The practical characterisation of ferrosilicon for dense medium applications

J Bosman[1] and W Blair[2]

ABSTRACT

The measurement of ferrosilicon rheology and characterisation of the parameters that have a direct influence on medium rheology provide the basis for evaluating, comparing and selecting an appropriate size and shape of medium to maximise separation efficiency in a dense medium application. The role of medium viscosity on particle separation in both static and dynamic separators is illustrated using relevant theory. The primary measurements of rheology are medium viscosity and stability. These in turn are determined by particle size, particle density and particle shape which in turn can be quantified. Additional characterisation parameters are also discussed. Different areas of application where ferrosilicon characterisation can be used are also discussed.

INTRODUCTION

Ferrosilicon is used in dense medium suspensions for processing iron ore, manganese, diamonds, chromite, andalusite, tungsten and lithium ores to name just a few. It is also used as a preconcentration stage to reduce the tonnage that a plant will have to process.

The characterisation of ferrosilicon and the insights and understanding that it brings has the potential to improve the separation efficiency of these operations through the correct selection of ferrosilicon size and shape to provide the correct medium rheology.

THEORY

Dense medium separators

Medium characteristics that are important can be determined by considering the equations that drive particle separation for dense medium separators. A distinction must be made between static separators where only gravity plays a role in the separation and dynamic separators where multiple gravitational forces play a role.

Static separators

The most common static separator used in the iron ore industry is the WEMCO® drum. The following equations describe the forces acting on a particle in a static separator:

In the Stokes regime, the terminal or free settling velocity of spherical particles in fluids of known density and viscosity can be described by the following equation as shown in Furstenau (2003):

$$v_t = \frac{\mu}{\rho_f d} 10^A \tag{1}$$

where:

v_t is the terminal or free settling velocity
μ is the fluid/medium viscosity
ρ_f is the fluid/medium density
d is the particle diameter

A is the Archimedes number which is given by the following equation:

$$A = 5[0.66 + 0.4\log(4/3Ga)]^{0.5} - 5.55 \tag{2}$$

Ga is the Galileo number which is given by the following equation:

$$Ga = \frac{gd^3\rho_f(\rho_s - \rho_f)}{\mu^2} \tag{3}$$

where:

g is the gravitational constant

Particles do not reach their free falling or terminal settling velocity instantaneously, but in fact experience an initial period of acceleration until the terminal velocity is achieved.

The particle velocity as a function of time can be described by the following equation:

$$v = v_t[1 - exp(-kt)] \tag{4}$$

where:

v is the particle velocity at a given point in time
k is given by the following equation

1. Principal Consultant, PESCO, Centurion 0157, South Africa. Email: jeremy@pesco.za.net

2. Managing Director, Ferroz, Perth WA 6009. Email: will@ferroz.com.au

$$k = \frac{18\mu}{d^2 \rho_s} \quad (5)$$

The fluid/medium viscosity term μ occurs in Equations 1, 3 and 5 demonstrating the important role that viscosity plays in particle separation.

Dynamic separators

The most commonly used dynamic separators are cyclones. The simplest model for particle motion in a dynamic separator such as a cyclone is given by the equilibrium orbit theory as described in Wills (2015):

$$d_{50} = k\left[\frac{D_c^3 \mu}{Q_f(\rho_s - \rho_l)}\right]^n \quad (6)$$

where:

d_{50} is the cut size
ρ_s is the particle density
ρ_l is fluid/medium density
Q_f is the feed flow rate
D_c is the cyclone diameter
μ is the fluid/medium viscosity
k is a constant
n is a hydrodynamic constant

The viscosity term is highlighted in Equation 6, showing the important role of viscosity in particle separation in dynamic separators as well.

Dense mediums

Medium viscosity

For dilute suspensions of solids and liquids, the relative viscosity of the suspension can be described by the Einstein equation as shown in Mueller, Llewellin and Mader (2010):

$$\mu_r = 1 + B\varphi \quad (7)$$

where:

μ_r is the relative viscosity
B is the intrinsic viscosity (typically 2.5)
φ is the volume fraction of solids in the suspension

For higher concentration suspensions Krieger and Dougherty derived the following relationship as shown in Mueller, Llewellin and Mader (2010):

$$\mu_r = \left(1 - \frac{\varphi}{\varphi\, max}\right)^{-B\varphi max} \quad (8)$$

With φ_{max} the maximum volume fraction of solids or packing density that can be achieved

Maron and Pierce in Mueller, Llewellin and Mader (2010) simplified Equation 8 by setting the term $B\varphi_{max}$ to equal two resulting in:

$$\mu_r = \left(1 - \frac{\varphi}{\varphi\, max}\right)^{-2} \quad (9)$$

ie the relative viscosity is only a function of the volume fraction of solids in the suspension and the maximum volume fraction solids or packing density.

