with cross-sectional area of 300 mm \times 300 mm. The results of these experiments are presented and discussed in [Section 4,](#page--1-0) with final conclusions in [Section 5.](#page--1-0)

2. Theory

This paper is focussed on the recovery of positively buoyant cenospheres that float, whereas existing theory was developed for negatively buoyant particles that settle. So in this section the existing theory is summarised, but then some further comments are provided on potential additional factors that may come into play when separating buoyant cenospheres from negatively-buoyant particles.

For fine particles, the terminal free settling velocity u_t of a spherical particle of diameter d can be predicted using Stokes law:

$$
u_t = \frac{d^2(\rho_p - \rho_f)g}{18\mu}.\tag{1}
$$

Here ρ_n and ρ_f are the particle and fluid densities respectively, μ is the fluid dynamic viscosity and g is the gravitational acceleration. The correct particle density to use is its apparent density i.e. the total mass of the particle divided by the total occupied volume including any gas cavities. For cenospheres, this value can be anywhere from 1000 down to 200 kg/ $m³$ or even lower. For positively buoyant particles where $\rho_p < \rho_f$, the terminal free "settling" velocity predicted by Eq. (1) is negative, indicating that particles rise in the upward direction.

When a suspension of many particles is present, their settling (or rise) velocity is lower. The semi-empirical Richardson and Zaki [\[20\]](#page--1-0) equation describes this effect for a single-component suspension:

$$
u_h = u_t (1 - \phi_s)^n \tag{2}
$$

where u_h is the hindered settling velocity of particles with a terminal free settling velocity u_t , when present in a suspension with a solids volume fraction ϕ_s . In Stokes settling regime $n = 4.65$.

Within an inclined section, particles have only a relatively short vertical distance to fall before they settle against the lower surface of the channel, from where they can slide down to the base of the channel. In the case of buoyant particles such as cenospheres, they will instead rise to the upper (downward facing) surface of the channel and then slide up to the top. This so-called Boycott effect [\[15\]](#page--1-0) produces an increase in the effective settling area compared to a purely vertical column with the same footprint. Hence parallel inclined channels are beneficial in lamellae thickeners [\[21\]](#page--1-0), offering very high hydraulic loadings. In lamellae thickeners the objective is to simply capture all of the solid, often flocculated, particles onto the inclined surfaces, allowing clear supernatant to pass through. We may define the throughput advantage, F, of inclined channels as the ratio of the superficial upward velocity of the fluid in the vertical section, U, divided by the terminal free settling velocity, u_t , of the largest particle that can in principle be elutriated from the inclined channels assuming that the solids concentration is very low:

$$
F = \frac{U}{u_t}.\tag{3}
$$

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