Vessel Diagnosis in the Bayer Process Using Ferromagnetic Tracers

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Abstract

In the Bayer process, having a good knowledge of the residence time distribution (RTD) of thickened slurry and pulp flows can help achieve a more efficient operation, diagnose problems such as channeling and dead zones, or evaluate the effectiveness of operational parameters. A tracing procedure and apparatus was developed by Rio Tinto in partnership with the Université du Québec à Chicoutimi to monitor slurry displacement patterns. The principle of the apparatus is the detection of a solid ferromagnetic tracer by inductance coils. The RTD obtained is then analyzed with a proprietary Rio Tinto deconvolution method to understand the different flows inside the vessels. Iron powder is usually utilized as a tracer for its compatibility with slurry of bauxite residue. However, magnetite has been successfully tested as an alternative tracer since it can be used at higher temperature and higher caustic concentrations than typical Bayer washer circuit conditions. The tracing technique was validated at plant scale for the diagnosis of slurry behavior in deep thickeners, stirred tanks, and pipes.

Keywords: Residence time distribution; tracer; flow; diagnosis; ferromagnetic; tanks; pipes.

1. Introduction

In the Bayer process, a number of steps are required to allow alumina extraction such as pre-desilication, digestion, settling, clarification, thickening, mud washing circuit, slurry transport in pipes, etc. The industrial facilities used are quite large and many were constructed at a time when process conditions were different from today. Moreover, the general decrease in bauxite quality results in processing larger volumes for a consistent alumina production rate, which requires a better equipment performance. In this context, a better understanding and optimization of these process steps become crucial. Having a good knowledge of the RTD of slurry and/or thickened slurry flows can help to achieve a more efficient operation as well as diagnose channeling and dead zone problems in operating facilities, or evaluate the effectiveness of various operating parameters.

Tracers are widely used to diagnose the behavior of continuous flow chemical reactors [1-2]. In these techniques, the reactor RTD, from which many physical parameters can be computed, is obtained by measuring the tracer at the reactor outlet. Analysis of the RTD can be useful in diagnosing the reactor's behavior. To be applicable to industrial facilities, this technique requires an easily measurable tracer that is compatible with the physical and chemical process environment. Tracing techniques have been used previously to simulate liquid flow in settlers [3-4] and thickened slurry behavior in thickeners [5]. Research on an on-line electromagnetic iron tracer detector capable of measuring RTD in industrial equipment has been published previously [6-8].

This paper presents a tracing apparatus developed by Rio Tinto in partnership with the Université du Québec à Chicoutimi [6-8]. This apparatus was used at plant scale to validate slurry behavior in the last deep thickener of the mud washing circuit, in a pre-desilication stirred tank, and in the pipe connecting the refinery to the Residue Management Area (RMA). Two types of tracers were used to carry out measurements in different operating conditions.

2. Tracing Technique

2.1. Ferromagnetic tracers

It is difficult to carry out RTD measurements in the Bayer process because of its aggressive chemical environment. Furthermore, the Bayer process is a two-phase system (solid and liquid) and the tracer must therefore be adapted to represent the real flow to be measured. The use of a ferromagnetic tracer allows the continuous monitoring of the slurry displacement with an on-line electromagnetic inductance measurement method. Because of the differences in Bayer process operating conditions, two tracers were used to carry out the measurements. The first was iron powder, which is used in the mud washing circuit under low caustic soda and relatively low temperature conditions. The second one was magnetite powder that can be used at higher temperature and higher caustic soda concentrations than normally found under typical red mud washing circuit conditions. Indeed, iron powder can be reactive under such conditions, potentially leading to iron oxides and hydrogen gas evolution. As the tracer must remain intact, the use of a final oxidation product that is ferromagnetic, like magnetite, is required. Indeed, both tracers show a good chemical resistance for the time span required to measure RTD under the specific trial conditions. Moreover, they are not toxic and may be easily disposed with the bauxite residue after use. Ground and digested bauxite has a particle size distribution ranging from less than 6 to ± 100 microns. It was previously shown [6] that a ferromagnetic powder with an appropriate average diameter can simulate the slurry displacement. In addition, iron oxide particles are already present in bauxite, so there is no contamination of the process. The large magnetic permeability of iron or magnetite allows the detection of the tracer by electromagnetic techniques. Consequently, the ferromagnetic powder is an ideal tracer to monitor slurry displacement in the Bayer process [3].