From Equation 9, the following qualitative relationships can be derived:

- increasing the solids volumetric concentration leads to an increase in relative viscosity
- increasing maximum volume fraction of solids or packing density leads to a decrease in relative viscosity.

Maximum volume fraction of solids or packing density is determined by several factors:

- *Fines and size distribution* – for a bimodal distribution, the relative proportion and size of the two size fractions impacts on the maximum packing density. An experiment was conducted with steel shot where 3.14 mm particles were mixed with a range of smaller particles in different proportions and the maximum packing density measured as reported in Chang, Wang and Ge (2015). The results are shown in Figure 1. As the fines increase, the packing density increases until the voids within the larger particles are filled. Increasing the fines further results in the larger particles being pushed apart and the packing density starts decreasing.

  The ratio between the diameter of the larger particles and the smaller particles also has an impact on the maximum packing density. The greater the ratio, the higher the maximum packing density until a limit is reached.
- *Particle shape* – the impact of particle shape is shown in Figure 2. As particle become more round, the maximum packing density increases.
- *Particle size distribution* – the packing densities of different size fractions with increasing average particle sizes (as measured using a Malvern Laser Sizer) were measured Parteli *et al* (2014). The results are shown in Figure 3. The finer the size, the lower the packing density. As the size distribution becomes finer, the maximum packing density decreases.

Medium stability

If the fluid/mediums that are used for dense medium separation were homogeneous, then it would only be necessary to measure the viscosity of different density fluid/mediums in order to understand their impact on particle

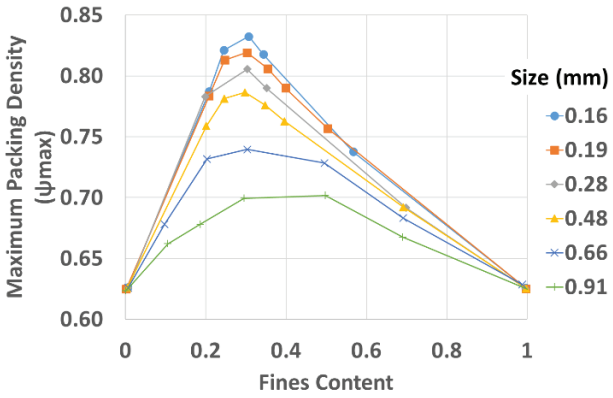

FIG 1 – Impact of fines on maximum packing density.

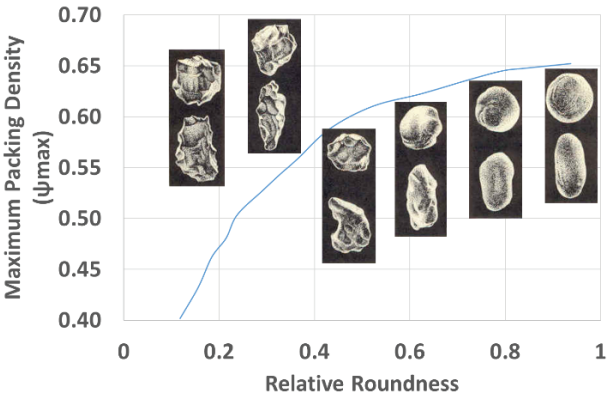

FIG 2 – Impact of particle shape on packing density.

