ELECTROMAGNETIC DETECTION OF IRON TRACERS FOR MONITORING PARTICLES DISPLACEMENT IN GRAVITY SETTLERS

Leclerc, A[1*], Boivin, A[1], Simard, G[1], Morin, L[1], Gagnon, M J[1], Verreault, R[1], Peloquin, G[2]

[1] Groupe de recherche universitaire en séparation solide-liquide,
Université du Québec à Chicoutimi, Québec, Canada
[2] Alcan International Limitée, Québec, Canada

Abstract

Knowledge of the residence time distributions of solid particles through gravity settlers of the Bayer's process is essential to verify that they are operated at an adequate level of effectiveness. For instance, residence times may be calculated using various mathematical simulations, but their actual measurement is difficult. The settling behaviour through the settlers could be analysed using these distributions, if they were available, and different physical parameters, such as residence times, could be measured. A novel electromagnetic inductance measurement method using iron particles as ferromagnetic tracers is presented here. In this method iron particles of a known size distribution are injected with the slurry in the feed well and are monitored in the carrying red mud as their presence modifies the magnetic permeability detected inside an inductive coil located in the underflow pipe. The inductive coil must be uniformly wound with great care and a frequency stabilized generator is required. The apparatus was tested in an industrial settler of an Alcan alumina refinery.

1　Introduction

The alumina required for making aluminium is extracted from bauxite by the Bayer process. Because of the decrease in the quality of the available bauxite, larger volumes of bauxite have to be processed to satisfy the needs of the aluminium smelters. Larger or better equipment is thus required to process these larger volumes of bauxite. In this context, optimizing the actual equipment becomes crucial. Solid liquid separation, which requires settlers that must process more and more volume at a faster rate, is one stage of the Bayer process where gains would be desirable. An apparatus to diagnose the running order of settlers would be very useful in achieving this goal. To the authors' knowledge no such diagnostic apparatus is presently available.

Using tracers is a widespread technique to diagnose the behaviour of continuous flow chemical reactors (Levenspiel, 1972; Fogler, 2002). The reactor residence time distribution, from which many physical parameters can be computed, is obtained by measuring the tracer at the reactor exit. These parameters can be used to diagnose the reactor behaviour. To be applicable to settlers, this technique requires an easily measurable tracer that is compatible with the physical and chemical environment of the settlers. The tracer must also be environmentally acceptable. This tracer technique has been used previously to simulate liquid flow in settlers (Grocott and McGuiness, 1990; Harrison et al., 1987) and mud behaviour in thickeners (Evans, 1982).

In this paper iron particles of a known grain size distribution have been used to simulate the sedimentation of a given class of solid particles in settlers and to simulate the mud displacement at the bottom of settlers. Because of its large magnetic permeability, iron is accessible to measurement with electromagnetic techniques. Moreover iron shows a good chemical resistance for the time span required to measure the residence time distribution in settlers. Iron is not particularly toxic and may be easily disposed with the mud after usage. The tracer is detected by measuring the root mean square (RMS) voltage across a resistor R connected in series to an inductor L in an electrical circuit. The inductor of this RL branch is a very carefully wound solenoid with a single uniform winding. An a.c. voltage generator with stabilized frequency is required. Calibration curves of the RMS voltage across the measuring resistor as a function of the quantity of iron inside the solenoid have been recorded. The apparatus and technique have been successfully tested in an experimental mud circulation loop in our laboratory and in an industrial settler of an Alcan alumina refinery.

2　Theoretical aspects

In tracer techniques two theoretical aspects must be considered: how the tracer simulates a particular target substance in the reactor and how the tracer is detected and measured at the exit of the reactor.