2.2. Tracers injection and measurement

The quantity of tracer injected into a vessel is evaluated using the instrument’s limit of detection (LOD, in g/L) and the volumeof slurry inside the vessel. The suspension (Bayer liquor with tracer powder) is injected rapidly at the usual inlet to generate a pulse. Operational parameters such as rake torque, agitator speed, feed flow, underflow, bed level, recirculation flow, etc. are recorded by the plant’s DCS (Distributed Control System).

2.3. Detection method

The detection method consists of injecting a given amount of tracer at the inlet of the vessel and detecting it at the outlet with an inductance coil. In fact, a magnetic field is generated around the coil by applying an electric current. When the ferromagnetic particles cross the coil, the permeability of the medium is modified, which causes variation of the magnetic field [6-8]. The electromagnetic detector is principally built with two inductance coils. The first one is used to measure the ferromagnetic tracer in the slurry flow. The second one acts as a reference coil and measures any extraneous signal originating from the electromagnetic environment. The final signal is obtained by subtracting one from each other and represents the global RTD. Figure 1 shows a picture of the detector. The latest generation of detectors includes two identical and larger coils, a temperature controller on the reference coil to mitigate temperature variations and a better and stronger electrical circuit. The electrical circuit is encased inside an easily transportable suitcase.

Calibration curves are obtained by circulating a suspension containing known amounts of ferromagnetic tracer through the measuring coil. As the calibration of the instrument can be affected by the nature of the suspension, its solid fraction and the volume of slurry inside the coil, a calibration curve must be established for each new measurement campaign.

Figure 1. Detector.

3. Results and discussion

3.1. Tracing in a Deep Thickener

Tracing was performed in the last deep thickener of a mud washer train. This thickener was chosen because it is operated under stable conditions with low caustic concentration and low temperature (~30 g/L and 20-40ºC). The selected tracer was iron. This thickener is a flat-bottom tank using a rake system at a conical angle. A recirculating loop at the bottom of the tank facilitates the flow of thick slurry [9]. Figure 2 presents the schematic of this deep thickener and its surrounding equipment in the tracing flow sheet.

