Evaluation of Ilmenite as Dense Medium for Dry Coal Fluidized Bed Beneficiation

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ABSTRACT

Published work on dry dense medium fluidized beds has mainly used magnetite and fine coal to make up the dense medium. Magnetite is used to achieve the required cut densities, but its recovery and reuse are problematic because it attaches to the surface of the coal and discard material, and its surfaces become contaminated. This study focused on using ilmenite ($FeTiO_3$) as an alternative medium in the dry dense medium fluidization process due to its favorable surface properties of hydrophilic and sphericity. The initial investigation considered a reference medium, which consisted of ilmenite and sand (used for base-case tests), and a second medium, consisting of ilmenite and fine coal, which resembled that currently used in the dry dense medium fluidized bed process. Experiments to evaluate the performance of the ilmenite were carried out in a laboratory-scale cylindrical fluidized bed. Losses of the ilmenite were investigated by mixing and recovering the ilmenite using two different coal samples of 13.2–50 mm particle size. Density tracers were used to determine the écart probable moyen (EPM). At optimal conditions, the bed media consisting of sand and fine coal with ilmenite had EPM values of 0.045 and 0.05 at cut densities of 1.8 and 1.58 g/cm^3, respectively. No ilmenite losses were observed. The ilmenite surfaces contained no contaminants after 10 cycles. The highest ilmenite recovery achieved from the bed after high-intensity magnetic separation was 99.79%.

KEYWORDS: Dry beneficiation, coal, fluidized bed, ilmenite, sand, fine coal, medium

1. Introduction

Coal is the main resource for electricity generation and industrial metallurgical applications in South Africa (De Korte 2010). Coal is a complex sedimentary rock that comprises both organic and inorganic matters. The separation of coal is a process whereby the combustible portion (the float) is separated from the inert ash-containing material (the sink) to produce a product of higher quality that generally has higher calorific value. A wet coal-processing route is extensively utilized in South Africa. Drums, cyclones, and dense medium separation (DMS) are used to wash coarse coal; spirals are employed to process fine coal. These processes require significant subsequent water treatment to comply with environmental legislation (Mohanta et al. 2013; De Korte 2015).

Wet processes are becoming less viable because the remaining coal reserves in South Africa are situated in arid areas, such as the Waterberg (Hartnady 2010). Alternative dry-coal-beneficiation technologies are therefore sought, especially for use in arid areas. One such process is dry dense medium fluidized bed separation, in which the product is separated from the ash using a fluidized bed. This dry beneficiation process has the benefits of higher separation precision and quick return on investment (Frankland 1995; Luo and Chen 2001; Chen and Wei 2003; De Korte 2015).

Approximately two-thirds of coal reserves in China are found in arid areas. China University of Mining and Technology Research Center has extensively developed dry beneficiation using an air dense medium fluidized bed (ADMFB). This uses density and pseudo-fluid characteristics of the medium as critical parameters to separate coal from ash-forming mineral matter (Chen and Wei 2003). The Bohou process, which has been implemented in China, is an example of a dry dense medium fluidization process that has shown positive results for upgrading of coal in the size fraction −200 + 13 mm (Zheng 2016). The Bohou plant was designed to handle 500 t/h dry coal. This process is operated with a high-frequency screen to recover the fluidization medium from the float and sink products. Its successful operation confirmed that coal can be efficiently separated in an ADMFB at a cut point of 1.58 g/cm^3 with feed coal containing less than 5% moisture, producing a clean coal of 9.85% ash content; the separation has an écart probable moyen (EPM) of 0.05–0.08.

Magnetite is extensively used as a medium for wet DMS processes in the coal industry. The ADMFB process in China uses magnetite powder (45–452 μm) as a solid medium (Sahu et al. 2009). Particles with a density lower than that of the bed report to the float (clean coal), whereas particles heavier than the bed density report to the sink (tailings), which is predictable from Archimedes' Law (Luo et al. 2010). This technology has some challenges when using coal with a surface moisture content of more than 2%. The magnetite medium readily adheres to a wet coal surface due to its hydrophilic nature and the fluidizing quality is significantly affected because of the increase in viscosity (Luo et al. 2010; Mohanta and Meikap 2015). A portion of the magnetite becomes agglomerated due to its magnetic properties, especially when it is reused. This decreases contact efficiency between the particles and gas, thereby causing deterioration in particle dispersal and increasing local and overall non-uniformity in the bed density. Consequently, the bed fluidization and splitting performance tend to decrease. The resulting contaminated medium may also be difficult to recover or only be liberated with difficulty. As a result, the coal (float and sink) split quality decreases and the operating cost increases (Luo et al. 2010). Dardis (1987) argued that the loss of media materials can be expensive and plays a critical role in determining the financial viability of any process. Luo et al. (2010) proposed hydrophobic surface modification of magnetite particles to control the surface moisture content of feed coal.

Ilmenite (nominally $FeTiO_3$), a titanate ferrous iron mineral, is one of the main TiO_2-bearing minerals and the world's most valuable titanium-bearing ore (Song and Tsai 1989). It is a naturally occurring heavy mineral associated with mineral sands deposits and is the primary source for the production of titanium metal and titanium dioxide. It is separated from other minerals in a heavy mineral concentrate based on magnetic susceptibility properties (Balderson 1999). Nell and Den Hoed (1997) stated that a crude ilmenite concentrate produced from a Southern African East Coast deposit typically contains 90% ilmenite, 5% Ti-hematite, 3% spinel (including chromite and magnetite) and 2% silicates by mass.

Ilmenite was identified as a potential alternative medium for use in an ADMFB owing to its specific surface properties of hydrophobicity, smoothness and sphericity. These properties give ilmenite an advantage when compared with magnetite because it will not attach so easily to wet coal particles. The effects of various parameters (pressure drop as a function of superficial gas velocity, bed density (ilmenite and sand compared with ilmenite and fine coal) and the recoverability of the ilmenite and magnetite media) were investigated on a laboratory scale to evaluate the density split achieved when using ilmenite as a medium. Recovery of the ilmenite by magnetic separation and its reusability were also investigated.

2. Background

Dry beneficiation of coal with an ADMFB, which is performed with a gas–solid fluidized bed as the separating medium, has been proven as an efficient method of coal separation (Dwari and Hanumantha 2007). Dry-processing technologies are being evaluated for implementation in South Africa and its neighboring countries because these techniques are perceived to be less expensive than wet beneficiation processes regarding both capital and operating costs. No water is required during this process, thereby significantly lowering the environmental impact of coal processing (De Korte, 2013; Mohanta et al. 2013).

The world's first industrial ADMFB modularized dry coal beneficiation plant was developed by Shenhua Xinjiang Energy Co., Ltd. (China). It has a handling capacity of 40–60 t/h. Coal of size −100 + 10 mm is used as the feed to the ADMFB (Zhao et al. 2017). A binary mixture of magnetic powder (−3 + 0.06 mm) with fine coal (−1 mm) is used as the fluidization medium. The raw coal is screened and crushed. The −100 mm fraction is loaded into a dryer to remove the surface moisture, which increases the screen efficiency. The dried coal is screened at an aperture of 10 mm and the undersize (−10 mm) is considered as a clean product. The bed density is controlled by varying the coal/magnetite ratio. The float and sink products are screened at 1 mm to separate the bed medium from the coal. The success of this ADMFB modularized coal processing plant confirmed that coal can be efficiently separated at a cut point 1.46 g/cm^3, producing clean coal of 3.46% ash content and an EPM of 0.055 (Zhao et al. 2017).