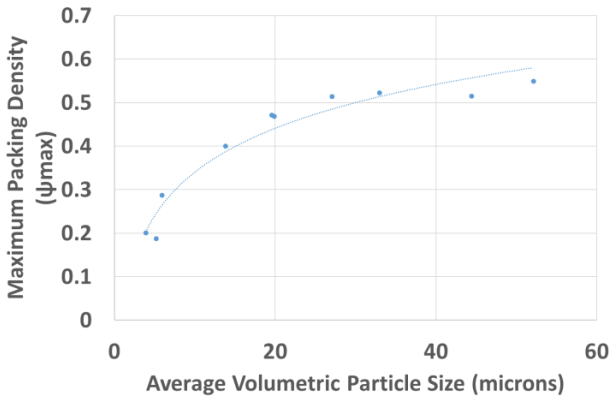

FIG 3 – Impact of particle size on packing density.

separation within the dense medium separators. However due to the lack of suitable homogenous mediums, heterogeneous suspensions of water and high density solids are commonly used to make up medium suspensions of varying density. As the density of the particles being used (ferrosilicon, magnetite or a blend of both) is greater than the liquid medium in which they are suspended (water), the particles settle out of the liquid over time.

The phenomenon of particles settling out of a liquid/solid suspension is referred to as stability and describes the rate at which particles settle in a stagnant medium under the influence of gravity.

The Richardson and Zaki equation as reported in Baldock *et al* (2004) can be used to describe the settling velocity of spherical particles from a suspension.

$$w = \frac{d^2(\rho_s - \rho_{liquid})g}{18\mu_{liquid}}(1-\varphi)^N \quad (10)$$

where:

w is the settling rate

ρ_{liquid} is the liquid density

μ_{liquid} is the liquid viscosity

N is a parameter related to both the flow regime and particle shape.

All other symbols have the same definitions as previously stated.

The parameters that drive medium viscosity and stability can be extracted from Equations 9 and 10 and are shown in Table 1.

PARAMETERS FOR THE CHARACTERISATION OF FERROSILICON

Medium rheology

Medium rheology refers collectively to both medium viscosity and medium stability.

TABLE 1
Medium rheology parameters.

Particle parameter	Medium viscosity (Equation 9)	Medium stability (Equation 10)	Particle parameter
ρ_s (particle density)	φ	φ	ρ_s (particle density)
d (particle size)	φ_{max}	d	d (particle size)
particle shape		N	particle shape

From Table 1, the parameters to be measured can be grouped into two categories:

1. Particle related parameters:
   - d – particle size
   - ρ_s – particle density
   - particle shape
   - φ_{max} – maximum packing density
   - surface area.
2. Medium related parameters – for a given φ (volume fraction of solids), the medium rheology can be determined by measuring:
   - μ_{medium} – medium viscosity
   - w – settling rate.

Additional characterisation parameters

Additional parameters that provide insight into the medium being characterised can be grouped into the following three categories:

1. Medium composition:
   - chemical analysis
   - homogeneity
   - non-magnetics.
2. Magnetic recovery:
   - magnetic susceptibility.
3. Corrosion:
   - corrosion rate.

PARTICLE PARAMETERS

As discussed above, the three main particle parameters are particle size, particle density and particle shape.

Particle size

Particles are three-dimensional objects and the particle size measurement that is produced is determined by the sizing technique used. There are two main techniques used for medium characterisation ie sieving and laser sizing.

Sieving

Sieving relies on particles passing through or being retained on a sieving mesh with square apertures. The aperture size only describes two of the three particle dimensions.

Laser sizing

Laser sizing measures the volumetric equivalent of a three-dimensional particle.

Sieving and laser-based comparison

Theoretically for a spherical particle, laser based and sieve based measurements will give the same dimension. For non-spherical particles, laser based sizing should always result in a coarser size than sieve based sizing. This can be demonstrated by considering a three-dimensional particle that has dimensions of 100 × 100 × 400 microns.

The particle will pass through a square aperture of 100 × 100 microns and will be deemed to have a dimension of 100 microns in spite of the fact that the third dimension is 400 microns.

The volume of the particle can be calculated by multiplying length × breadth × height:

$$Volume_{particle} = 100 \times 100 \times 400 = 4000000 micron^3$$

The diameter of a sphere is given by:

$$Volume_{sphere} = \frac{\pi d^3}{6}$$

Therefore, the diameter of a sphere with a particle volume of 4 000 000 micron^3 is:

$$d = \left(\frac{4000000 \times 6}{\pi}\right)^{\frac{1}{3}} = 197microns$$

Laser based measurements typically result in coarser size distributions than those measured with sieve sizing. For spherical particles, the two methods should give the same results but this is rarely the case.

