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Evaluation of ilmenite as a possible medium in a dry dense medium fluidized bed

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Abstract— Alternative dry coal beneficiation method is in high demand, especially in arid geographical environments. This dry process eliminates the need for water usage and there are suggestions that it has the benefits of high separation precision and quick return on investment. Current published dry dense medium separation work has mainly focused on using magnetite as a medium, but various complications have been encountered when magnetite was used. This study will extend the approach by investigating the use of ilmenite as an alternative medium for this dry dense medium fluidization process. The experiment was conducted on coal sized between 13-50 mm in a laboratory scale cylindrical fluidized bed, and density tracers were used to determine the Ecart Probable Moyen. The initial investigation considered a medium consisting of fine sand with ilmenite and fine coal with ilmenite. The results revealed that a uniform and stable fluidized bed can be achieved in both scenarios. At optimal conditions, the bed medium mixtures consisting of Ilmenite with fine sand and fine coal had a separation EPM of 0.045 and 0.05, and a cut density of 1.8 and 1.58g/cm^3 respectively. Losses of ilmenite on coal were found to be 24.17 kg/t at external moisture of 4%.

INTRODUCTION

Coal is a complex sedimentary rock that contains both organic and inorganic matter, however, in general, raw coal does require the removal of ash forming inorganic matter, and this can be achieved through beneficiation processes.[1] In South Africa, this beneficiation process is mainly based on using wet float and sink processes, and is therefore a water intensive process. This process water requires significant water treatment to comply with environmental policies. Moreover, these wet processes are becoming less viable because much of the remaining coal reserves within South Africa are situated in environments that are arid, where pipelines must supply water. Consequently, alternative dry coal beneficiation methods are being sought, especially in the arid geographical environment. This dry beneficiation process eliminates the need for water, and there are suggestions that it has the benefits of higher separation precision and quick return on investment. An example of this is the dry dense medium fluidization processes, which have shown positive results for the upgrading of coal in the size fraction + 13-50 mm.[2]

The loss of medium solids can be costly and plays a critical role in determining the economics of any process. Magnetite is currently widely used as a medium during wet dense medium separation (DMS) processes in the coal industry. There are some questions on the future availability of magnetite for use in the conventional wet DMS processes, and therefore it is important for the coal industry to investigate alternative medium possibilities, not only for wet beneficiation but also for dry beneficiation.[3]

Ilmenite (nominally $FeTiO_3$) is a naturally occurring heavy mineral associated with the heavy minerals sands deposits. Ilmenite is separated from other minerals in the heavy mineral concentrate, based on magnetic susceptibility properties.[4] A crude ilmenite concentrate produced from a southern African East Coast deposit, contains typically 90% ilmenite, 5% Ti-hematite, 3% spinel (including chromite and magnetite) and % silicates by weight.[5] Ilmenite is considered due to its specific surface properties and sphericity. These properties give it an advantage compared to magnetite as it does not attach to the coal particles as much as magnetite does, consequently ilmenite also possesses hydrophobicity which is also desirable for the process.

Current published dry dense medium separation work has mainly focused on using magnetite as a medium, but various complications have been encountered when magnetite was used. This study will extend the approach by investigating the use of ilmenite as an alternative medium for this dry dense medium fluidization process.

EXPERIMENTAL PROCEDURE

Experimental Setup

The purpose of this project was to determine the minimum fluidizing velocity, bed composition of binary medium (ilmenite with fine coal and ilmenite with fine sand), bed density, Ecart Probable Moyen, recovery of the ilmenite and the yield with two types of coal.

The coal samples were dry according to air drying of coal sample ASTM D3303/D3302M-12, and screening was conducted to size the coal between 13 and 50 mm. A particle size distribution, as shown in Figure 1, was done on the medium (ilmenite, fine sand and fine coal) and the chemical analysis and SEM image were done on ilmenite. Table 1 and Figure 2 show the chemical analysis and the SEM image respectively.