2.1. Fluidization

Gupta and Sathiyamoorthy (1999) described fluidization as the phenomenon of imparting the properties of a fluid to a bed of particulate solids by passing a fluid (liquid or gas) through the latter at a velocity that brings the fixed or stationary bed to its loosest possible state just before its transformation into a fluid-like bed. The bed material determines the density of the bed. In an ADMFB, the bed acts like a liquid. Particles with density below that of the bed report to the float (top of the bed), whereas heavier particles report to the sink (bottom of the bed). Stable fluidization and micro-bubbles must be achieved to obtain efficient dry separation conditions (Dwari and Hanumantha 2007). Desirable physical properties of an ADMFB include a bed medium of low viscosity and high fluidity and a bed density that is well distributed in three-dimensional space and remains stable over time. The criterion to fix the operating velocity depends on various parameters, including the pressure drop through the fluidized bed and gas distributor, the intensity of back-mixing, particle attrition or agglomeration, maximum bubble size, weeping through grids, bed expansion and gas channeling (Mohanta et al. 2013). It has been proposed that the optimum operating velocity in an ADMFB is about twice the minimum fluidization velocity to achieve uniform, smooth, stable fluidization using Geldart Group B particles (Chan and Beeckman 1982; Dong and Beeckmans 1990; Kozanoglu et al. 1993; Sahan and Kozanoglu 1997).

Dong et al. (2016) studied the development of ADMFB into a pulsing dense-phase gas–solid fluidized bed (PDGFB) that reduced the minimum fluidization of the magnetite medium when compared with that of an ADMFB, adjusted the stability of the bed density, and normalized the motion of the magnetite medium. Fine anthracite coal of −6 + 1 mm size was cleaned using a PDGFB with a true density of 2.03 g/cm^3 and EPM of 0.09 g/cm^3. Ling et al. (2018) studied the distribution of the size, ash content and density of coal particles along the front discharge section of a compound dry separator. The fine particle content of the raw coal had a significant effect on the clean coal: the high-density minerals contained in clean coal were mainly derived from the fine gangue −6 + 0.5 mm, which cannot be effectively separated. The low and high separation densities of fine coal (−6 + 0.5 mm) were much higher than those of coarse coal (−50 + 6 mm) under the same operating conditions.

2.2. Medium

The criteria required for media selection include the composition of the suspension, particle size, viscosity, type of medium solids, cost, recoverability and ease of cleaning of medium solids (Hand et al. 2002). The ideal separating medium for wet processing should be a suspension that is cheap, immiscible in water, capable of adjustment over a wide range of relative densities, stable, non-corrosive, low in viscosity and easily recoverable for reuse (Hand et al. 2002). Dry-processing technology is based on the differences in physical properties between coal and the medium, such as those of density, frictional coefficient, size, magnetic susceptibilities, shape, electrical conductivity, surface properties and lustrousness (Dwari and Hanumantha 2007). In practice, such an ideal medium does not exist: the selection of a medium for practical use will represent a compromise between these properties and factors such as availability and cost.

After careful observation of various types and sizes of solids during fluidization, Geldart established four clear identifiable types of particle behavior. When magnetite particles are less than 20 µm (Geldart Group C), it is challenging to obtain normal fluidization because the interparticle forces are more significant than those that air can exert on the particles (Geldart 1973). Several authors noted that a particle size ranging from 20 to 45 µm (Geldart Group A) appeared to be inappropriate for ADMFB due to back-mixing of coal particles (Choung et al. 2006); the range of 45–452 µm (Geldart Group B) is most suitable to conduct normal fluidization (Mak et al. 2008). He et al. (2016) studied the hydrodynamic characteristics of a dense medium gas–solid (DMGS) fluidized bed for coal separation. The process was evaluated using a magnetite medium (0.3–0.15 mm). The outcomes proved that the hydrodynamic characteristics of the fluidized bed, as well as the bed pressure drop and splitting density distribution, maintained stable and uniform conditions without extreme fluctuations.

Interest in the use of ilmenite in coal beneficiation lies in its reasonably high specific gravity (SG) of 4.5–5 g/cm^3 (Wills 2016). The high SG, together with the typically smooth pebble-like structure of beach sand, makes ilmenite a very suitable material for a fluidized bed. Ilmenite has a hydrophobic surface with a contact angle of 14° and does not readily adhere to a wet mineral surface (Drzymala 2007). It is also a paramagnetic mineral so it can be subsequently separated from the coal using an industrial high-gradient magnetic separator (HGMS) (Wills 2016).

3. Experimental

3.1. Materials

A run-of-mine (ROM) sample and a Anglo Final Export product sample (AFE) were obtained from Greenside Colliery in Witbank, South Africa. The coals were dried in air according to ASTM D3302/D3302M-12 (2012) to remove the surface moisture. No inherent moisture was removed. The samples were screened using a vibrating horizontal screen to create a sized feed of 50–13.2 mm, as currently used in the Bohou process (Zheng 2016).

Table 1. Proximate analysis of coal samples.

Sample	Moisture (%)	Volatiles (%)	Ash (%)	Fixed Carbon (%)
ROM	2.39	22.82	39.39	35.39
AFE	1.61	20.14	49.22	29.03

Table 2. Minerals identified in ilmenite sample by X-ray diffraction.

Ilmenite	Chemical formula	Mass %
Actinolite	$CA_2(Mg, Fe)_5Si_8O_{22}(OH)$	15.49
Rutile	TiO_2	2.80
Hematite	Fe_2O_3	12.56
Ilmenite	$FeTiO_3$	63.70
Quartz	SiO_2	0.80
Srebrodolskite	$CaFe_2O_4$	4.51

Proximate analysis was undertaken according to ASTM D5142-09 (2009) to determine the volatile matter, moisture content, ash content and, by difference, the fixed carbon within the coal sample. The ROM sample had higher moisture, volatiles and fixed carbon content in comparison with the AFE sample. The latter had a higher ash content (Table 1).

Ilmenite used for the experiments was supplied by Tronox Limited from Hillendale, South Africa. X-ray diffraction (XRD) analysis was used to identify the mineral phases and crystal structures in the sample. The samples were prepared according to the standardized PANalytical back-loading system, which provides nearly random distribution of the particles (Ermrich and Opper 2013). The samples were analyzed using a X'Pert Pro powder diffractometer (PANalytical, Netherlands) in θ–θ configuration with an X'Celerator detector and variable divergence and fixed receiving slits with Fe-filtered Co-Kα radiation (λ = 17.89 nm). The phases were identified using *X'Pert High score plus*® software. The relative phase amounts (mass%) were estimated using the Rietveld method (*Autoguan*® software). Errors were at the three-sigma level. The mineral phases identified by XRD are shown in Table 2. The average grade of the ilmenite was 63.70%.

