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SOLIDS COMPOSITION MEASUREMENTS OF PHOSPHATE SLURRY USING IMPEDANCE SPECTROSCOPY, PHASE I: FEASIBILITY STUDY

FINAL REPORT

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FINAL REPORT

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PERSPECTIVE

Patrick Zhang, Research Director - Beneficiation & Mining

On-line analysis and process automation are powerful tools for achieving optimal industry efficiency. The benefits of an effective on-stream analysis technique coupled with a modern process control scheme include improved recovery of P₂O₅, reduced chemicals consumption, and improved concentrate grade. As the quality of phosphate reserves declines, improvement in plant efficiency is even more critical. Unfortunately, process control in phosphate beneficiation plants is still primitive. This is due to the following difficulties in analyzing the plant slurry: (1) multi-element analysis (P₂O₅, CaO, SiO₂, and MgO) is required for meaningful process control, (2) the slurry is a heterogeneous system with about 20-30% solids, and (3) the major elements to be analyzed, phosphorus and calcium, are low in atomic number, thus preventing the use of the x-ray fluorescence technique that is widely adopted in other mineral industries.

Since its inception, FIPR has sponsored five major projects for evaluating on-line analytical systems: a nuclear magnetic resonance (NMR) technique, an optical sensor, a neutron activation probe, an X-ray diffraction system, and LIBS (Laser Induced Breakdown Spectroscopy). Except for some limited applications of the NMR analyzer and an in-line LIBS analyzer for the pebble fraction on a moving belt, none of the techniques has gained broad industry endorsement. Some analyzers have problems with their sample feeding systems, while others do not give accurate analyses.

Electrochemical impedance spectroscopy (EIS) (sometimes also called AC impedance) is an electrochemical technique in which a low-amplitude alternating potential (or current) wave is imposed on top of a DC potential (often the corrosion potential and zero imposed current). As such, this technique is, in essence, built on DC techniques. In DC technology, a voltage (or current) is imposed on an electrochemical system and the resulting current (or voltage) is measured. The relationship between the voltage and current is used to make a judgment about the corrosion rate of a material and other characteristics. Currently, EIS is most widely used in studying corrosion.

The idea of developing an impedance-based in-situ analyzer for measuring solids concentration for phosphate, sand, clay and water constituents is intriguing because such an analyzer would cost about \$20,000, a fraction of the cost for any other type.

This project demonstrated the feasibility of measuring percent solids and sand concentration from a phosphate slurry flowing through a pipeline. It remains challenging to measure other constituents of major interest, such as phosphate and dolomite.

ABSTRACT

The accurate online concentration measurement of solid constituents within a slurry mixture entering a phosphate beneficiation plant can assist to significantly improve the phosphate flotation efficiency. This report includes an impedance spectroscopy feasibility study for the online determination of solid constituent concentrations flowing in a slurry pipeline. A bench scale slurry flow facility has been fabricated, and the test section has been configured with various electrode combinations. An Agilent 4294A impedance spectrometer is used to measure the impedance characteristics of a flowing slurry consisting of water, sand, and amine concentrate over a wide range of frequency. It has been observed that signal quality obtained from the spectrometer is largely dependent on the electrode surface area to spacing ratio. Mathematical algorithms have been developed to measure the amine, sand, and water concentrations from the impedance spectrometer signal, provided the impedance characteristics of the water are known a priori. Also, an algorithm was developed to distinguish the total solids concentration from that of the water, without having to know the impedance characteristics of the water a priori. Thus, it is concluded that with further effort, it should be possible to construct an impedance based instrument to measure the online concentration of water, amine, and sand constituents from a phosphate slurry flowing through a pipeline.

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EXECUTIVE SUMMARY

One of the research priorities for the Florida Institute of Phosphate Research (FIPR) is to improve phosphate flotation efficiency through the development and evaluation of on-line analyzers and process control systems. Phosphate beneficiation comprises several processing stages, the last being flotation. Getting the material through the flotation stage is expensive, and thus maintaining a high efficiency of the process is particularly important in producing a competitive product. During flotation, costly reagents are used to enhance the phosphate recovery. Common practice of reagent control remains largely intuitive since there does not exist a widely accepted real-time precision measurement of the mineral fractions of interest. The development of a simple and inexpensive analyzer would greatly improve progress toward the automation and optimization of the flotation process.