In this paper the emphasis is on the displacement of the residual bauxite solid particles through the settler. Particle settling velocity is a very complicated issue but Stoke's law provides a basis to simulate the displacement of solid particles through a liquid under the action of gravity. The free settling velocity of spherical particles due to gravity is given by

$$v = (\pi\, d^2/18\, \eta)(\rho_p - \rho_l) \qquad (1)$$

where d is the particle diameter, η is the liquid viscosity and where ρ_p and ρ_l are respectively the densities of the particle and the liquor. The iron tracer particle must have the same free settling velocity as the simulated solid bauxite residue particle. This condition gives the ratio of the tracer particle to the solid particle diameter:

$$(d_p / d_{Fe})^2 = (\rho_{Fe} - \rho_l)/(\rho_p - \rho_l) \qquad (2)$$

where the subscripts p, Fe and l refer to the solid particle, the iron tracer particle and the liquor. Taking 3000, 7900 and 1100 kg/m³ as the densities of bauxite residue, metallic iron and Bayer liquor, this last equation yields $d_p \approx 1.9\, d_{Fe}$. A shape factor is often incorporated into Stoke's law to account for non spherical particles. A tracer particle of a given shape would then simulate a mud particle of the same shape. Densities of flocculated aggregates are less than those of non flocculated particles. Flocculation would thus increase the above ratio. Settling in the denser zone near the mud bed is hindered and this effect was not specifically taken into account. The movement of the mud at the bottom of the settler towards the underflow is controlled by the action of the rake. Although compaction occurs vertically through the mud bed, it seems acceptable to simulate the movement of the mud under the action of the rake as a single phase movement. Under such conditions no scaling factor is required to describe the movement of the tracer particle with respect to the bauxite residue particle in the mud bed.

The electromagnetic measurement of the iron tracer particle makes use of the dependence of a solenoid self-inductance L upon the magnetic permeability of the medium inside the solenoid (Borody, 1985):

$$L = K \, \mu_r \, \mu_0 \, N^2 \, \pi \, r^2 / L \qquad (3)$$

where K is the geometrical factor of the solenoid, μ_r is the relative permeability of the medium inside the solenoid, μ_0 is the magnetic permeability of vacuum, N is the winding turn number, and r and L are the internal radius and the length of the solenoid. Let x_i be the fractional volume of the solenoid filled with the ith magnetic substance, then the relative permeability of the medium inside the solenoid is

$$\mu_r = \Sigma \, x_i \, \mu_i \qquad (4)$$

The sum of the fractional volumes equals unity. The fractional volume of the ith magnetic substance is related to the mass m_i of substance inside the solenoid, to its density ρ_i and to the internal volume V of the solenoid:

$$x_i = m_i / \rho_i V \qquad (5)$$

In this way the self-inductance of the solenoid is related to the mass of magnetic substances that it contains. The reactance X_L of the solenoid is directly proportional to its inductance:

$$X_L = j \, 2\pi \, f \, L \qquad (6)$$

where f is the frequency of the voltage generator and j is the imaginary unit.

3   Experimental set-up and apparatus

Because the reactance of a solenoid is proportional to the applied frequency, high frequencies increase the inductive effect of a solenoid. The voltage generator should then oscillate at a high frequency, but too high a frequency complicates the electrical circuit. Because any variation in the reactance is ascribed to a variation in the mass of the magnetic substances inside the solenoid, the frequency of the generator must be very stable. Such a voltage generator has been conceived and assembled in our laboratory. The output voltage of the generator was monitored with an Agilent 34401A multimeter for more than twenty hours to detect any drift in the output voltage. To make available the most sensitive voltage scale of the multimeter ($\pm 1 \, \mu V$), the source output voltage was read through a voltage divider in an approximate ratio of 10 to 1 using a 100 Ω and a 1000 Ω ($\pm 1\%$) resistors connected in series. The recorded a.c. signal is shown in Figure 1. A voltage drift of approximately 0.3 mV, which corresponds to a relative drift of 0.08% is observed. Although small, this drift has to be taken into account so as to minimize the limit of detection of the iron detecting circuit. To achieve this purpose, a reference branch has been incorporated into the iron detecting circuit. The short oscillations ($\pm 10 \, \mu V$) are caused by electronic noise and give the ultimate detection limit of an apparatus using this voltage generator.
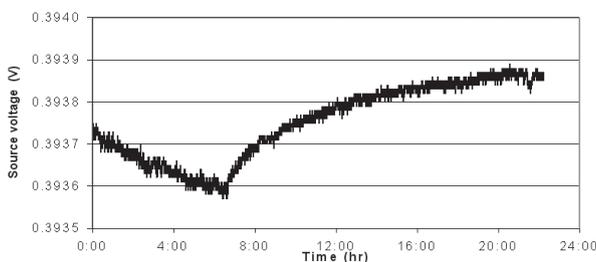

Figure 1. Voltage drift of the voltage generator.