Chemical composition of the samples was determined by X-ray fluorescence (XRF) analysis using an ARL Perform'X Sequential XRF instrument (Thermo Fisher Scientific, USA). Analyses were executed using *Quantas*® software, which showed that titanium (Ti) and iron (Fe) dominated in the ilmenite, at averages of 37.38% and 48.37%, respectively (Table 3).

Scanning electron microscopy (SEM; JSM-IT300HR, JEOL, Japan) was conducted using back-scattered electron mode. A micrograph of the particle morphology is shown in Figure 1. The density of the sample was 4.78 g/cm^3, as determined by gas pycnometry (AccuPyc II 1340, Micromeritics, USA).

4. Medium 1: sand and ilmenite

Foundry Sand (SiO_2) was used in conjunction with ilmenite as a reference fluidization medium to test whether separation by density could be achieved in a fluidized bed. Sand–ilmenite

Table 3. X-ray fluorescence analysis of ilmenite.

Element	Si	Ti	Al	Fe	Mn	Mg	Ca	K	Cr	V	Zr	Nb
Mass %	6.66	37.38	2.67	48.37	1.08	1.47	0.62	0.11	0.47	0.52	0.37	0.11

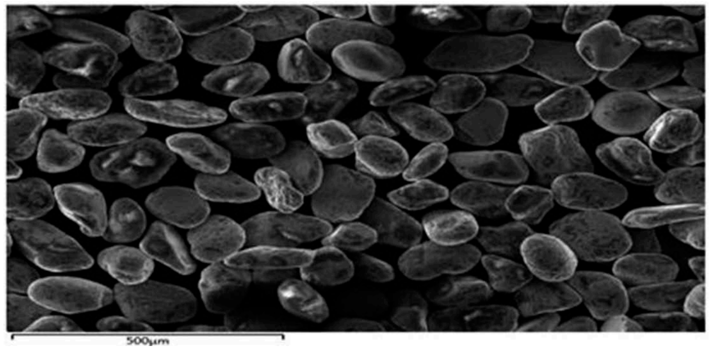

Figure 1. Back-scattered scanning electron micrograph of ilmenite particles.

medium was used as the base case for proof of concept, prior to measuring the performance of the actual medium proposed for industrial use (fine coal and ilmenite). Sand with an SG of 2.6 g/cm^3 was sourced from Rolfes Silica, Brits, South Africa. The sand was sub-rounded and available in a wide range of particle sizes. The dry graded silica sand comprised 98% SiO_2 and 0.18% Fe_2O_3. The medium was not recovered – it was discarded because it was only used to evaluate the performance of ilmenite as the dense medium in a controlled environment.

5. Medium 2: fine coal and ilmenite

The −13.2 mm coal was sieved to a size range −300 + 53 µm, defined as the fine coal fraction. This fine coal was used to reduce the bed density. A binary medium of ilmenite with fine coal (Group B particles) was used to achieve the bed split required in the coal beneficiation industry. The fine coal had a SG of 1.7 g/cm^3.

The particle size distributions of representative samples of ilmenite, sand and fine coal were determined using a laboratory sieve shaker (Figure 2). The cut points at 50% (d_{50}) were 151, 191 and 155 µm, respectively.

5.1. Apparatus

The experimental setup was designed to evaluate the fluidization behavior of ilmenite in an ADMFB and its subsequent recovery. The minimum fluidizing velocity, bed compositions of two binary media (ilmenite with fine coal and ilmenite with sand), bed density, EPM and yield were determined. The fluidization characteristics were first determined using density tracers; thereafter, splitting of the two different coal samples was evaluated.

The test work was carried out in a 150 mm diameter Perspex fluidized bed. Compressed air provided the fluidizing air. The air volume was controlled by an air bleed valve and measured using an orifice plate and U-tube manometer. The pressure drops across the bed and distributor plate were measured using U-tube manometers (Figure 3). Repeatable results were obtained at an operating superficial gas velocity of 0.060 m/s. In the first set of experiments, the fluidized bed was allowed to stabilize for 10 min and then tracers were gradually introduced onto its surface. After stratification for 30 s, the compressed air was shut off and all stratified tracers were retained in their positions in the mixture. The static bed was divided into five layers, comprising three floats (L5, L4 and L3) and two sinks (L2 and L1), as shown in Figure 3. The tracers were discharged layer by layer, using a scoop to remove material from top to bottom of the column.

The tracer particles ranged in SG from 1.3 to 3 g/cm^3; 10 tracers were available for each SG. The tracer particles comprised a magnetically susceptible material. They were cubic, with side dimensions of 12 mm. The fluidization characteristics were first determined using density tracers; thereafter, splitting of the two different coal samples was evaluated.

Sand or fine coal was used to reduce the bed density of the ilmenite to achieve the density split required by the coal

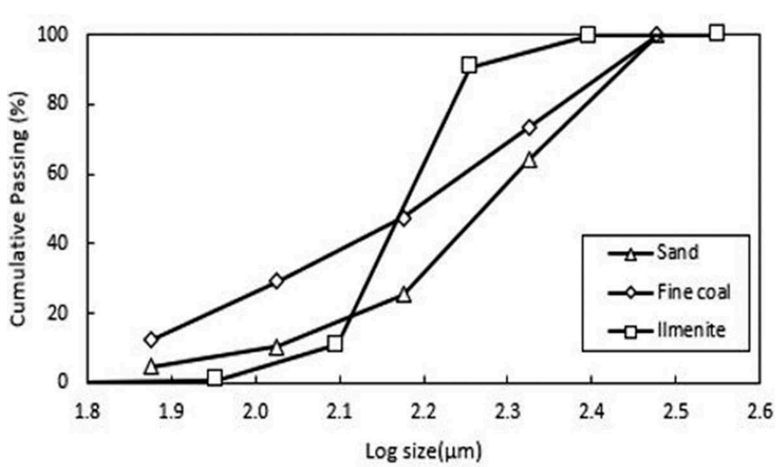

Figure 2. Particle size distributions of three media components.

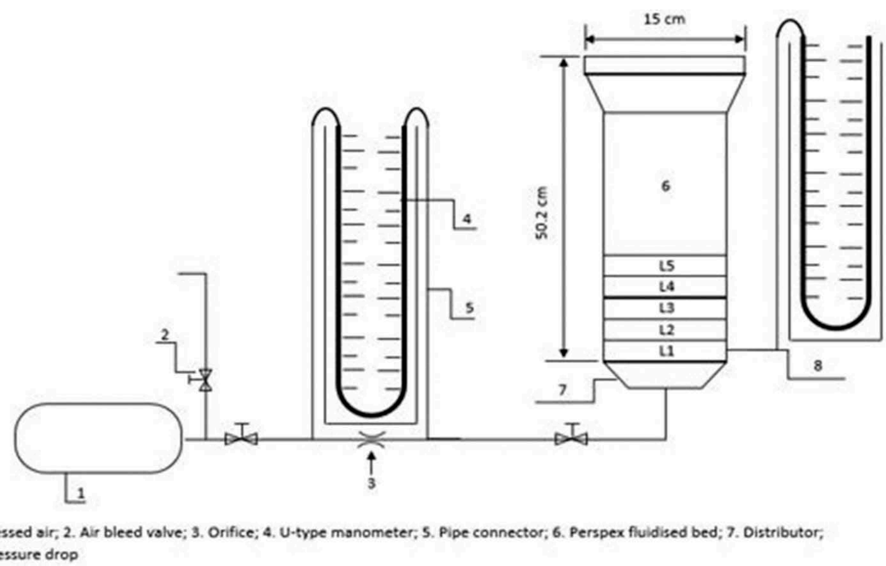

Figure 3. Schematic of air dense medium fluidized bed.

beneficiation industry. This resulted in a binary medium of Geldart Group B particles. The sand or fine coal were mixed in different ratios with the ilmenite and then used as the medium in the fluidized bed. The experiments using sand were carried out at low density for initial proof of concept.