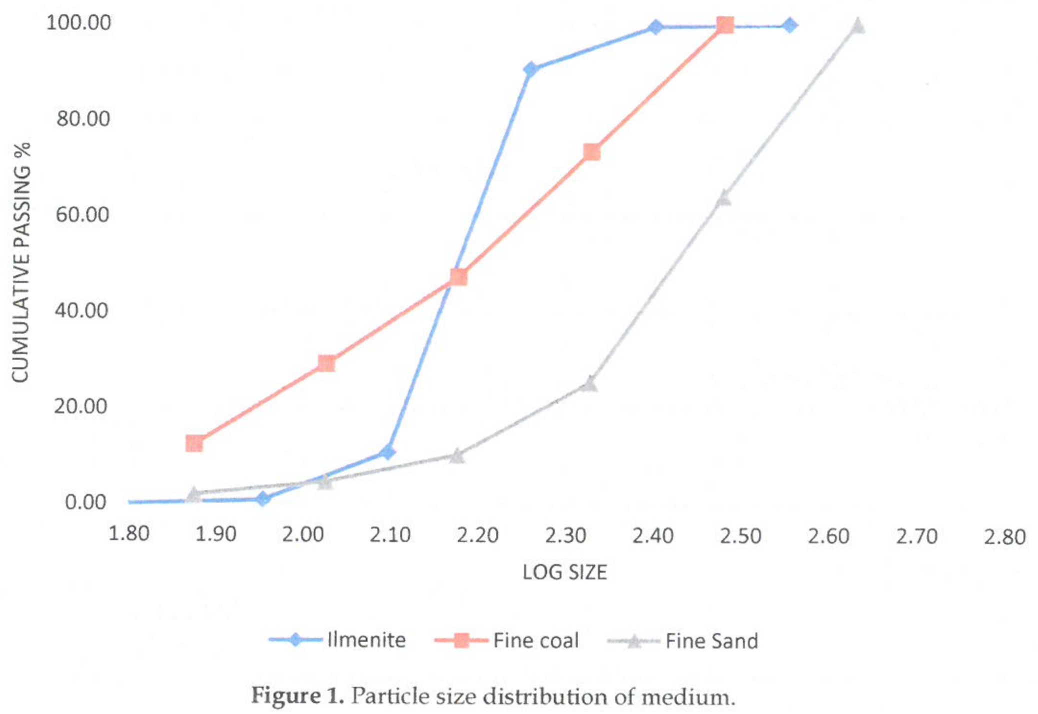


Figure 1. Particle size distribution of medium.

Table 1. XRD analysis of ilmenite.

High-grade ilmenite	Weight %
Actinolite	15.49
Rutile	2.85
Hematite	12.56
Ilmenite	63.79
Quartz	0.80
Srebodolskite	4.51

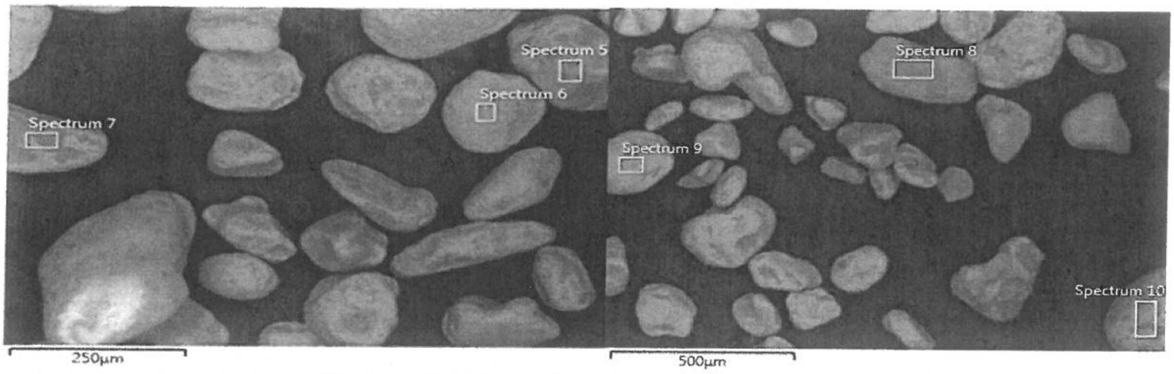
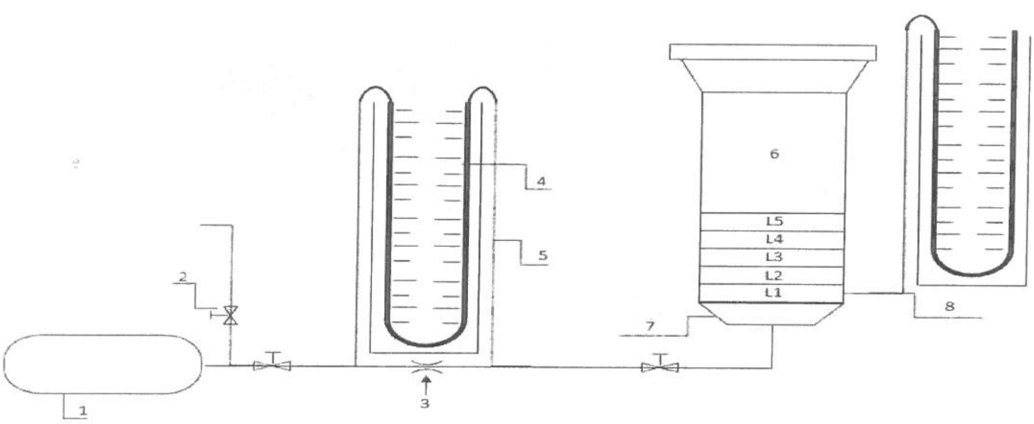