Here, the University of Florida and Mosaic have teamed together to explore the feasibility of developing an in-situ analyzer for measuring solids concentration for phosphate, sand, and water constituents using impedance spectroscopy. Impedance is usually used to describe the resistance to electrical flow across an electrode pair when subjected to a periodic electric potential. Since there are amplitude and phase differences, the impedance is a complex quantity which is an extension of the concept of resistance, and it is frequency dependent. Impedance spectroscopy is a measurement technique that exploits the frequency dependence of dielectric and conducting materials.

Considering the requirement for a wide frequency range coverage and short sampling time, an Agilent 4294A impedance analyzer was chosen for the present investigation. A slurry flow test loop was fabricated and test sections with different electrode configurations were tested in the slurry loop. Because the impedance is temperature sensitive, a simple heating and cooling section was incorporated into the slurry loop to maintain a uniform temperature.

Using the slurry flow loop, the impedance characteristics of various slurry mixtures were analyzed using water and solid samples provided by Mosaic that were extracted from a typical phosphate matrix. These samples include rougher tails, amine concentrate and amine circuit water. They contain impurities because they were extracted directly from the field. This source of impurities resulted in some uncertainty in the calibration. The equivalent circuit method was used to model the impedance measurements and to estimate the concentrations of the two solids in the slurry. A very simple RC parallel circuit is assumed. The mathematical model identifies the values of R and C which minimize the square error between the measured impedance and the impedance of the model RC parallel circuit over the same frequency range. By recording the impedance of the slurry with different unknown tail and amine concentrations for calibration and assuming a linear relationship between R, C and concentrations of the two solids, an estimate for the unknown concentrations of the two solids is determined through optimization. The experiment results are satisfactory. However, this method requires that the impedance characteristics of the water carrier be known a priori.

In order to address the water carrier reference requirement, a substantial effort has been made to develop a measurement method that does not require a reference signal for the water carrier. The key to addressing this problem is the recognition that solid particles with high capacitance and low conductance in a slurry tend to block more AC current at low frequency compared to high frequency. To exploit this behavior, a new test section was fabricated with new electrodes which are more reliable at low frequencies. Experiments were carried out with different carriers and solid concentrations. Since the influence from temperature is easy to be compensated through careful calibration, all the experiments are run at 26°C. The determination of the solids concentration relied on a non-parametric method. The real part of impedances measured at 1 MHz and 25.56 MHz were used to deduce the total solids concentration. No information on the water carrier impedance is required. Again, satisfactory results are obtained.

The results of this investigation demonstrate that impedance spectroscopy is a useful instrument for measuring solids concentration in slurries. Two different algorithms have been established for predicting the solids concentration from impedance spectroscopy measurements. One requires information on the water carrier impedance and the other does not. With some further effort it should be feasible to develop an impedance spectroscopy based solids concentration measurement instrument that will be useful for phosphate mining applications.

INTRODUCTION

One of the research priorities for the Florida Institute of Phosphate Research (FIPR) is to improve phosphate flotation efficiency through the development and evaluation of on-line analyzers and process control systems. Phosphate beneficiation comprises several processing stages, the last being flotation. Getting the material through the flotation stage is expensive, and thus maintaining a high efficiency of the process is particularly important in producing a competitive product. During flotation, costly reagents are used to enhance the phosphate recovery. Common practice of reagent control remains largely intuitive since there does not exist a widely accepted real-time precision measurement of the mineral fractions of interest. The development of a simple and inexpensive analyzer would greatly improve progress toward the automation and optimization of the flotation process.

Here, the University of Florida and Mosaic are teaming together to explore the feasibility of developing an in-situ analyzer for measuring solids concentration for phosphate, sand, and water, constituents using impedance spectroscopy. Using impedance spectroscopy, a measuring technique has been developed to quantify the concentration of phosphate and sand in water slurry. The difficulty in using this technique is that it requires a reference signal for the water carrier. This technique will first be described. In further studies it has been found that by increasing the size (surface area) of the measuring electrodes, both the high and low frequency signals are useful, and thus a new measuring algorithm has been developed which will allow the determination of the overall solids concentration without the need of the water carrier reference signal. This will provide for a robust and reliable solids concentration instrument. This measuring technique will be described second.