Figure 2 shows the iron detecting electrical circuit. It consists of the voltage generator energizing a reference branch connected in parallel with a measurement branch. The reference branch is used to monitor any variation in the output voltage of the generator and any change

in the ambient magnetic field that is expected to occur in an industrial environment. The two loads, R_{ref} and Z_{ref}, act as a potential divider in an approximate ratio of 10 to 1, allowing the potential across the resistor R_{ref} to be read on the most sensitive scale of the Agilent multimeter. The resistor R in the measurement branch serves the same purpose. The potential across this measurement resistor R is read with a second Agilent 34401A multimeter. Two versions of the reference branch have been used. For calibration purposes carried out in the stable magnetic environment of our laboratory, R_{ref} and Z_{ref} are simple resistors. For monitoring purpose in the industrial environment, Z_{ref} becomes a solenoid. In the measurement branch, the solenoid has a d.c. resistance RL and an inductance L. The solenoids have been wound with insulated copper wire. The temperature coefficient of resistivity of copper, α, is 0,0038 K^{-1}. This is large enough for ambient temperature variations to have an observable effect on the measured voltage across the resistor R. A thermocouple monitoring any temperature variation has thus been fixed to the solenoid in the measurement branch. The red mud circulates inside this solenoid.
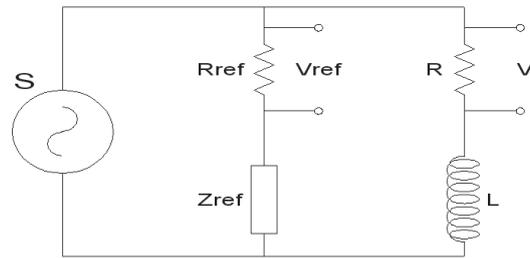

Figure 2. Schematic of the iron tracer detecting circuit.

The data from the multimeters have been recorded with two personal computers using the HP-IB Data Acquisition Protocol. To eliminate all variations in the measured signal due to the source variation, the data from the two branches must be recorded simultaneously. The simultaneity of data recording is not sufficient to eliminate fast oscillation, such as the electronic noise. A data averaging technique is used to decrease the influence of this electronic noise. The ultimate detection limit is taken to be three times the amplitude of the electronic noise of the voltage generator, i.e. $\sim 30 \, \mu V$. The detection limit of the actual apparatus is somewhat worse than this. The data from the thermocouple have been recorded at the same rate as those from the multimeters. All data were mathematically processed with a Microsoft Excel work sheet.

4   Calibration

Any voltage variation V_{ref} due to the source must be subtracted from the voltage V read across the resistor R. Because the potential is not exactly divided between the reference and measurement branches, a correcting factor, Q, must be applied to the reference voltage before subtraction:

$$Q = [\, R \, (\, R_{ref} + Z_{ref} \,) \,] / \{ \, R_{ref} \, [\, (\, R_L + R)^2 + (\, 2\pi \, f \, L)^2 \,]^{1/2} \} \qquad (7)$$

This yields a value of Q = 1.001 for the calibration circuit. The measured voltage compensated for the voltage source variation is then

$$\Delta V = V' - Q \, V_{ref} \qquad (8)$$

where V' is the voltage read across the measurement resistor R corrected for thermal variation induced by the solenoid. An increase in the solenoid temperature increases slightly the resistance R_L of the copper wire of the winding copper wire, decreasing the measured voltage V and the factor Q.
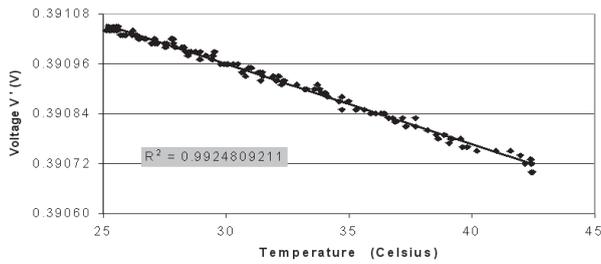
Figure 3. Effect of the solenoid temperature on the measured voltage.