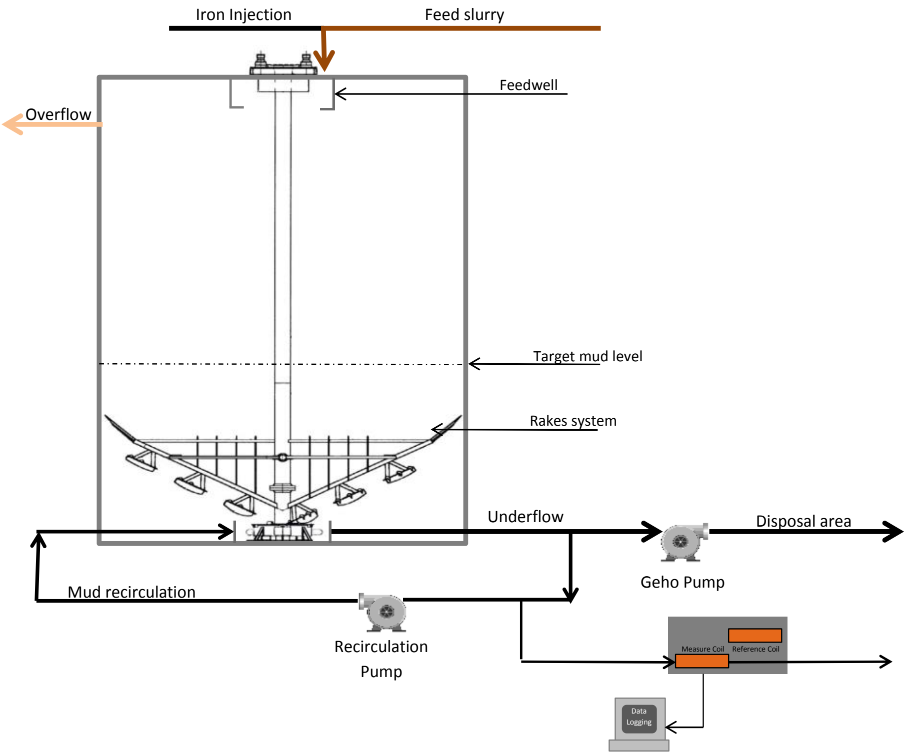

Figure 2. Deep thickener tracing flow sheet

The level of thick slurry inside the thickener is normally variable, but for the experiment it was kept as constant as possible at a set target level. The estimated volume does not take into account the volume of the rake or the possibility of a dead volume under the rake. The calculated global residence time $\tau_{calculated}$ in the thickener is estimated at ~ 8.4 h ($\tau_{calculated} = V/Q$), where V is the volume of thick slurry and Q the flow rate of the underflow.

Raw data from experimentation consist of coil measurements expressed as Corrected Signal (µH) as a function of time. Time t = 0 represents iron injection. Recording of both coils inductance starts before the iron injection (not presented on graph) to get a stable signal with the thickened slurry circulating inside the electromagnetic detector. These data are converted into iron concentrations using the calibration curve (section 2.3), which allows the calculation of the recovered amount of iron. In the present case, ~ 98.4% of the injected iron was recovered.

The residence time of the thickener is obtained from the E(t) curve [1-2], which is usually known as the RTD curve and represents the probability per time unit that a particle exits the settling tank at time t. Figure 3 presents the RTD curve obtained from the deep thickener tracing. The measured residence time is 3.7 hr, which is less than half of the calculated vessel residence time. This means that the flow through the vessel is faster than expected ($\tau_{measured} < \tau_{calculated}$), indicating the presence of a dead volume inside the thickener. Figure 3 suggests that the dead volume is likely to be situated under the rake system. See Figure 4 for a schematic of dead volume. The estimated dead volume represents 52.7% of the total thickened slurry volume inside the thickener, which is consistent with the measured residence time being less than half the calculated one.

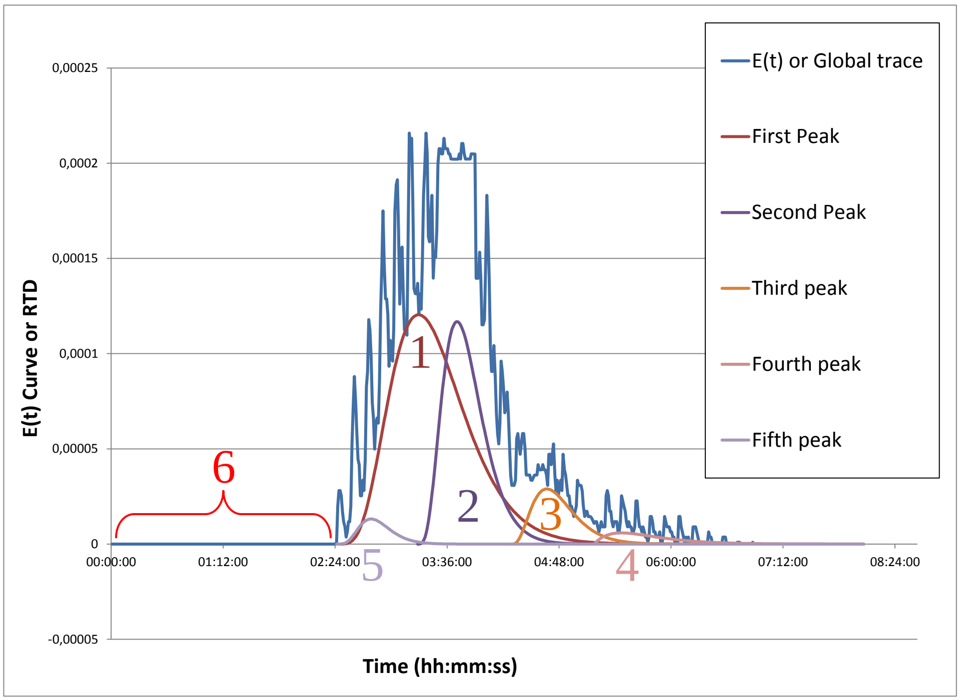

Figure 3. Deconvolution of the RTD curve from deep thickener tracing.