Based on the feasibility study described herein, these measuring techniques should be useful for the development of an in-situ solids concentration sensor for phosphate mining applications.

ELECTRICAL IMPEDANCE SPECTROSCOPY

Electrical impedance, or simply impedance, describes a measure of an object's response to a sinusoidal alternating current (AC) electrical signal. It can be represented by the change of amplitude and phase of the output signal from the input signal. It is usually noted as a complex value Z.

$$\boldsymbol{Z} = \boldsymbol{V} / \boldsymbol{I} = |\boldsymbol{Z}| \boldsymbol{e}^{j\theta} = \boldsymbol{Z}' + \boldsymbol{j}\boldsymbol{Z}''$$
(1)

Z' is called resistance and Z'' is called reactance. When Z'' < 0, it is called capacitive reactance or capacitance; when Z'' > 0, it is called inductive reactance or inductance.



Figure 1. Simple Sketch of an Electrode Pair Used for Impedance Measurement.

FigureFigure 1 shows a simple electrode pair used to measure impedance. It consists of two parallel plate-shaped electrodes with a certain material between them. The distance between two electrodes is d, with an area A for each electrode. When the material between two electrodes is an ideal conductor, the impedance is:

$$\mathbf{Z} = \mathbf{Z}' = \rho \frac{d}{A} \tag{2}$$

where ρ is the resistivity which is a physical characteristic of the material. $\sigma = 1/\rho$ is the conductivity. The conductance G, which is the reciprocal of the resistance, is expressed as:

$$G = \frac{1}{Z'} = \sigma \frac{A}{d} \tag{3}$$

When the material between two electrodes is an ideal dielectric, the impedance is:

$$Z = jZ " = -j\frac{1}{\omega C} = -j\frac{1}{\varepsilon \omega}\frac{d}{A}$$
(4)

where $\varepsilon = \varepsilon_r \varepsilon_0$ is the permittivity. ε_r is the relative permittivity and $\varepsilon_0 = 8.85 \times 10^{-12} F / m$ is the permittivity of a vacuum. The susceptance *G* is the reciprocal of the reactance B = 1/Z". The admittance is defined as:

$$Y = \frac{1}{Z} = G + jB \tag{5}$$

The permittivity ε and conductivity σ , of a material are respectively, the charge and current densities induced in response to an applied electric field across the electrodes. When $\frac{\sigma}{\omega\varepsilon} \gg 1$, the material is considered to be a good conductor. Conversely, when $\frac{\sigma}{\omega\varepsilon} \ll 1$, it is considered to be a good dielectric. Those materials that do not fall within either limit are considered to be general media.

Actual materials are quite complex and the material behavior can be frequency dependent. It has been observed that the permittivity ε usually varies with frequency ω and the concept of complex permittivity is introduced as:

$$\varepsilon(\omega) = \varepsilon'(\omega) + j\varepsilon''(\omega) \tag{6}$$

The dielectric constant usually refers to static, zero-frequency relative permittivity. For general or dielectric media, both the real and imaginary parts of the impedance may be frequency dependent:

$$\mathbf{Z}(\boldsymbol{\omega}) = \mathbf{Z}'(\boldsymbol{\omega}) + \mathbf{j}\mathbf{Z}''(\boldsymbol{\omega}) \tag{7}$$

Electrical impedance spectroscopy (EIS) is the study of the character of impedance $Z(\omega)$ over different frequencies. It has been widely used for the following applications: investigating the behavior of materials (Dobbelaar and De Wit 1990), monitoring changes in the structure of cement paste (Gu and others 1993), determining the reaction rate in chemical reactors (De Oca and others 2001) and determining the physiological conditions of living tissues (Grimnes and Martinsen 2000). The application of EIS to fluid mechanics has been rather limited.