According to Hovmand and Davidson (1971), the operating superficial gas velocity U for the transition from bubbling to slug flow is given by Equation (1):

$$(U - U_{mf})/\left(0.35(gD)^{0.5}\right) = 0.2 \tag{1}$$

where U_{mf} is the minimum fluidization velocity, D is the bed diameter and g is the acceleration due to gravity. Equation (1) gives good correlation for most experiments (Hovmand and Davidson 1971). When (U – U_{mf}) is larger than the value found from Equation (1), the bed will be in the slugging zone. Experiments were therefore conducted at a bed height of 0.120 m to prevent slugging: slugging only occurs in beds where the bed height-to-diameter ratio exceeds 2.

Baeyens and Geldart (1974) proposed the use of Equation (2) to calculate the maximum bed height below which the bed would be freely bubbling:

$$H_{fb} = \left(D - 2.51D^{0.2}\right)/\left(0.13D^{0.47}\right) \tag{2}$$

where H_{fb} and D are the height and diameter of the fluidized bed, respectively.

The maximum bed height of the freely bubbling bed was found to be 0.23 m; the static bed height was therefore fixed at 0.12 m for all experiments.

5.2. Magnetic separation

Ilmenite was separated from the bed materials using dry HGMS (Eriez Magnetics, South Africa), operated with a drum speed of 40–80 rpm and 4200 G interpole magnetic element (average on drum surface). The magnetic elements were high-temperature neodymium–iron–boron magnets. A rare-earth Magna Chute (Eriez Magnetics, South Africa) was used to determine the ilmenite losses. The relative magnetic strength was 3150 G.

After each run, the ilmenite was recovered was reused; fresh coal was introduced. The Magna Chute was used to clean the magnetic ilmenite recovered to enable accurate accounting of recovery of the medium.

6. Results and discussion

6.1. Effect of fluidization medium on coal splitting

The effect of fluidization medium density on the dry separation of coal was investigated using different concentrations of sand or fine coal relative to ilmenite as the fluidization medium.

The ilmenite samples fell into Group B of Geldart's classification of powders, which meant that the minimum fluidization velocity was equal to the minimum bubbling velocity (Geldart 1973). A plot of pressure drop across the bed as a function of superficial gas velocity was experimentally determined. U_{mf} is the superficial gas velocity at which the pressure drop is equal to the weight of the bed. Figure 4 shows that the U_{mf} of the ilmenite sample was 0.030 m/s.

The U_{mf} of ilmenite, sand and fine coal were also calculated using the Ergun Equation (3) (Ergun 1952):

$$\frac{1,75}{\Phi_s\varepsilon_{mf}^3}\left(\frac{d_p u_{mf}\ {}_g}{\mu}\right)^2 + \frac{150(1-\varepsilon_{mf})}{\Phi_s^2\varepsilon_{mf}^3}\left(\frac{d_p u_{mf}\rho_g}{\mu}\right) = \frac{d_p^3\rho_g\left(\rho_s-\rho_g\right)g}{\mu^2} \tag{3}$$

where d_p is particle diameter d_{50}, U_{mf} is the superficial gas velocity at minimum fluidizing velocity, ρ_g is gas density, μ is viscosity of the gas, $\emptyset_s$ is the sphericity of a particle, ε_{mf} is the void fraction in a bed at minimum fluidizing conditions and ρ_s is density of the solids.

The value of U_{mf} of ilmenite calculated using the Ergun Equation (3) was found to be 0.031 m/s using values of bed voidage: 0.37 (0.44 recommended value by Haughey and

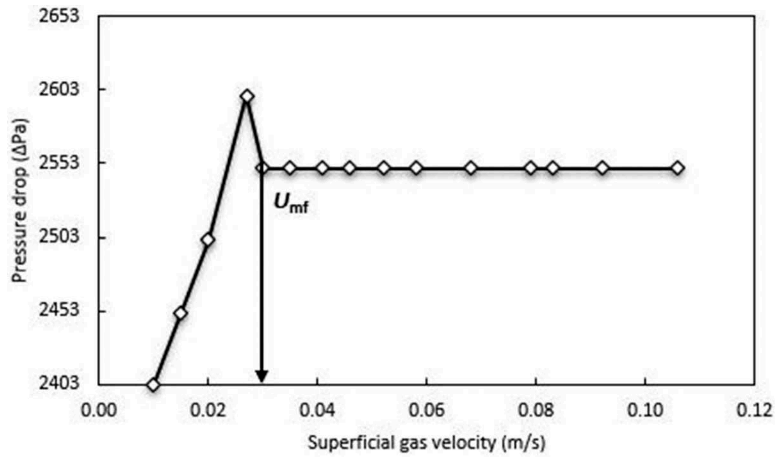

Figure 4. Pressure drop as a function of superficial gas velocity of ilmenite.

Beveridge (1969)); particle diameter d_{50} (mm): 0.151; particle density (kg/m^3): 4780; gas pressure (kPa): 88; gas temperature (°C): 25; molecular mass (g/mol): 28.84; gas density (kg/m^3): 1.0244; gas viscosity (Ns/m^2): 1.8 × 10^{-5}. It was found that a lower bed voidage significantly affected the minimum fluidization velocity and would increase the drag component.

An experiment was first conducted using ilmenite only as the medium; it was found that the observed bed split occurred at 3 g/cm^3 with a bed voidage of 37%. This bed split was too high for beneficiation of coal in a density ranging between 1.3 g/cm^3 and 2.3 g/cm^3. Sand or fine coal was therefore added to the ilmenite to reduce the bed density. The sand or fine coal and ilmenite were mixed in different ratios and used as the medium in the fluidized bed. Figure 5 displays the pressure drop across the bed as a function of superficial gas velocity for the two binary media. The results are summarized in Table 4.

6.2. Performance of air dense medium fluidized bed: partition curve

Figure 6 displays the relative deportments of tracers of different density into the five layers of the bed using the two binary media (fine coal with ilmenite and sand with ilmenite).