Various attempts have been made to correlate laser sizing with sieve sizing. For a consistent particle shape it is possible to obtain a correction factor for a given particle size distribution, but any shape changes will change the correction factor.

Practical sizing

Ferrosilicon is supplied bone dry by the various manufacturers and lends itself to dry screening using sieving technology. Critical parameters are initial mass and screening time. In addition, it is important to use screening aids to deagglomerate the material.

For a 200 mm diameter sieve, an initial mass of 200 g and a screening time of 30 minutes provides consistent results for a stack of six screens from 300 to 45 microns.

Sometimes knowing the percentage passing 45 microns is all that is required especially for quality assurance. It is possible to do a 'Quick 45' using 25 g and a screening time of 20 minutes.

The minus 45 micron fraction is traditionally used to differentiate different ferrosilicon grades, but the finer fractions play an important role in rheology. An alternative to laser sizing for the finer fractions is the air jet sieve.

A range of sieve apertures are available, but the 20 micron aperture provides a practical size that can be measured and the size measurement is based upon the same principal as sieving so the problems of size measurements with different meanings is avoided.

Particle density

Primarily particle density is indicative of the chemical composition of ferrosilicon which is a metal alloy. With 14 to 16 per cent silicon and more than 80 per cent iron, densities can range from 6.7 to 7.1 t/m^3. A secondary effect of particle density is its contribution to φ, the volume fraction of solids in a medium suspension. The lower the particle density, the higher the volume fraction required for a given medium density and the higher the resultant medium viscosity.

One of the more accurate determinations of particle density is done by using helium pycnometery. A known mass of sample is placed in a container of known volume. The amount of volume not occupied by the sample is measured using helium gas and the volume of the sample can then be calculated. With a known sample mass and volume, the sample density can then be calculated.

Particle shape

Although there are many measurement techniques to determine particle shape, ferrosilicon shape is normally classified according to the shape that the manufacturing process produces. The following three categories currently exist:

1. angular – produced by milling
2. low sphericity – produced by water atomisation
3. high sphericity – produced by gas atomisation.

The three categories are shown in Figure 4.

Maximum packing density

The maximum packing density is determined by filling a mass of sample into a chamber with a known volume. Once the chamber is full the mass of sample is measured and the freely settled or poured bulk density is calculated. Note that the chamber can be tapped to compact the sample and in this case a tapped bulk density will be measured. What is important is not to mix results from freely settled and tapped measurements.

Surface area

The specific surface area of a sample provides an indirect indication of size distribution. For a mono sized particle, a smaller particle will have a larger specific surface area compared to a larger particle. A sample with a narrow size distribution will have a smaller specific area compared to one with a wider size distribution.

The specific surface area is measured using a Blaine apparatus. In essence, the apparatus measures the pressure drop across a packed bed and uses the Karman-Cozeny equation to calculate the specific surface area as discussed in Arvaniti *et al* (2015). Cement samples of known specific surface area are used for calibration purposes.

MEDIUM RHEOLOGY

Viscosity

Viscosity is defined as the relationship between shear rate and shear stress and a plot of shear rate versus shear stress for various types of fluids is shown in Figure 5. Newtonian fluids are defined as those that have a linear relationship between shear stress and shear rate. For some fluids a certain amount of stress (yield stress) must be applied before they will start shearing. These fluids are called Bingham plastics. Fluids that do not exhibit a linear relationship are called non-Newtonian and their viscosity is a function of shear rate.

As previously discussed, medium suspensions settle under the influence of gravity which complicates viscosity measurement as viscosity measurement must be done at a constant density to produce meaningful results.

A Stormer viscometer was modified by De Beers in the early 1980s. The medium suspension was pumped past the measuring bobbin to counteract the settling of particles. Further development of this unit was done by the Julius Kruttschnitt Mineral Resource Centre (JKMRC) as discussed in Shi and Napier-Munn (1996).