A lot of information can be obtained from the shape of the E(t) curve. As shown in Figure 3, the global shape is similar to that of a PFR (Plug Flow Reactor) in non-ideal operation [1, 2, 7, 8]. The RTD obtained was analyzed with a proprietary Rio Tinto deconvolution method to understand the different flows inside the vessel [7-8]. The shape of the global RTD may be interpreted as the sum of individual RTD curves, each being specific to different pathways inside the thickener. The global trace represents all the surface of the thick slurry bed inside the deep thickener. The global RTD was decomposed into five individual peaks indicating the presence of five areas in the thickener. The first peak represents 43.5% of the global area; thus, the major part of the thick slurry flows through this pathway. Its τ_i (specific residence time of the area) is around 3.5 hr. This pathway has therefore a smaller residence time than that of the global trace (3.7 hr), indicating a slightly faster zone. To simulate this peak, six CSTR (Continuous Stirred Tank Reactor) in series were needed so that the flow pattern is much closer to a PFR (Several CSTR in series result in a PFR flow). The thick slurry that flows in this area is not well mixed and travels directly to the underflow. Table 1 presents a summary of the analysis.

Table 1. Global RTD deconvolution.

	Global	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5
Area (%)	75,7	43,5	22,7	5,8	1,7	2,0
τ_i (h)	3,7	3,5	3,8	4,8	5,8	2,9
Type of Flow	--	(Fast zone)	(Intermediate zone)	(Slow zone)	(Dead zone)	(Bypass)
N	--	6	5	4	2	6
Mud Processed (%)	100%	60,5	28,8	5,9	1,4	3,4

The RTD decomposition indicates that the thick slurry bed is composed of three zones: a fast active zone (Peaks 1 and 5), an intermediate zone (Peak 2) and a slow zone (Peaks 3 and 4), as shown in Figure 4. The flat part at the beginning of the RTD curve (element 6 in Figure 3), which represents 2.4 hr, corresponds to the time taken by the tracer to travel with the thick slurry bed in the cylindrical section of the thickener (portion between the target level and the rakes in Figure 2 and Figure 4). It was previously demonstrated [7-8], for a smaller thickener that each zone is associated with a thick slurry volume located at a specific distance from the underflow. As shown in Figure 4, the slowest zone will be associated with a mud volume located near the wall (r = r) of the thickener and the fastest zone will be located near the center (r = 0). In addition, the type of flow associated with Peaks 3 and 4 being similar to that of a CSTR (N is low), it demonstrates that the longer the thick slurry has to travel to the underflow, the better it is mixed. This is consistent with the fact that the thickened slurry in contact with the rake for a longer time will be better mixed, which corresponds to the observed flow behavior shown by peaks 3 and 4.

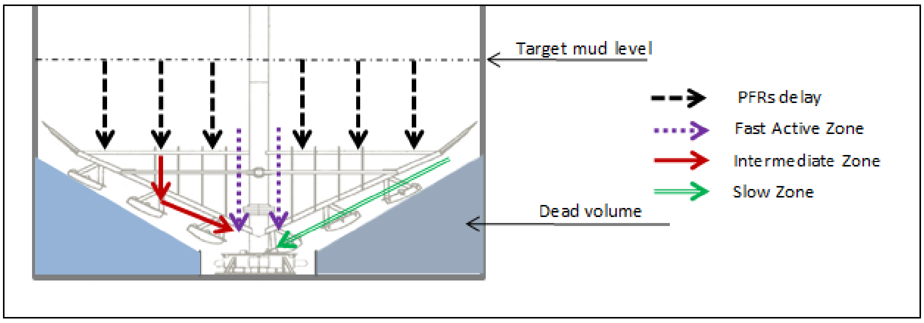

Figure 4. Decomposed flow pattern in the deep thickener.