The tracer positions were used to determine the EPM values of the fluidized bed for the different media compositions. An ADMFB is efficient when the EPM ranges from 0.04 to 0.12 (Chen and Wei 2003). The tracer particles were stratified within the bed into floats and sinks in the same way that coal would be separated. The tracer particles recovered from each layer were sieved and counted, and the data used to construct a partition curve according to Equation (4) (Wills 2016):

$$EPM = \frac{\rho_{25} - \rho_{75}}{2}, \quad (4)$$

where ρ_{25} and ρ_{75} are the density cut points at 25% and 75% of the partition curve, respectively. The cut point at 50% (ρ_{50}) was read from the partition curve, as illustrated in Figure 7. This is the lower cut point and the required bed density in the coal beneficiation industry (De Korte 2015). The Napier–Munn (1991) correction (Equation (5)) was applied to the partition curve:

$$Y = 1/(1 + \exp[1.099(\rho_{50} - \rho)/Ep\ ]) \quad (5)$$

where Ep is the EPM. This provided statistical calculations, knowing the cut point at 50% (required cut density), particle density and EPM.

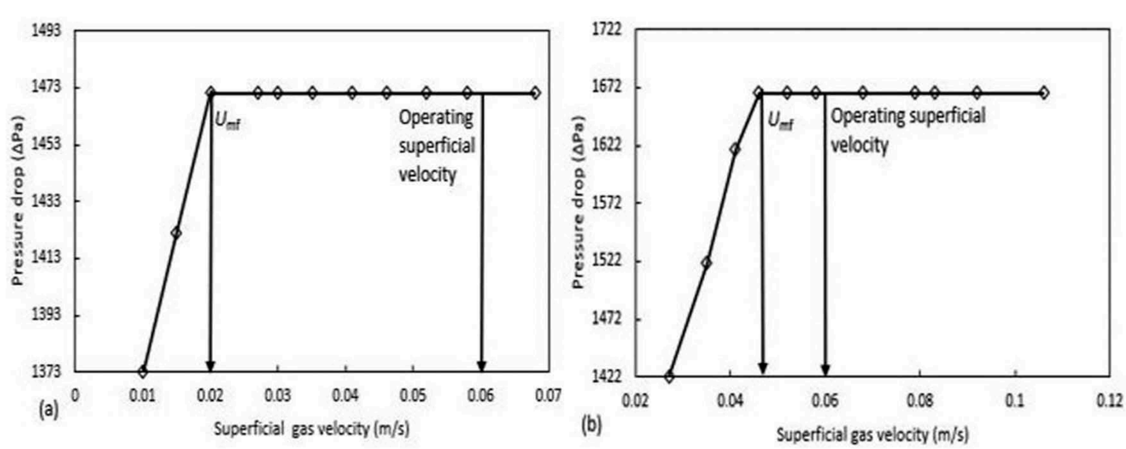

Figure 5. Pressure drop as a function of superficial gas velocity using binary media of (a) 60% fine coal and 40% ilmenite and (b) 70% sand and 30% ilmenite.

Table 4. Split results at different fine coal- or sand-to-ilmenite ratios.

Fine coal (%)	U_{mf} (m/s)	Pressure drop (ΔPa)	Average SG (g/cm^3)	Observed split (g/cm^3)	Sand (%)	U_{mf} (m/s)	Pressure drop (ΔPa)	Average SG (g/cm^3)	Observed split (g/cm^3)
0	0.030	2550	2.94	3.00	0	0.030	2550	2.94	3.00
10	0.028	2403	2.78	2.70	10	0.032	2354	2.78	2.75
20	0.025	2354	2.62	2.50	20	0.034	2305	2.62	2.60
30	0.020	2157	2.27	2.30	30	0.034	2305	2.47	2.45
40	0.020	1912	2.05	2.00	40	0.035	2108	2.31	2.30
50	0.021	1716	1.83	1.80	50	0.036	2059	2.15	2.10
60	0.020	1520	1.60	1.58	60	0.042	1814	1.99	2.00
70	0.020	1128	1.38	1.40	70	0.046	1667	1.83	1.80
80	0.017	883	1.16	1.20	80	0.048	1569	1.67	1.65
100	0.015	539	0.71	0.70	100	0.052	1275	1.36	1.35

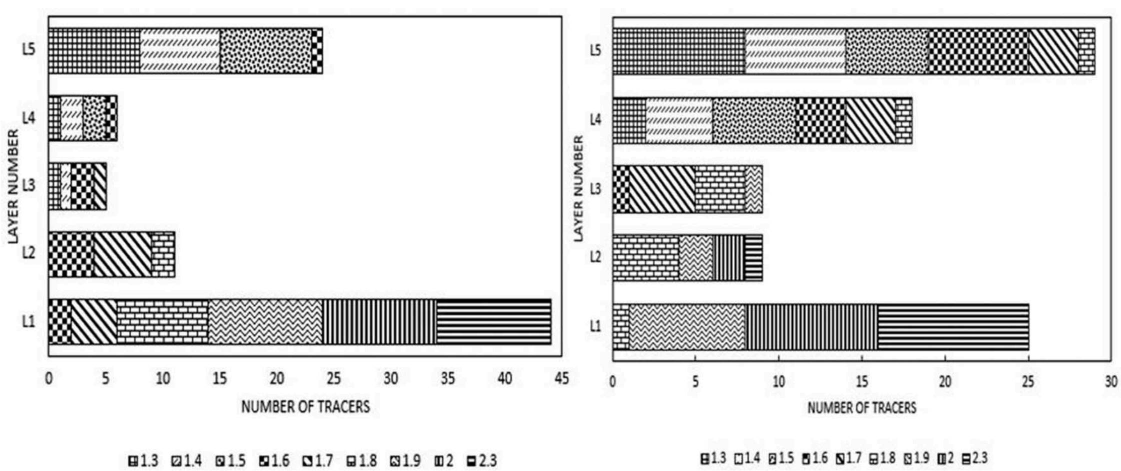

Figure 6. Deportment of tracers by density into five bed layers during fluidization using binary media of (a) 60% fine coal and 40% ilmenite and (b) 70% sand and 30% ilmenite.

All tracers reported in the top of the bed for both media in the range 0–30% fine coal and 0–40% sand mixed with ilme-nite. For the 80–100% fine coal media, all tracers sank (Table 5) so the EPM could not be calculated. Stratification of the sand or fine coal media quickly increased when their concentrations were above 50% and 40%, respectively, and the separation efficiencies were classified as excellent for an ADMFB process (Kalenda et al. 2017).

6.3. *Stability of bed using binary medium of fine coal and ilmenite*

A binary medium of Group B 40% ilmenite with 60% fine coal at a cut point of 1.58 g/cm^3 was used to achieve the bed split required in the coal beneficiation industry. The static bed was divided into three layers: top, middle and bottom. The bed was found to be uniform and stable, with no segregation taking place (Figure 8).