Figure 2. SEM image ilmenite.

The minimum fluidizing velocity (U_{mf}) of the binary medium was determined experimentally using a perspex fluidized bed and calculated using the Ergun equation:

$$\frac{1.75}{\phi_s \varepsilon_{mf}^3}\left(\frac{d_p u_{mf} \rho_g}{\mu}\right)^2 + \frac{150(1-\varepsilon_{mf})}{\phi_s^2 \varepsilon_{mf}^3}\left(\frac{d_p u_{mf} \rho_g}{\mu}\right) = \frac{d_p^3 \rho_g(\rho_s - \rho_g) g}{\mu^2} \quad (1)$$

The specific gravity range of the tracers used for the tests was between 1.3 to 3 g/cm^3; and for each density value there were ten tracers available. The tracers are made out of a magnetically susceptible material, they are of a cubic shape and with side dimensions of 12 mm. Ten minutes was permissible for the fluidized bed to stabilise, and the tracers were then gradually introduced onto the surface of the fluidized bed. After stratification for 30 seconds, the air bleed valve stream was abruptly close off and all the stratified tracers were retained in their positions in the mixture. A schematic drawing of an air dense fluidized bed is shown in Figure 3.



1. Compressed air; 2. Air bleed valve; 3. Orifice; 4. U-type manometer; 5. Pipe connector; 6. Perspex fluidised bed; 7. Distributor; 8. Bed pressure drop

Figure 3. A schematic air dense medium fluidized bed.

RESULTS AND DISCUSSION

Mixture of fine coal and Ilmenite

The experiment was first conducted using ilmenite as the medium; it was found that the bed split was at 3 g/cm^3. The observed split of ilmenite is too high for beneficiation of coal in the density ranging between 1.3 g/cm^3 to 2.3 g/cm^3. As a result, the fine coal (-300 +75 μm) or fine sand (-424+75 μm) was used to reduce the bed density. A binary medium of group B ilmenite with fine coal and ilmenite with fine sand were used to achieve the bed split required in the coal beneficiation industry, and the bed was found to be uniform and stable with no segregation taking place.

Fine coal or fine sand and ilmenite were mixed in the different ratios, and then used as a medium in the fluidized bed. The experiments were conducted at the bed height of 12 cm to prevent the slugging regime which takes place in beds with a bed height (H) over bed diameter ratio (D) larger than about 2. The results are shown in Table 2.

Table 2. Split results at different fine coal or fine sand/ilmenite composition.

Fine coal %	U_{mf} (cm/s)	Pressure Drop (cmH_2O)	Average Density (g/cm^3)	Observed Split (g/cm^3)	Fine sand %	U_{mf} (cm/s)	Pressure Drop (cmH_2O)	Average Density (g/cm^3)	Observed Split (g/cm^3)
0	3.0	26.0	2.94	3.00	0	3.0	26.0	2.94	3.00
10	2.8	24.5	2.78	2.70	10	3.2	24.0	2.78	2.75
20	2.5	24.0	2.62	2.50	20	3.4	23.5	2.62	2.60
30	2.0	22.0	2.27	2.30	30	3.4	23.5	2.47	2.45
40	2.0	19.5	2.05	2.00	40	3.5	21.5	2.31	2.30
50	2.1	17.5	1.83	1.80	50	3.6	21.0	2.15	2.10
60	2.0	15.5	1.60	1.58	60	4.2	18.5	1.99	2.00
70	2.0	11.5	1.38	1.40	70	4.6	17.0	1.83	1.80
80	1.7	09.0	1.16	1.20	80	4.8	16.0	1.67	1.65
100	1.5	05.5	0.71	0.70	100	5.2	13.0	1.36	1.35