Figure 4. Calibration curve for the iron powder dispersed in the non magnetic medium.

The voltage drop across the measurement resistor varies with RL according to:

$$V = V_0 R / [(R_L + R)^2 + (2\pi f L)^2]^{1/2} \qquad (9)$$

where V_0 is the output voltage of the voltage generator. A temperature variation ΔT produces a variation in the resistance of the winding, resulting in a variation ΔV of the measured voltage V given by:

$$\Delta V = - [\alpha V R_L (R_L + R) \Delta T] / [(R_L + R)^2 + (2\pi f L)^2]^{1/2} \quad (10)$$

To compensate for the above thermal variation, the voltage V must be brought back to the value it would have had, if the temperature had been maintained constant, using the following correction:

$$V' = V \{ 1 + [\alpha V R_L (R_L + R) \Delta T] / [(R_L + R)^2 + (2\pi f L)^2]^{1/2} \} (11)$$

The above value is used in Equation 8 to calculate ΔV. Although the resistance RL appears in the expression for the factor Q, this factor must not be corrected for thermal effect because the corrected voltage V' is used in the expression for ΔV. Figure 3 shows the effect of the solenoid temperature on the measured voltage V. The measured voltage decreases linearly with temperature as expected from Equation 10.

The calibration curves are obtained by introducing a known mass of iron powder into the solenoid. The iron powder was produced by Hosakawa Micron Inc. using an air jet grinding technology. The grain size volume distribution of the powder was measured using a Coulter particle size analyser. The distribution is slightly skewed towards small particles. The magnetic field inside the solenoid is not perfectly homogeneous, and the iron powder must be uniformly spread inside the volume of the solenoid. To achieve this goal, the iron powder standards were dispersed in a non magnetic medium and in red mud. The calibration with the non magnetic supporting medium is used to detect any effect that the bauxite residue matrix may have on the measurement of the iron tracer. The mixture was introduced into a tube that fits perfectly into the inside space of the solenoid. Figure 4 shows the calibration curve in the non magnetic medium. The slope of the calibration curve is given by

$$d\Delta V / dm = - V' (\mu_{Fe} - 1) / \{ \rho_{Fe} V [1 + (R_L + R)^2 / (2\pi f L)^2] \} (12)$$

where m is the mass of the iron powder, μ_{Fe} is the initial relative magnetic permeability of the iron powder and V is the solenoid volume below the copper winding. The least squares fit slope corresponds to a relative magnetic permeability of ~6 for the iron powder. In this respect the initial relative permeability of pure iron may be as low as 200 (Reitz et al., 1992), but when iron powder is used instead of massive iron it may be twenty times smaller (Trémolet de Lacheisserie,1999). The detection limit corresponds 0.54 g/L in the non magnetic medium.
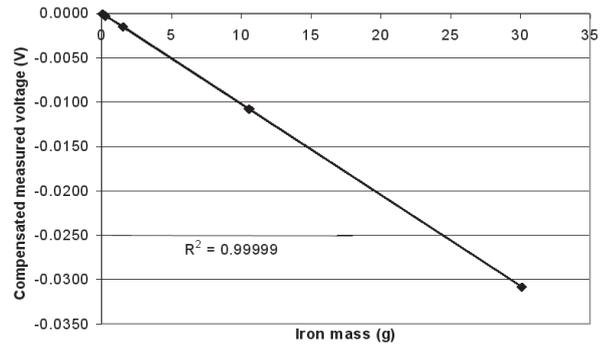
The red mud used for the calibration was reworked and had a solid fraction in the range of 35–40% (w/w). The iron powder was dispersed in red mud instead of the non magnetic medium but the same calibration procedure as above was followed. The measured voltage is decreased when red mud is filling the solenoid. The decrease corresponds to a relative magnetic permeability of 1.005 for the red mud. If one takes the Bayer liquor to be non magnetic, then a relative magnetic permeability of 1.01 would correspond to the solid phase of this red mud. Figure 5 shows the calibration curve of the iron powder dispersed in the red mud. Analysis of the data also yields a relative permeability of ~6 for the iron powder. The detection limit corresponds to 0.51 g/L in red mud.