6.4. *Stratification of coals in bed with fluidization by ilmenite and fine coal medium*

Raw coal needs to be washed at a low cut density to produce an export-grade coal. A cut density at 1.58 g/cm^3 using the medium comprising 40% ilmenite with 60% fine coal appeared to be acceptable. Experiments were therefore con-ducted at a cut point of 1.58 g/cm^3. The ROM or AFE coal

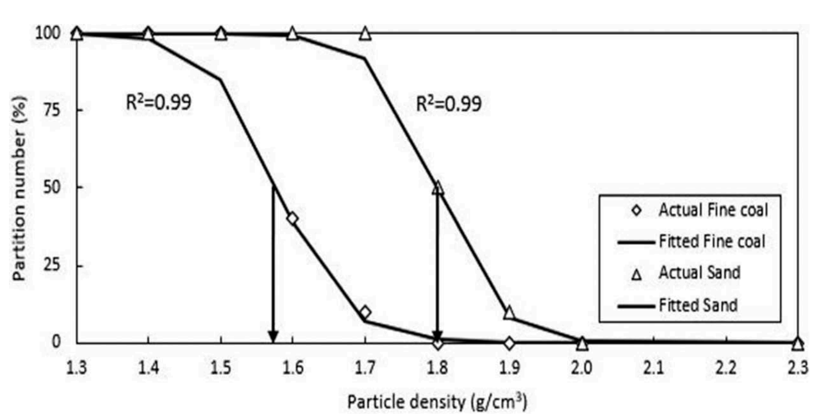

Figure 7. Partition curves for fluidization of density tracers using binary media of 60% fine coal and 40% ilmenite and 70% sand and 30% ilmenite.

Table 5. Effect of fluidization medium composition on EPM, as determined from distribution of density tracers.

Fine coal (%)	Écart probable moyen	Sand (%)	Écart probable moyen
0	–	0	–
10	–	10	–
20	–	20	–
30	–	30	–
40	0.11	40	–
50	0.06	50	0.12
60	0.05	60	0.11
70	0.07	70	0.045
80	–	80	0.05
100	–	100	0.05

particles (−50 + 13.2 mm) were loaded into the bed and the compressed air was shut off after stratification.

The ash content was determined for the coal particles discharged from the float and sink, as shown in Table 6. The yield was calculated using Equation (6) (Gupta and Yan 2006):

$$Yield = \frac{\text{Ash in feed (\%)} - \text{Ash in float (\%)}}{\text{Ash in sink (\%)} - \text{Ash in float (\%)}} \times 100. \qquad (6)$$

The yields of the ROM and AFE coals were 61.44% and 71.27%, respectively. These results proved that good splitting can be achieved using a binary medium of ilmenite mixed with fine coal.

6.5. *Recovery of medium using dry magnetic separator*

Three samples of ROM coal were fluidized with magnetite as the medium and the mixtures were sieved for 10 min on a sieve size of 3.35 mm. A dry low-intensity magnetic separator of 1680 G was used to recover the magnetite and a Magna Chute of 1350 G was used to clean the magnetic magnetite recovered to achieve true accounting of the recovered medium. Figure 9 shows that dry coal had the highest recovery of magnetite; the lowest recov-ery was observed in coal containing 2% moisture.

It was necessary to ascertain whether ilmenite could be subsequently recovered from the bed material. Seven samples of ROM coal were fluidized with a binary medium of ilmenite with fine coal. The mixtures were then sieved for 10 min on a sieve size of 3.35 mm. A dry 4200 G HGMS was used to recover the ilmenite. The 3150 G Magna Chute was then used to clean the magnetic ilmenite fraction to determine the true recovery. Figure 10 shows that when ilmenite was used only once with dry coal (containing only surface moisture), it was recovered with an efficiency of 99.79%. The recovery dropped to 99.11% after the ilmenite was reused 10 times with dry coal. For wet coal, a linear decrease in recovery was observed with an increase in moisture content. The lowest recovery, measured for 4% surface moisture of the coal, was still almost 99%, which is considered acceptable.

Figure 11 shows the associated losses of ilmenite under these conditions. Ilmenite did not attach to the surface of dry coal. The lowest loss of 5.12 g ilmenite/kg coal was reported for ilmenite used only once; the wet coal (4% moisture content) reported the highest loss of 24.25 g/kg coal.

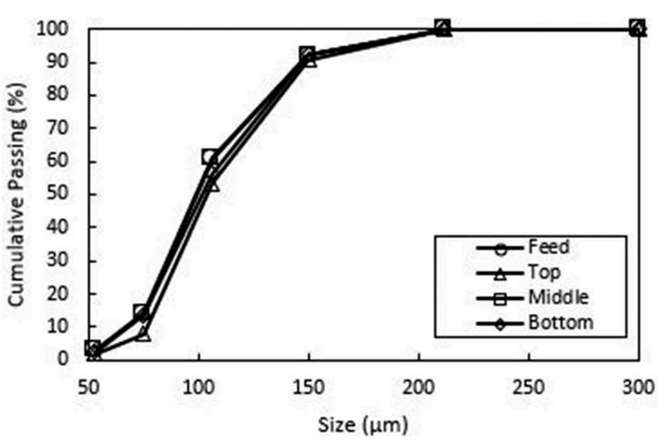

Figure 8. Stability of bed by size using a medium of 60% fine coal and 40% ilmenite.

Table 6. Stratification of coal samples in the bed when using 40% ilmenite and 60% fine coal as fluidization medium.

Coal	Bed location	Ash content (%)	Yield (%)
ROM	Float (clean coal)	11.0	61.4
	Sink (reject coal)	19.3	
AFE	Float (clean coal)	13.8	71.3
	Sink (reject coal)	55.6	

7. Conclusions

This research established the feasibility of dry coal beneficia-tion using a binary medium of ilmenite with fine coal or sand. The ilmenite properties in an ADMFB were studied to deter-mine its potential for use as a fluidization medium in dry coal beneficiation. Its specific surface properties and sphericity are beneficial to this application. It was found that ilmenite as a medium (−355 + 63 μm) delivered cleaner surfaces and higher recoveries at higher moisture levels when compared with magnetite. This can be attributed to the hydrophobicity of the ilmenite, which resulted in minimal ilmentite attach-ment on the coal and discard surfaces. The ilmenite surfaces also stayed clean of contaminants even after 10 cycles of reuse.

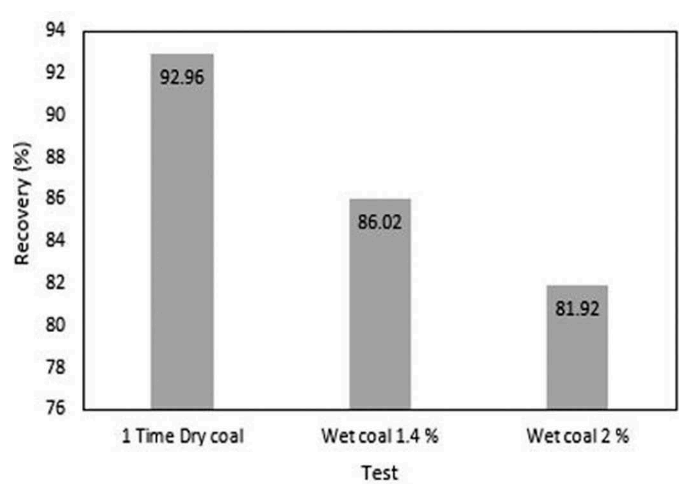

Figure 9. Effect of moisture content of coal on recovery of magnetite from fluidized bed material by Magna Chute.