The flow of solid-liquid mixtures (slurries) is encountered in many industrial processes. There is significant interest in slurry transport through pipelines as an alternative to truck and rail transport. The phosphate mining industry makes extensive use of slurry flow to transport raw material from the mining pit to the processing facility. The electrical conductivity method has been previously used to determine the local solids concentration in mixing tanks and pipelines; see Nasr-El-Din and others (1996), Klausner and others (2000), and Micheletti and others (2003). The capacitance technique has been used by Brunazzi and others (2004) for flow characteristics and mixing properties in stirred slurry reactors. Kendoush and Sarkis (1995) used the capacitance technique to measure gas-solid two-phase flow characteristics. Dyakowski and others (1997) applied capacitance tomography to gas solid flows. They obtained on-line images of permittivity distribution.

The EIS technique combines both the conductivity and the capacitance method into a single device. A Nyquist plot is usually used to show the variation of the complex impedance with frequency in the complex plane. For example, consider a simple RC electrical circuit shown in Figure 2 (R=100 ohm; C=1×10⁻⁶ F); the corresponding complex impedance over a frequency range from 100 Hz to 100 MHz is shown as a Nyquist plot in Figure 3.



Figure 2. A Simple RC Electrical Circuit.



Figure 3. Nyquist Plot for the Simple RC Circuit.

A Nyquist plot provides a more direct view of the change of complex impedance over a specified frequency range. It is also the most popular data representation format for EIS studies.

Actually, for many real materials, the measured impedance has a similar Nyquist plot as that shown in Figure 3. So, instead of representing the electrical character of a material in the form or its conductivity σ or its complex frequency dependent permittivity $\varepsilon(\omega)$, another representation of the material property is to use an electrical analog that consists of a circuit with ideal resistors, capacitors and inductors. Such a representation is more efficient and easy to understand because all the components are

real valued and frequency independent. Such a representation is usually referred to as an equivalent circuit.

IMPEDANCE SPECTROMETER SELECTION

In order to use impedance spectroscopy for industrial characterization of slurry flow, the choice for purchasing an impedance spectrometer must satisfy a number of different criteria. Therefore, a very thorough comparison of different instruments was made as part of this investigation. Companies that manufacture and market impedance spectrometers include: Gamry Instruments, Novocontrol Gmbh, Solartron Analytical, and Agilent (HP). A comparison of the operating characteristics of the different instruments is shown in Table 1 below.

Manufacturer/ Model	Freq. Range (Hz)	10% Accuracy Range (Ω)	Additional Information
Gamry Instruments (US)/ Reference 600	1μ-1M	0.01-10 ¹²	Gamry software included.
Novocontrol Gmbh	3µ-40M	0.1-10 ¹⁴	Optional WinDETA control and
(DE)/			evaluation software. ZG2 interface
Alpha-A			needed.
Solartron Analytical	10µ-32M	0.1-10 ⁷	Optional ZPlot/CorrWare software.
(UK)/ 1260A			1296A dielectric interface needed.
	40-110M	0.025-4×10 ⁷	Free IntuiLink interface software
Agilent (HP, US)/			and Agilent I/O libraries. Full
4294A			programming instructions and
			other technical documents.

 Table 1. Operating Characteristics of Different Impedance Spectrometers.

The best instrument for on-line characterization of slurries will operate over a wide range of frequencies. The sampling time will be short, and the instrument will possess a data buffer than can be rapidly transferred to a processor for data analysis. The frequency range of Gamry Instruments/Reference 600 (see Table 1) is not sufficient for the current application and was screened out. One consideration is that low frequency operation requires a longer sampling time, and thus it is not likely that very low frequency operation of the spectrometer will be feasible for an on-line instrument. It is obvious that it takes longer time to sample data at lower frequency. The interfaces required by Novocontrol/Alpha-A and Solartron Analytical/1260A will cut their upper frequency range to 3μ Hz-20MHz and 10μ Hz-32MHz, respectively. Thus, the Agilent (HP)/4294A stands out for having the best frequency range. Agilent provides its own dedicated software, interface libraries and an assortment of peripheral equipment which enables the ability to easily transfer data from the machine to a PC processor for analysis.

Both the Solartron and Agilent machines were rated as high quality machines by users. The Solartron was rated as more user-friendly. However, the Solartron could not

capture the impedance data with sufficient speed, and the transfer of data to a PC would be difficult. Thus it was decided that the Agilent Model 4294A impedance spectrometer linked to a PC processor has the best operating characteristics to meet the goals of the project. An Agilent Model 4294A impedance spectrometer was acquired to complete this investigation.