5   Plant test

The apparatus was first tested in the laboratory with a mud circuit (Figure 6) that simulates mud circulation through the underflow of an industrial settler. The impedance Z_{ref} consisted of a second solenoid to take into account the noisy electromagnetic environment in the plant as explained previously. This second solenoid was filled with static red mud free of the tracer. The apparatus was then tested in a thickener of an Alcan alumina refinery. The iron powder described previously was manually injected through the feed well. The injection of the iron powder lasted ten minutes. Part of the red mud in a recirculation loop of the underflow was diverted through the measurement solenoid.
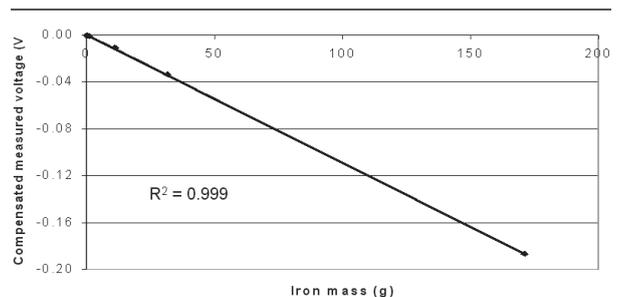

Figure 5. Calibration curve for iron powder dispersed in red mud.
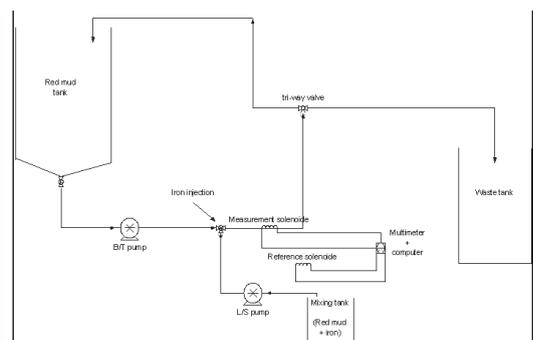

Figure 6. Mud circuit for the laboratory test.

Figures 7 and 8 show the raw data that was collected. It is observed from Figure 8 that the temperature of the solenoid in the measuring branch dropped by one degree near the end of the experiment. No thermocouple was fixed on the solenoid in the reference branch, but it is estimated that its temperature drop was more pronounced, approximately three degrees, because it was not protected by the thermal inertia of the flowing red mud. The data was processed to subtract extraneous variations from the measured signal. Using the calibration curve established in the laboratory, the processed signal was converted into iron concentration. Using the known value of the red mud flow rate in the underflow, concentration was converted into an iron mass flow rate. This final result is shown in Figure 9. The mass flow rate distribution rises rapidly at first and decreases slowly at the end. This is typical of the distributions obtained with continuous flow reactors (Fogler, 2002). The modulation on the curve has a period that is characteristic of that of the rake in the mud bed. A detailed analysis of the mass flow rate distribution is required for the diagnosis of the settler. Integration of Figure 9 with time yields the cumulative distribution with time (Figure 10). The median residence time t_r (the time required to recover half the iron injected at the feed well) can be read from this. The asymptotic level of iron recovery was reached after approximately twice this median residence time, with ~92% of the injected iron powder accounted for.
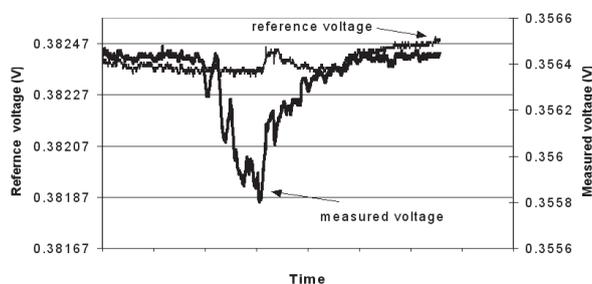