Prediction of the tracers in an air dense medium fluidized bed

According to [6], the operating superficial gas velocity was found by using equation 2 for the transition from bubbling to slug flow, and it gives good agreement with most experiments.[6]

$$\frac{U - Umf}{0.35\,(gD)^{\frac{1}{2}}} = k \qquad (2)$$

When U - U_{mf} is larger than the value found from equation 2, the bed will be in the slugging zone, which is not desired. The operating superficial gas velocity used was 6 cm/s. From equation 2, this operating superficial gas velocity gave k values which were less than 0.2, which means the bed was operating in the bubbling region.

The static bed was divided into five layers which were three floats (L5, L4 & L3) and two sinks (L2 & L1); the tracers were discharged layer by layer using a scoop from top to bottom. Some of the results are shown in Figures 4 and 5.

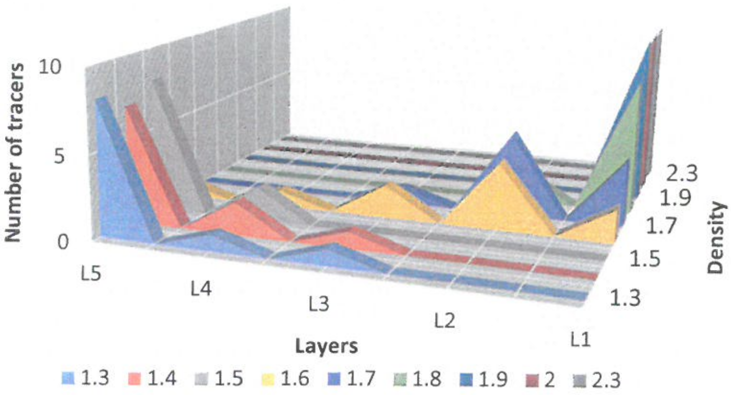


Figure 4. Binary medium of fine coal 60% and ilmenite 40%.

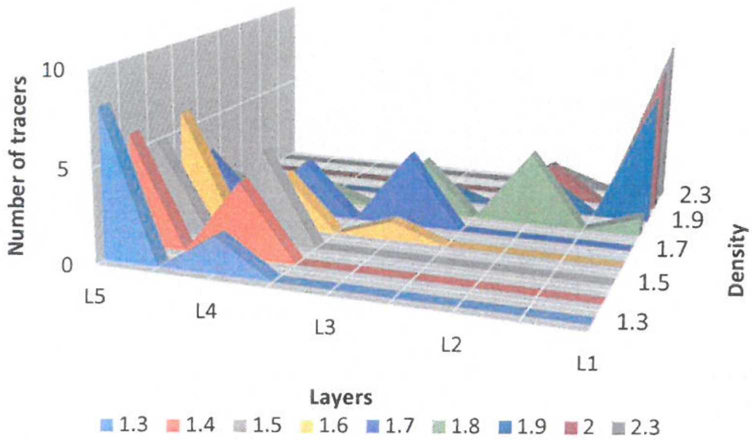


Figure 5. Binary medium of sand 70% & ilmenite 30%.

Partition curve

The tracers were used to determine the EPM of the fluidized bed. The tracer particles were stratified within the bed into floats and sinks, in the same way, which coal would be separated.[7]

The tracer particles recovered from the float and sink were sieved and counted, and the number of tracers recovered was then used to construct a partition curve. The Ecart Probable Moyen (EPM) in Table 3 was calculated using equation 3 and specific gravity 50% (SG50), it can be read from the partition curve as illustrated in Figure 6 and 7, which are derived from two randomly picked sets of data coming from a larger pool.

$$EPM = \frac{\rho_{25}-\rho_{75}}{2}$$
(3)

Napier-Munn[8] gives a good overview of the statistical calculations knowing the cut point at 50%, particle density and the EPM which correct the actual partition curve. As a result, the R^2 shows a much closer fit to equation 4.

$$Y = 1/(1 + \exp[1.099 * (\rho_{50} - \rho)/Ep\,]) \quad (4)$$

Table 3. Ecart Probable Moyen.