DEVELOPMENT OF THE MEASURING SCHEME

Existing schemes for EIS analysis on materials are mostly done in a static environment. But with this project, the slurry being tested must be flowing, otherwise the solid particles in the slurry will settle down quickly. Unfortunately, most test fixtures sold by Agilent for the 4294A impedance analyzer are for use in a static environment. Therefore, it was necessary to custom fabricate a test section and slurry flow system for data experimentation. The design of the system has been upgraded numerous times for different flow configurations, test sections, wiring configurations, and processing algorithms.

PRELIMINARY TESTING WITH WATER BATH

Initial testing was done in a water bath in order to gain experience operating and collecting data with the 4294A impedance spectrometer. In order to validate the impedance spectrometer measurements, several non-flow experiments were conducted in an acrylic water bath with a stirrer. The initial test electrodes consisted of two parallel 2 cm diameter circular copper disks oriented face-to-face. A rough illustration of the configuration is shown in Figure 4.



Figure 4. Water Bath Electrode Configuration.

The benchmark for the present test is a Nyquist plot of impedance data for tap-water at 25°C reported by Coverdale and others (1993) shown in Figure 5. It is noted that its y-axis is -Z". The real and imaginary components of impedance were measured for tap-water at 25°C in the water bath using the 4294A Agilent impedance spectrometer. The results are presented as a Nyquist plot as shown in Figure 6. As observed, the comparison is quite good.



Figure 5. Nyquist Plot for Tap Water at 25°C.



Figure 6. Nyquist Plot for Tap Water at 25°C Measured in Water Bath Using Agilent 4294A Impedance Spectrometer.

PHOSPHATE AND SAND SLURRY CONCENTRATION MEASUREMENT

The utilization of the water bath described above proved unsuitable for impedance measurements of the slurry. The reason is that the rapid settling of the slurry biases the slurry concentration exposed to the measuring electrodes. The use of two stirrers inside the bath was insufficient to yield a homogenized slurry.

Therefore, a bench-scale slurry flow loop was fabricated. It is driven by a pump with a compatible test section. The utilization of an equivalent circuit analysis achieved some appealing results.

Configuration of the Slurry Flow System

A simple schematic diagram of the slurry flow loop is shown in Figure 7. The slurry is pumped through the facility using a centrifugal pump and stainless steel impeller with sufficient velocity to sustain an approximately uniform concentration in the test section. The transparent test section is constructed with Lexan tubing with an inner diameter of 19.1 mm (³/₄ in). Two diametrically opposed stainless steel bolts (hex head cap screw 9.5 mm (3/8 in - 16 tpi), 15.9 mm (5/8 in) length, fully threaded) were mounted flush with the inner tube wall to serve as the two electrodes for the slurry measurement as shown in Figure 8.



Figure 7. The Slurry Flow Experiment Loop.



Figure 8. Electrode Arrangement within the Test Section.

Figure 9 shows the electrical connection diagram between the 4294A impedance analyzer and the electrodes. The cables shown here are all coaxial cables with a shielding layer. This configuration solves the mutual coupling problem and reduces the effects of stray capacitance. The test fixture calibration and compensation were performed using a 100 ohm reference resistor supplied with the 4294A as specified by the manufacturer.



(b) Actual connection

Figure 9. Terminal Configuration and Electrical Connections.

Data Collection

The slurry flow facility has worked well to insure good mixing and has facilitated repeatable measurements using the impendence spectrometer. Precise amounts of solid constituents are measured using an electronic balance (precision up to 0.1 gram) and are added to a known volume of water in the flowing system in order to determine the solids fraction by mass.

Using amine concentrate (phosphate), rougher tails (sand) and tap water, slurries with known sand and phosphate concentrations are prepared. The amine and tail circuit water samples provided by Mosaic were not initially used to mix the slurries. Denoting the sand concentration as S and phosphate concentration as P, the percent mass of phosphate and sand in the solids can be calculated as P/(S+P) and S/(S+P) respectively, and the total solids concentration in slurry is S+P, provided phosphate and sand are the dominant solid constituents in the slurry. It is noted that there is no accumulation tank in the flow circuit, and thus the concentration dispersed through the transport line is relatively uniform.