In addition, Peak 1 begins to appear at 2.4 hr, Peak 2 after 3.3 hr and Peak 3 after 4.3 hr. By removing the time associated with the flat part of the RTD curve, the remaining times (0, 52 and 113 min) would be the approximate time needed for the thick slurry to travel from each zone toward the underflow by rotation of the rake. In fact, the thick slurry circulation in the cylindrical section is similar to a PFR flow, and the decomposition mainly represents the work of the rake. The quantity of thick slurry processed through the pathways of each peak is proportional to the areas of the peaks divided by their τ_i value. This approach suggests that more than half of the thickened slurry travels through the fast zone (Table 1).

3.2. Tracing in a Pre-desilication Tank

Tracing was also performed in Vaudreuil refinery’s first pre-desilication tank (PDS 1). This tank was chosen because it is operated under stable conditions with high caustic concentration and medium temperature operation (~260 g/L and 65ºC). The selected tracer was magnetite. This tank has a conical bottom and comprises two injection points, an underflow and a stirring system. Figure 5 presents the schematic of this tank and surrounding equipment in the tracing flow sheet.

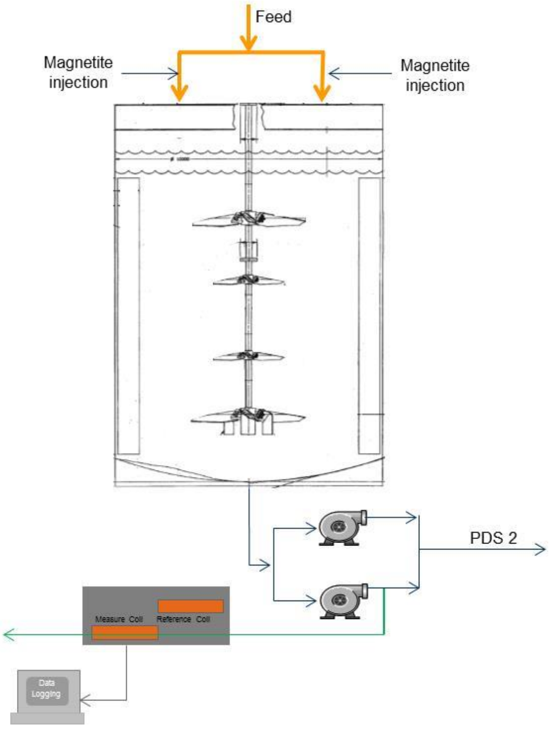

Figure 5. Pre-desilication tank tracing flow sheet.

The calculated global residence time $\tau_{calculated}$ of this PDS tank is estimated at ~ 2.1 hr. Figure 6 presents the RTD curve obtained for PDS-1. The amount of magnetite recovered from that tracing represents ~ 72% of the total injected tracer. As observed in Figure 6, it was impossible to record all the trace due to operational problems. However, by analyzing the RTD with the RT deconvolution method, it was possible to simulate where it should end (see First Peak in Figure 6). The measured residence time using the global trace is 1.0 hr. The residence time obtained from the simulated curve is 1.3 hr. These measured residence times are lower than expected ($\tau_{measured} < \tau_{calculated}$), indicating the presence of a non-operational zone inside the tank. Assuming that the baffles in the tank (Figure 5) generate an annular volume that does not contribute to the active flow, a dead volume representing about 32% of the total pulp volume is present in the tank. The recalculated global residence time then becomes ~ 1.4 hr, which is similar to the residence time obtained with the simulated curve. The difference of 0.1 hr probably comes from the volume associated with the agitators' structure.

The global RTD was decomposed into two individual peaks. The overall shape of the RTD is consistent with a CSTR flow. In fact, the first decomposed peak (Figure 6, Peak 1) of the RTD curve represents 96.8% of the global area, thus the major part of the pulp flows inside the tank through this pathway. To simulate that peak, one CSTR was required so that we can consider that the flow is perfectly agitated. The second peak (Figure 6, Peak 2) represents 3.2% of the total area. This second peak could be explained by the time needed by the tracer to be homogenized. In addition, there is a short delay (Figure 6, zone 3) that could be associated to a pulp bed that forms at the bottom of the tank.