Fine coal %	Ecart Probable Moyen	Fine sand %	Ecart Probable Moyen
0	-	0	-
10	-	10	-
20	-	20	-
30	-	30	-
40	0.110	40	-
50	0.060	50	0.120
60	0.050	60	0.110
70	0.070	70	0.045
80	-	80	0.050
100	-	100	0.050

The empties in Table 3 show that the Ecart Probable Moyen of ilmenite with fine coal and ilmenite with fine sand. Table 3 indicates that, from 0 to 30 % ilmenite with fine coal, all tracers floated. Furthermore, the table shows that ilmenite with fine sand, tracers floated from 0 to 40%.

From 80 to 100% ilmenite, with fine coal, all the tracers did sink. Consequently we were unable to calculate the EPM.

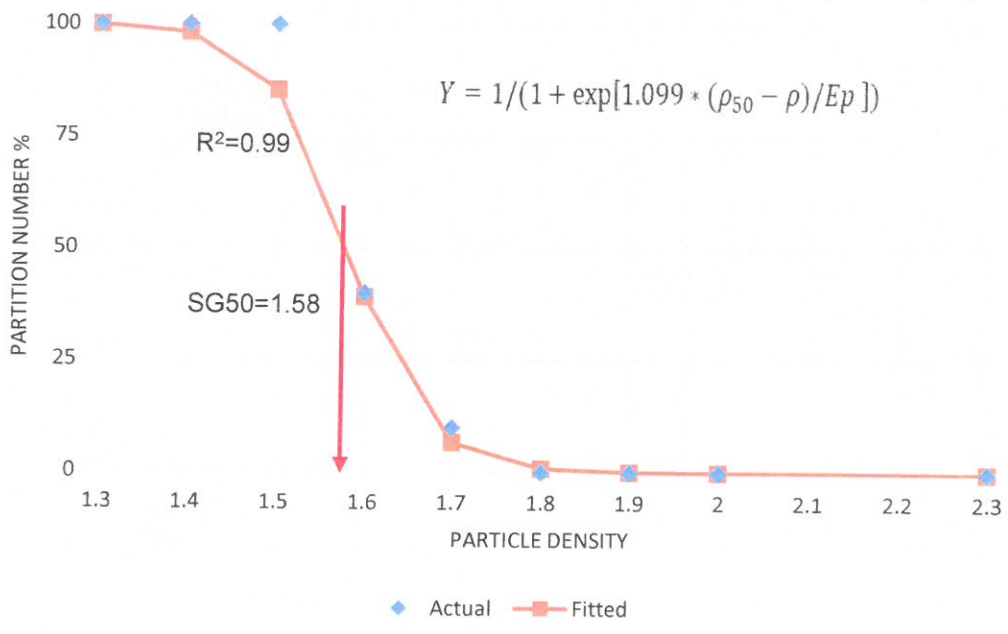


Figure 6. Partition curve of Binary medium of Fine coal 60% and ilmenite 40%.