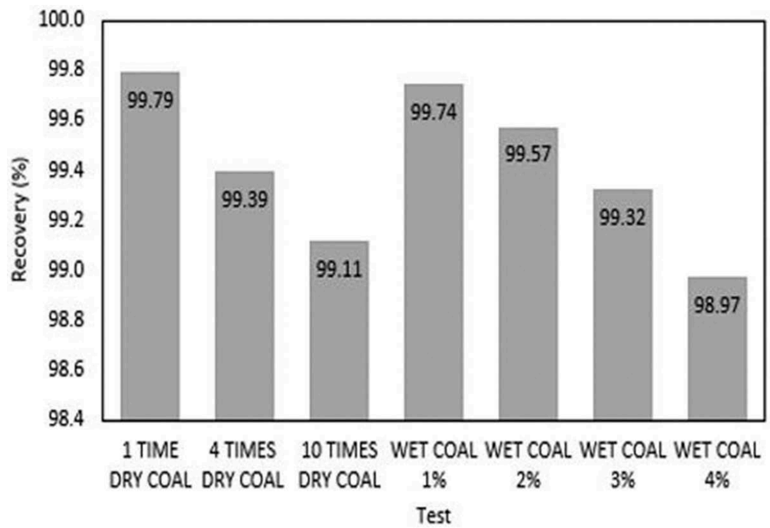

Figure 10. Effect of moisture content of coal and number of reuses on recovery of ilmenite from fluidized bed material by Magna Chute.

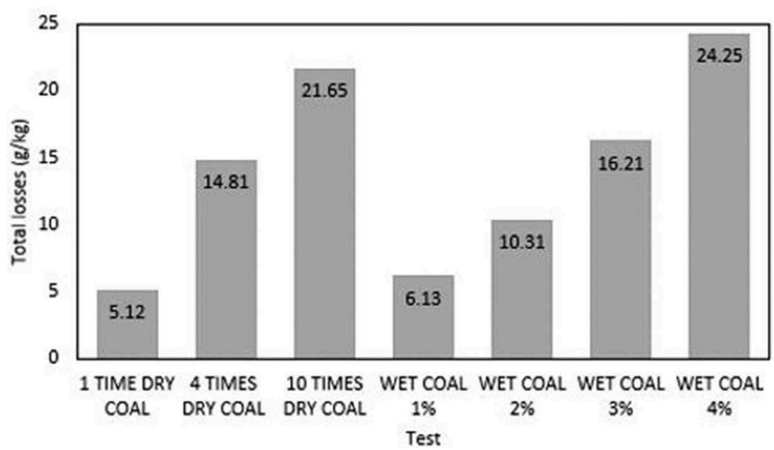

Figure 11. Effect of moisture content of coal and number of reuses on loss of ilmenite from fluidized bed material after Magna Chute treatment.

For a medium comprising fine coal with ilmenite, density stratification did not occur below a fine coal concentration of 30%. Stratification of the fine coal quickly increased when its concentration exceeded 40% and the EPM ranged between 0.05 and 0.11.

Coal was efficiently separated in a laboratory-scale ADMFB. Yields of feeds of ROM and AFE coal samples of size −50 + 13.2 mm were 61.44% and 71.27%, respectively, when using a binary medium of 60% fine coal with 40% ilmenite at a bed split of 1.58 g/cm^3 and EPM of 0.05. Such a configuration is expected to perform well in an industrial application.

The binary medium of 40% ilmenite with 60% fine coal was mixed with both dry and wet coal and the ilmenite recovered using a dry HGMS. The results revealed that ilmenite did not attach to the surface of dry coal under conditions that gave the highest recovery of 99.79%. The recovery of ilmenite slightly decreased on increasing the surface moisture content of the coal. The results revealed that magnetite did attach to the surface of dry coal: the highest recovery was 92.96%. Ilmenite thus performed better than magnetite as a fluidization medium.

In conclusion, ilmenite is considered a viable alternative medium to magnetite for use in a dry dense medium fluidized bed process due to its favorable material properties.

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References

ASTM D3302/D3302M-12, Standard test method for total moisture in coal, ASTM International, West Conshohocken, PA, 2012. www.astm.org. doi:10.1094/PDIS-11-11-0999-PDN.

ASTM D5142-09, Standard test methods for proximate analysis of the analysis sample of coal and coke by instrumental procedures (Withdrawn 2010), ASTM International, West Conshohocken, PA, 2009. www.astm.org.

Baeyens, J., and Geldart, D., 1974, "An investigation into slugging fluidized beds." *Chemical Engineering Science*, 29. pp. 255–265. doi:10.1016/0009-2509(74)85051-7.

Balderson, G. F., 1999. Flowsheet development options for a Greenfield titanium minerals project, In *Proceedings of Heavy Minerals Conference. Symposium Series S23*, (R. G. Stimson, Ed.), Johannesburg: South African Institute of Mining and Metallurgy, pp. 125–136.

Chan, E. W., and Beeckman, J. M., 1982, "Pneumatic beneficiation of coal fines using the counter-current fluidized cascade." *International Journal of Mineral Processing*, 9. pp. 157–165. doi:10.1016/0301-7516(82)90024-2.

Chen, Q., and Wei, L., 2003, "Coal dry beneficiation technology in China: the state-of-the-art." *Particuology*, 1(2). pp. 52–56. doi:10.1016/S1672-2515(07)60108-0.

Choung, J., Mak, C., and Xu, Z., 2006, "Fine coal beneficiation using an air dense medium fluidized bed." *Coal Preparation*, 26. pp. 1–15. doi:10.1080/07349340600619345.

Dardis, K. A., 1987. The design and operation of heavy medium recovery circuits for improved medium recovery. *Proceedings of the Dense Medium Operators Conference*. Brisbane: Australasian Institute of Mining and Metallurgy, pp. 157–184.

De Korte, G. J., 2010, "Coal preparation research in South Africa." *Journal of the Southern African Institute of Mining and Metallurgy*, 110. pp. 361–364.

De Korte, G. J., 2013, Dry processing versus dense medium processing for preparing thermal coal, In *17th International Coal Preparation Congress*, Istanbul, Turkey, pp. 1–6.

De Korte, G. J., 2015, "Processing low-grade coal to produce high-grade products." *Journal of the Southern African Institute of Mining and Metallurgy*, 115. pp. 569–572. doi:10.17159/2411-9717/2015/v115n7a2.

Dong, L., Zhou, E., Cai, L., Duan, C., Zhao, Y., and Luo, Z., 2016, "Fluidization characteristics of a pulsing dense-phase gas–solid fluidized bed for high-density separation of fine anthracite." *Energy & Fuels*, 30(9). pp. 7180–7186. doi:10.1021/acs.energyfuels.6b01468.

Dong, X., and Beeckmans, J. M., 1990, "Separation of particulate solids in a pneumatically driven counter-current fluidized Cascade." *Powder Technology*, 62. pp. 261–267. doi:10.1016/0032-5910(90)80113-D.