Figure 7. Reference and measured voltages at the underflow of an industrial settler.
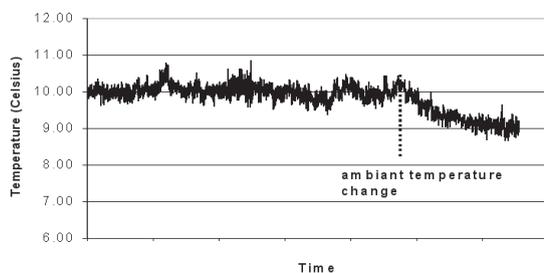

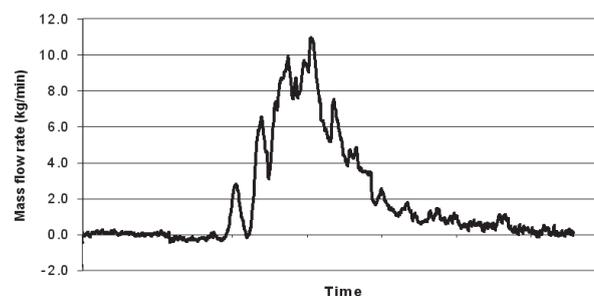Figure 8. Temperature variation of the measurement branch solenoid.

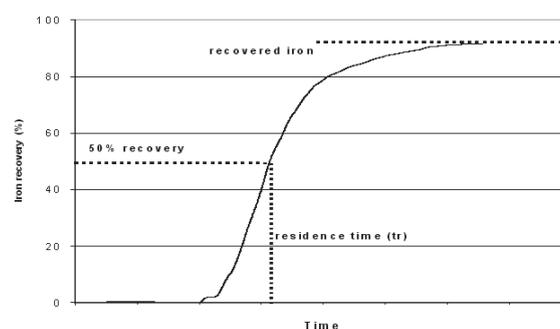Figure 9. Iron mass flow rate distribution at the underflow of an industrial settler.

Figure 10. Iron cumulative residence time distribution.

6 Conclusion

Tracer technique using iron powder to simulate solid particle behaviour has been shown to be applicable to the diagnosis of gravity settlers. To achieve this goal, an apparatus to detect and measure iron particles dispersed in red mud at a concentration level of 0.5 g/L has been designed, put together in our laboratory and successfully tested in an industrial settler. The working principle of the apparatus makes use of the electromagnetic induction of a solenoid incorporated in an a.c. R_L electrical circuit. Because the required level of detection corresponds to magnetic fields similar in magnitude to that of the earth, great care must be taken in eliminating extraneous sources of electromagnetic induction in the circuit and to minimize any source of electric noise originating in the circuit itself. This is partly achieved by using an electrical source that is very stable in frequency and continuously monitoring a reference branch to take into account any residual electric noise down to the required level. Data processing may also be useful in this respect. The apparatus was successfully tested in an Alcan industrial thickener. The residence time distribution was typical of those obtained with continuous flow reactors. Even the rake action is discernable through the data, and a diagnosis for the rake might be obtainable with this apparatus. The chemical engineering literature is abundant with the mathematical treatment of tracer data pertaining to continuous flow reactors and it could be easily applied to settlers.

References

Borody, E, 1985, Électromagnétisme, théorie et applications, Presse de l'Université du Québec.

Evans GV, 1982, 'The use of tracers in water pollution control', Physics in Technology, vol 13, pp. 3–10.

Fogler, HS, 2002 Elements of chemical reaction engineering, Prentice Hall PTR, third edition.

Grocott SC & McGuiness L, 1990, 'Residence-time distributions in Bayer process vessels. Development of a suitable liquor tracer', Light Metals 1990, pp. 95–102.

Harrison EH, Lépine A, Gaboury A & Thé K, 1987, 'The use of sodium nitrate as a tracer in Bayer liquor: analysis by ion chromatography', Light Metals 1987, pp. 73–76.

Levenspiel O, 1972, Chemical reaction engineering, Wiley, second edition, New York.

Reitz, JR, Milford, MJ & Christy RW, 1992, Foundations of electromagnetic theory, Addison-Wesley, third edition, New York.

Trémolet de Lacheisserie, E, 1999, Magnétisme: II Matériaux et applications, Presse universitaire de Grenoble, Grenoble.