Programmable rotating vane rheometers provide a simpler solution where the vane is initially used to suspend the particles and then to measure the yield stress at a given shear rate for a limited period of time to minimise the effect of particle settling on the results. Provided the same procedure

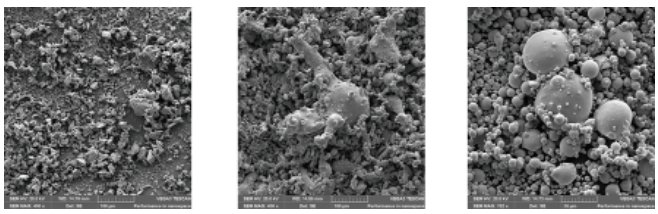

FIG 4 – Ferrosilicon shape – milled, water atomised and gas atomised.

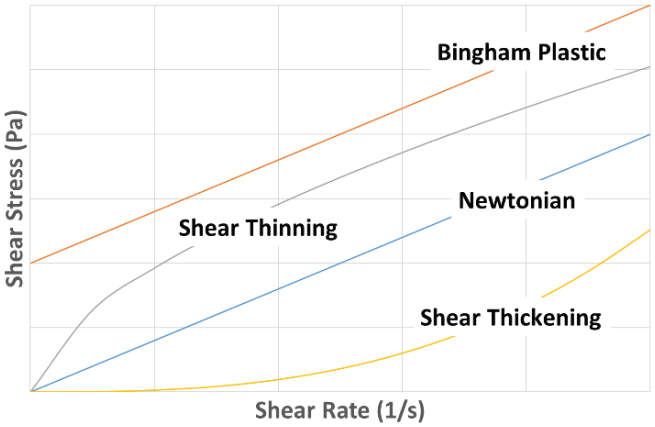

FIG 5: Shear rate versus shear stress plot.

is followed for each test, the results are internally consistent and allow the viscosity of different mediums to be compared. The result for a milled ferrosilicon for three different densities is shown in Figure 6.

The complex Non-Newtonian behaviour of medium suspensions is immediately apparent and the relative increase in apparent viscosity with increasing density (volume fraction of solids) is also apparent.

Stability

Stability measures the propensity of the solid fraction of a solid liquid suspension to settle out from the liquid fraction. This is an important parameter for both static and dynamic separators. If the medium is too unstable (the solid fraction settles out very quickly), a range of densities is created within the separator which adversely affects the separation performance of the unit. On the other hand, if the medium is too stable (the solid fraction settles out very slowly), the separation performance of especially finer particles can be adversely affected. The optimum stability range is different for each combination of medium density and separator configuration.

The measurement of stability is extremely simple and provides a valuable tool for on-site rheology measurement. A medium suspension at the required density (volume fraction) is made up in a measuring cylinder. Both 250 ml and 500 ml measuring cylinders are commonly used. The cylinder is inverted a number of times to ensure the suspension is well mixed and the mudline (interface between the solids and liquid) is measured as a function of time. The position of the mudline is then plotted against time and the slope of the linear portion of the graph is the settling rate expressed in mm/s. In the literature, a stability value is also used and it is the inverse of settling rate expressed in s/cm.

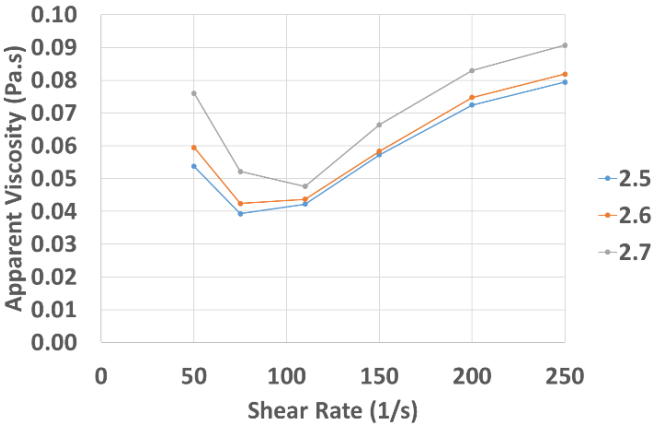

FIG 6 – Apparent viscosity shear rate plot.

Medium composition

Chemical analysis

Chemical analysis on ferrosilicon can be done using either X-ray fluorescence or inductively coupled plasma optical emission spectroscopy (ICP-OES) and must be done by an accredited laboratory.