Drzymala, J., 2007, *Mineral Processing: Foundations of Theory and Practice of Minerallurgy,* Wroclaw: Wroclaw University of Technology, p. 271.

Dwari, R. K., and Hanumantha, R. K., 2007, "Dry beneficiation of coal—A review." *Mineral Processing and Extractive Metallurgy Review*, 28. pp. 177–234. doi:10.1080/08827500601141271.

Ergun, S., 1952, "Fluid flow through packed columns." *Chemical Engineering Progress*, 48. pp. 89–94.

Ermrich, M., and Opper, D., 2013, *XRD for the Analyst*, Netherlands: PANalytical.

Frankland, S. C., 1995. *Dry beneficiation of coal*. Department of Trade and Industry, Coal Research and Development Programme Contractors, Report No. Coal-R063. Energy Technology Support Unit, Harwell, Oxfordshire, United Kingdom.

Geldart, D., 1973, "Types of gas fluidization." *Powder Technology*, 7. pp. 285–292. doi:10.1016/0032-5910(73)80037-3.

Gupta, A., and Yan, D. S., 2006, *Mineral Processing Design and Operation: An Introduction*, Perth: Elsevier. pp. 549.

Gupta, C. K., and Sathiyamoorthy, D., 1999, *Fluid Bed Technology in Materials Processing,* Boca Raton, FL: CRC Press, p. 498.

Hand, P. E., England, T., Michael, D. C., Falcon, L. M., and Yell, A. D., (eds.), 2002, *Coal Preparation in South Africa*, Johannesburg: The South African Coal Processing Society. pp. 298.

Hartnady, C., 2010, "South Africa's diminishing coal reserves." *South African Journal of Science*, 106(9/10). pp. 1–5. doi:10.4102/sajs.v106i9/10.369.

Haughey, D. P., and Beveridge, G. G., 1969, "Structural properties of packed beds—a review." *The Canadian Journal of Chemical Engineering*, 47. pp. 130–140. doi:10.1002/cjce.v47:2.

He, J., Zhao, Y., He, Y., Luo, Z., Li, H., and Duan, C., 2016, "Hydrodynamic characteristics of the dense medium gas–solid fluidized bed for coal beneficiation and cleaning." *Particulate Science and Technology*, 34(2). pp. 173–183. doi:10.1080/02726351.2015.1055353.

Hovmand, S., and Davidson, J. F., 1971, Pilot plant and laboratory scale fluidized reactors at high gas velocities: the relevance of slug flow, In *Fluidization*, (J. F. Davidson and D. Harrison, Eds.), New York: Academic Press, pp. 193–259.

Kalenda, T. N., North, B., and Naude, N., 2017. Evaluation of ilmenite as a possible medium in a dry dense medium fluidized bed, In *Proceedings of IFSA 2017, Industrial Fluidization South Africa*, (T. Hadley and B. North, Eds.), Johannesburg: Southern African Institute of Mining and Metallurgy, pp. 81–90.

Kozanoglu, B., Levy, E. K., Ulge, T., Sahan, R., and Schmitt, T., 1993. Prediction of rates of coal cleaning in a fluidized bed of magnetite. In *AIChE Symposium Series*, Miami Beach, FL, vol. 89, pp. 150–161.

Ling, X., He, Y., Zhao, Y., Li, G., Wang, J., and Wen, B., 2018, "Distributions of size, ash, and density of coal particles along the front discharge section of a compound dry separator." *International Journal of Coal Preparation and Utilization*, 38(8). pp. 422–432. doi:10.1080/19392699.2016.1278214.

Luo, Z., and Chen, Q., 2001, "Effect of fine coal accumulation on dense phase fluidized bed performance." *International Journal of Mineral Processing*, 63(4). pp. 217–224. doi:10.1016/S0301-7516(01)00050-3.

Luo, Z. F., Zhu, J. F., Tang, L. G., Zhao, Y. M., Guo, J., Zuo, W., and Chen, S. L., 2010, "Fluidization characteristics of magnetite powder after hydrophobic surface modification." *International Journal of Mineral Processing*, 94(3–4). pp. 166–171. doi:10.1016/j.minpro.2010.02.009.

Mak, C., Choung, J., Beauchamp, R., Kelly, D. J. A., and Xu, Z., 2008, "Potential of air dense medium fluidized bed separation of mineral matter for mercury rejection from Alberta sub-bituminous coal." *Industrial and Engineering Chemistry Fundamentals*, 28. pp. 115–132.

Mohanta, S., and Meikap, B. C., 2015, "Influence of medium particle size on the separation performance of an air dense medium fluidized bed separator for coal cleaning." *South African Journal of Chemical Engineering*, 115. pp. 761–766.

Mohanta, S., Rao, C. S., Daram, A. B., Chakraborty, S., and Meikap, B. C., 2013, "Air dense medium fluidized bed for dry beneficiation of coal. Technological challenges for future." *Particulate Science and Technology*, 31(1). pp. 16–27. doi:10.1080/02726351.2011.629285.

Napier–Munn, T. J., 1991, "Modelling and simulating dense medium separation processes." *Minerals Engineering*, 4(3/4). pp. 329–346. doi:10.1016/0892-6875(91)90139-M.

Nell, J., and Den Hoed, P., 1997. Separation of chromium oxides from ilmenite by roasting and increasing the magnetic susceptibility of Fe,O,-$FeTiO_3$,(ilmenite) solid solutions. *Proceedings of Heavy Minerals Conference. Symposium Series S17*. R. E. Robinson (ed.). Johannesburg: South African Institute of Mining and Metallurgy, pp. 75–78.

Sahan, R. A., and Kozanoglu, B., 1997, "Use of an air-fluidized bed separator in a dry coal cleaning process." *Energy Conversion and Management*, 38. pp. 269–286. doi:10.1016/S0196-8904(96)00044-1.

Sahu, A. K., Biswal, S. K., and Parida, A., 2009, "Development of air dense medium fluidized bed technology for dry beneficiation of coal—A review." *International Journal of Coal Preparation and Utilization*, 29. pp. 216–241. doi:10.1080/19392690903113847.

Song, Q., and Tsai, S. C., 1989, "Flotation of ilmenite using benzyl arsonic acid and acidified sodium silicate." *International Journal of Mineral Processing*, 26. pp. 111–121. doi:10.1016/0301-7516(89)90046-X.

Wills, B. A., 2016, *An Introduction to the Practical Aspects of Ore Treatment and Mineral Recovery*, 8th edn., Amsterdam: Elsevier. pp. 496.

Zhao, Y., Li, G., Luo, Z., Zhang, B., Dong, L., Liang, C., and Duan, C., 2017, "Industrial application of a modularized dry-coal-beneficiation technique based on a novel air dense medium fluidized bed." *International Journal of Coal Preparation and Utilization*, 37(1). pp. 44–57. doi:10.1080/19392699.2015.1125344.

Zheng, S., 2016. Large-scale high separation precision air dense medium dry coal beneficiation system, In *Proceedings of XVIII International Coal Preparation Congress*, *Russia*, (V. Litvinenko, Ed.), Switzerland: Springer International Publishing, pp. 1–15.