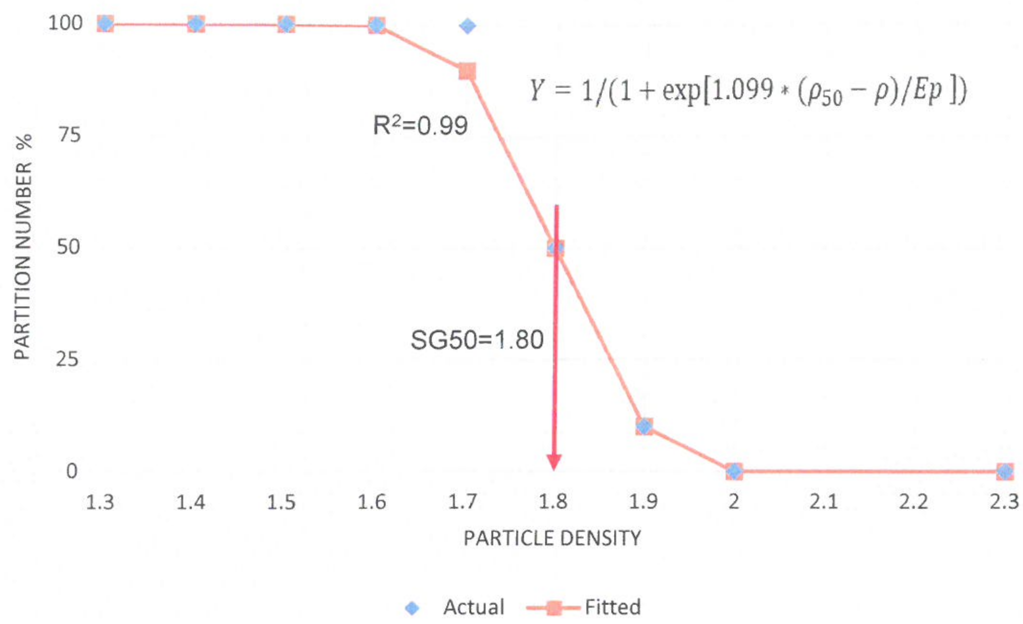


Figure 7. Partition curve of Binary medium of Fine sand 70 % and ilmenite 30%.

Stratification in the bed with different types of coal

The stratification in Table 4 proved to be satisfactory with the bed split observed at 1.58 g/cm³ of medium ilmenite with fine coal. The static bed was divided into two layers which are a float and sink; the coal particles were discharged from top to bottom and chemical analysis was conducted. Based on the result, heavier coal particles reported to the bottom quickly, leaving behind the coal lighter particles that remained in the top layer.

Since coal is lighter than the medium, the higher the density of separation the higher is the yield, as calculated using equation 5:

$$Yield = \frac{weight\ of\ coal\ floats\ product \times 100\%}{total\ feed\ weight} \quad (5)$$

Table 4. Stratification of Witbank coal.

Type of Coal	Height of bed	Ash%	Mass (g)	Yield %
Sample AFE	Float	11.00	159.74	60.06
	Sink	19.30	106.21	
Sample ROM	Float	13.80	200.21	74.86
	Sink	55.60	67.24	

The yield of samples AFE and ROM were found to be acceptable, which were 60.06% and 74.86%, respectively.

Recovery of ilmenite using a Dry High Gradient Magnetic Separator (HGMS)

Seven samples of coal were fluidized with the binary medium of ilmenite and fine coal and the mixtures were sieved for 10 min on a sieve size of 3.350 mm at amplitude 40 Hertz. A Dry HGMS of 4200 Gauss was used to recover the ilmenite and the Magna chute of 3150 Gauss, as shown in Figure 8, was used to clean the magnetic ilmenite recovered to achieve a real account of the medium recovered. It can be seen in Figure 9 that the dry coal 1 time dry coal had the highest recovery of ilmenite, and the lowest recovery was observed in the wet coal 4% respectively.

Figure 8. Magna chute.

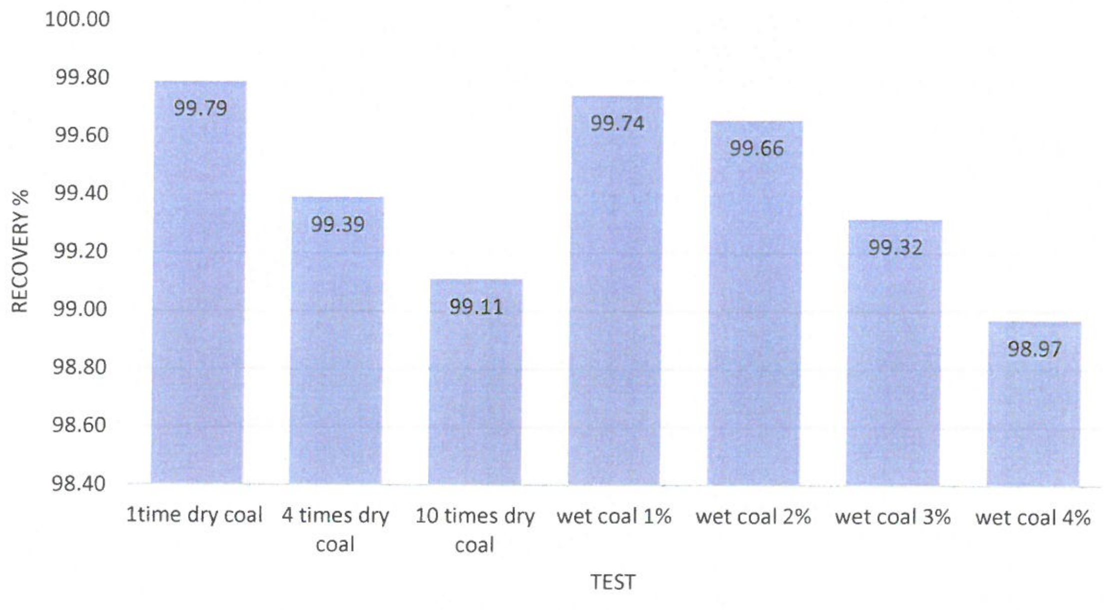


Figure 9. Recovery of ilmenite.