There are two main elements of interest ie Si and Fe. For maximum corrosion resistance and minimum impact on particle density, silicon must be in the range of 14–16 per cent. The Fe value must be greater than 80 per cent

Homogeneity

Homogeneity refers to the number of individual particles that have 14–16 per cent silicon. The accepted standard is 85 per cent minimum ie 85 per cent of the number of particles in a sample by will have 14–16 per cent silicon. A scanning electron microscope with energy dispersive X-ray spectroscopy (SEM/EDX) can be used to perform this test. Care must be taken to ensure that the number of particles counted is representative. This test is seldom done.

Non-magnetics

The percentage of non-magnetics is important because it will be lost the first time the ferrosilicon passes over a magnetic separator. It is measured using a Davis Tube operating at 1000 Gauss (similar to the low intensity magnetic separators used in a dense medium circuit). Values typically should be less than three per cent.

Magnetic mass susceptibility

Magnetic susceptibility is the degree of magnetisation that occurs in a particle that is exposed to an external magnetic field (a low intensity magnetic separator in the case of dense medium circuits). Dense medium circuits rely on magnetic separators to recover ferrosilicon for reuse. Low magnetic susceptibilities will adversely affect the ferrosilicon recovery and lead to higher operational costs. The units of mass susceptibility are m^3/kg.

A Magnasat, which uses an alternating current magnetic field to measure magnetic susceptibility is used. The device is calibrated using a calibrant. The magnetic susceptibilities of ferrosilicon can vary from 1.6 to 2.1 x 10^{-4} m^3/kg. Work still needs to be done to correlate these values to losses over a magnetic separator.

Over time some residual magnetism remains in the calibrant and the value of the changes. A demagnetisation coil is used to remove this residual magnetism and restore the value of the calibrant.

Corrosion rate

Ferrosilicon corrodes and in the presence of oxygen passivates to form a protective layer which prevents further corrosion. In an operating dense medium circuit, ferrosilicon is continuously exposed to air which maintains the passive layer.

When all the oxygen surrounding the ferrosilicon is consumed eg when ferrosilicon is dumped on the floor, it goes into active corrosion. The main chemical reaction is:

$$Fe_3Si + 9H_2O \rightarrow 3Fe(OH)_2 + SiO_2 \cdot H_2O + 5H_2$$

One mole of ferrosilicon will generate five moles of hydrogen.

The standard corrosion test measures the volume of hydrogen produced over time from a sample of ferrosilicon at 80 degrees centigrade.

One of the problems with this test is that finer size distributions produce hydrogen at a faster rate compared to coarser size distributions. From the Blaine measurements, the specific surface area increases with particle fineness. To eliminate the size effect, the hydrogen evolution volumes were converted to mg/cm^2/day using the chemical reaction and surface area obtained from the Blaine measurement. The results are compared in Table 2. While the finer sample generate more hydrogen, the calculated mass loss due to corrosion for the two samples is similar.

APPLICATIONS OF FERROSILICON CHARACTERISATION

Quality control

Using the Quick 45, particle density and magnetic susceptibility measurements, quality assurance on large batches of samples can be done in a relatively short time. For example, 72 samples were completed in a two day period providing rapid feedback.

Evaluate different suppliers

The full suite of tests provides all the required information to evaluate the product supplied from different suppliers and their suitability for a given application.

Optimising blends

In certain cases, magnetite/ferrosilicon blends are required to provide a medium with the required rheology. In other cases, blends of different shapes of ferrosilicon have potential to improve rheology ie stabilising an unstable medium by blending with a medium with a finer size distribution or different shape. The viscosity and stability tests are particularly appropriate for this purpose.

TABLE 2
Comparative corrosion results.

Sample ID	Minus 45 microns (%)	Specific surface area (cm^2/g)	Average hydrogen evolution (ml/mg/day)	Ferrosilicon loss (mg/cm^2/day)
A	69.7	884	0.028	0.051
B	78.2	988	0.031	0.049

Medium comparisons

The rheology of different size distributions and shapes of medium can be evaluated over a range of medium densities (volume fraction of solids) to gain insight into their rheological response and suitability for different applications.

Research and development

One example is using a combination of coarse and fine size distributions to reduce viscosity by increasing the maximum packing density while maintaining medium stability.

Modelling

The characterisation data can be used to model both the settling rate using the Richardson and Zaki equation. Modelling of the viscosity holds potential using the simplified viscosity Equation 9 but presents challenges due to the non-Newtonian nature of ferrosilicon suspensions.

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