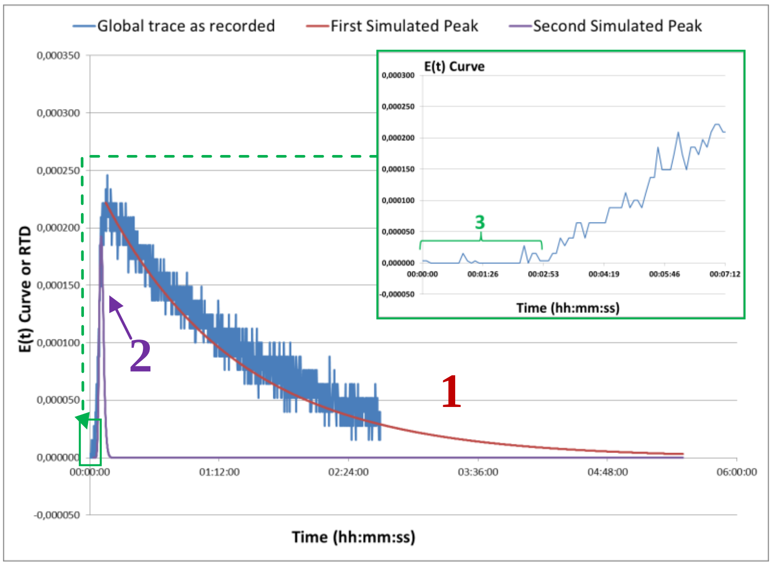

Figure 6. Deconvolution of the RTD curve from the first pre-desilication tank.

3.3. Tracing in the Pipework to RMA

Tracing was performed in the pipe connecting the last mud washer to the RMA. This pipe was chosen to confirm the residence time historically measured by water injection and to evaluate internal scaling. The tracer was iron. Figure 7 presents the schematic of the tracing flow sheet in this configuration. The pipe was around 1.6 km long (thick line in Figure 7).

The calculated maximum residence time $\tau_{calculated}$ for the pipe is estimated at ~ 0.29 hr. Figure 8 presents the RTD curve obtained from tracing in the pipe connecting the last mud washer to the RMA. The total amount of iron injected was recovered. The measured residence time is 0.23 hr of the global trace, indicating the presence of dead zones.

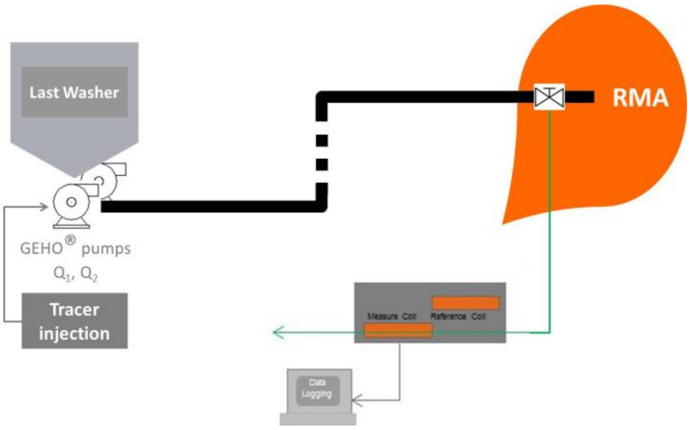

Figure 7. Tracing flow sheet for the pipe connecting the last mud washer to the RMA.