According to Figure 10, the lowest losses were found at 1 time dry coal and the highest losses of ilmenite were seen mostly at wet coal 4%. The 4 and 10 times dry coal were done by recycling the ilmenite into the process in respective to their numbers, using fresh coal after each cycle in order to see the effect of ilmenite surface. The ilmenite surface was found to be clean as in the initial one.

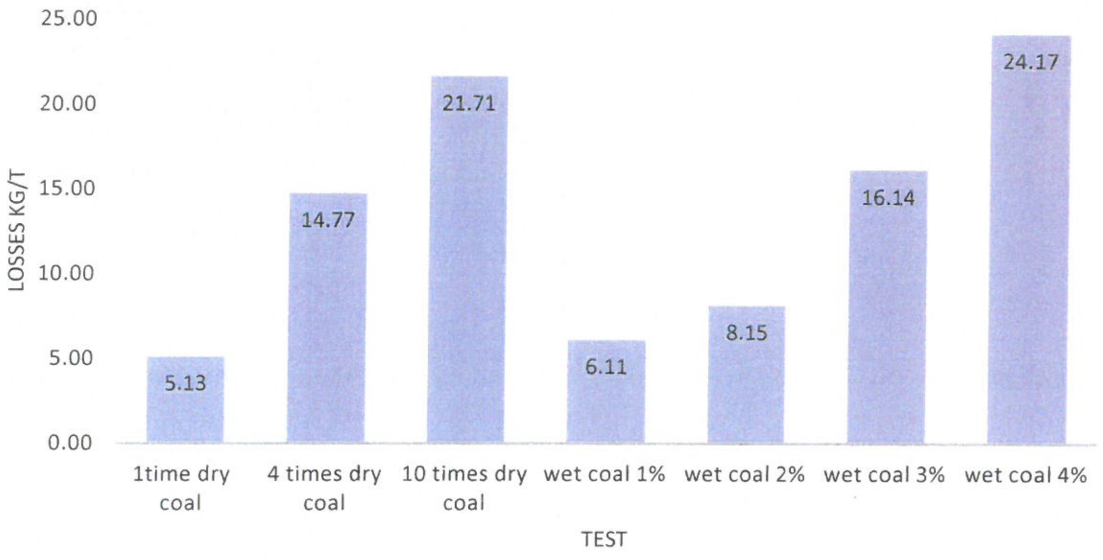


Figure 9. Losses of ilmenite.

CONCLUSION

The research has established the feasibility of dry coal beneficiation using a binary medium of ilmenite with fine coal and ilmenite with fine sand. A good Ecart Probable Moyen was achieved on the coal size -50+13 mm and the yield of both coals used were 60.06 and 74.86%, respectively. The ilmenite with fine coal medium were fluidized with both dry and wet coal, and the ilmenite was recovered using a dry HGMS. The losses of ilmenite was found to be lesser than 25 kg/t at the external moisture content of coal 4%.

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NOMENCLATURE

d_p	: Particle diameter d50, m
U_{mf}	: Superficial gas velocity at minimum fluidizing velocity, m/s
g	: Gravitational acceleration, m/s^2
D	: Bed diameter, cm
EPM	: Ecart Probable Moyen
HGMS	: High Gradient Magnetic Separator
Y	: Partition number
L	: Layer into the perspex fluidized bed
H	: Height of the bed
DMS	: Dense medium separation

Greek letters

ρ_{50}(SG50)	: Cut point density at 50%
ρ	: Particle density
ρ_{25}	: Cut point density at 25%
ρ_{75}	: Cut point density at 75%
ρ_g	: Gas density, kg/m^3
μ	: Viscosity of gas, kg/m.s
$\emptyset_s$	: Sphericity of a particle, dimensionless
ε_{mf}	: Void fraction in a bed at minimum fluidizing conditions
ρ_s	: Density of solids, kg/m^3

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