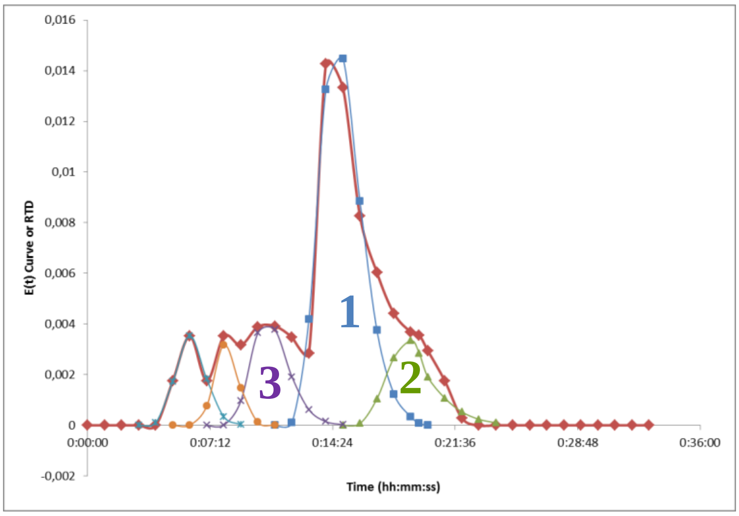

Figure 8. Tracing curve obtained for the pipe between the last mud washer and the RMA.

The global RTD was decomposed into five individual peaks indicating the presence of five types of flows in the pipe. The first major peak (Figure 8, Peak 1) represents 54% of the global area, the major part of the mud flowing inside the pipe through this pathway. Its τ_i (0.25 hr) is near that of the global curve. Its shape is similar to a PFR flow, which is expected. Peak 2 represents 16% of the global area with a 0.33 hr τ_i, longer than the global residence time. This means that this portion of the flow was delayed, possibly by a restriction like an elbow, a bend or scale accumulation for instance. In addition, the flow associated with Peak 2 is much closer to a CSTR behavior, which could indicate a turbulent zone. Peak 3 represents 13% of the global area with a 0.20 hr τ_i, lower than the global residence time. Its shape indicates a piston behavior. It can be assumed that this portion of the flow has a laminar behavior and is situated in front of the main flow in the pipe. Simulations at lab scale are necessary to validate these hypotheses, however, the main purpose of this measurement, to validate the RTD in pipes using the tracing technology, was achieved.

4. Conclusion

A tracing technique using a ferromagnetic tracer to understand the flow patterns in three different vessels proved very effective and easily applicable in an industrial environment. It was also shown that the RTD decomposition method is applicable for analyzing the slurry behavior in a deep thickener, a stirred tank and pipework. Moreover, it was previously shown that this method is able to detect and quantify some common problems like bypass or dead zone and, in this respect, may be used for troubleshooting purposes [7-8]. The apparatus is robust and may easily be used in plant facilities. This technique may be adapted to various other types of mineral industries to assist in the comprehension and/or the diagnosis of industrial vessels. It could also be helpful in the evaluation of tank aging in order to optimize the cleaning schedule, or in the evaluation of plant modifications. In the current depressed economic context of the mining and mineral industries, equipment that helps to understand, improve or diagnose an industrial process at low cost can be very advantageous.

5. References

1. O. Levenspiel, Chemical reaction engineering, Third edition, *John Wiley and Sons*, 1999, 668 p.
2. H.S. Fogler, Elements of chemical reaction engineering, Fourth edition, *Prentice Hall Professional Technical Reference*, 2006, 1080 p.

3. S.C. Grocott and L. McGuiness, Residence-time distribution in Bayer process vessels development of a suitable liquor tracer, *Light Metals*, 1990, 95-102.
4. E.H. Harrison et al., The use of sodium nitrate as a tracer in Bayer liquor: analysis by ion chromatography, *Light Metals*, 1987, 95-102.
5. G.V. Evans, The use of tracers in water pollution control, *Physics in Technology,* Vol. 13, 1982, 3-10.
6. A. Leclerc et al., Electromagnetic detection of iron tracers for monitoring particles displacement in gravity settlers, *Proceedings of the 7th International Alumina Quality Workshop*, Perth, Western Australia, 2005, 127-130.
7. M.L. Bouchard et al., Diagnosis of mud displacement in gravity settlers from analysis of their residence time distribution, *Paste* 2010, Toronto, 61-72.
8. M.L. Bouchard, Utilisation d’une technique de traçage ferromagnétique pour étudier le comportement et le déplacement des boues rouges dans un décanteur, Ph.D. thesis, *Université du Québec à Chicoutimi*, 2011.
9. United States Patent No US 6,340,033 B2, 22 Jan 2002.

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