For different values of S and P, a series of impedance spectroscopy measurements are made for calibration purposes. For each test result, a feature extraction method is used. Then a calibration surface is constructed. For the slurry mixture with unknown sand and phosphate concentrations, the impedance spectroscopy measurements utilize feature extraction, calibration, surface searching, and maybe interpolation, to determine the corresponding concentrations. Figure 10 shows a sample matrix of experimental measurements that need to be made for the various sand and phosphate concentrations. Each circle in the plane represents an experimental calibration point. Due to limitations on the load of the pump, the largest concentration considered is 30% by mass.



Figure 10. Experimental Calibration Matrix.

Slurries loaded with rougher tail (sand) concentrations of 0% (water only), 5%, 10% and 15% were analyzed. Tap water was used as the carrier. The measured data are shown in both three dimensions and two dimensions in Figure 11. Slurries loaded with amine (phosphate) concentrations 0%, 5%, 10% and 15% were also analyzed. Tap water was used. The data are shown in Figure 12.



Figure 11. Impedance of Slurries with Rougher Tail (Sand).



Figure 12. Impedance of Slurries with Amine Concentrate (Phosphate).

Slurries loaded with rougher tail (0%, 5%, 10% and 15%) but different carrier fluids were also analyzed. Two kinds of carrier fluids were considered; one is tap water and the other is amine circuit water from Mosaic. The purpose of the analysis is to explore the influence of the carrier on the impedance signal. The data are shown in Figure 13. As observed in the Nyquist plots, the impedance characteristics of the carrier fluid have a strong influence on the impedance characteristics of the slurry and must be accounted for in the data analysis.



Figure 13. Impedance of Slurries with Rougher Tail (Sand) in Different Carrier Fluids (Tap Water and Amine Circuit Water).

Impedance spectroscopy data were collected for the slurry concentration matrix shown in Figure 10. Using the impedance data for the calibration matrix with sand and phosphate slurries, an algorithm was developed to determine the sand and phosphate concentrations from the impedance data. This measurement algorithm is described next.

Data Representation

Typically, the raw data acquired from the impedance analyzer is a series of complex impedances measured over a fixed frequency range. For a given slurry sample, the measured raw data are denoted as:

$$D = \{D(f_i) \mid i = 0, 1, ..., n\}$$
(8)

where D is a complex impedance. For the current Agilent analyzer, n = 604 frequency intervals is used. This impedance series is redundant and requires processing in order to be useful. In order to provide a fundamental representation of the signal, an equivalent circuit analysis technique is adopted and a simplified RC parallel model, shown in Figure 14 is chosen.

The equivalent impedance for this model is computed as:

$$I(\omega) = \frac{R}{1 + (\omega CR)^2} - j \frac{\omega CR^2}{1 + (\omega CR)^2}$$
(9)

or

$$I(f) = \frac{R}{1 + (2\pi fCR)^2} - j \frac{2\pi fCR^2}{1 + (2\pi fCR)^2}$$
(10)

In order to fit this model with the actual measured data, an optimization technique is required. The values of R and C which satisfies the least mean square criterion are chosen as the representative parameter of the measured data. Mathematically, this is expressed as:

$$(R,C) = Arg \min(\frac{1}{n} \sum_{i=1}^{n} I(f_i, R, C) - D(f_i)^2$$
(11)

A typical fitted result with a prepared slurry mixture containing 5% amine by weight is shown in Figure 14. As observed, the fit is quite good over a wide range of frequency.



Figure 14. Typical Impedance Characteristics of a 5% Amine Slurry Compared with Equivalent Circuit Simulation.

Concentration Estimation Algorithm

Using the equivalent circuit analysis described above, the impedance character of the slurry is represented by two parameters R and C. The next step is to make use of these two parameters and determine the concentrations of sand and phosphate constituents in the slurry.

Here several assumptions are made: (1) both sand and phosphate will contribute to both the R and C characteristics of the slurry; (2) the dependence of R and C on the concentration will be different for sand or phosphate; (3) their respective contributions to R and C are linearly additive; and (4) R and C are monotonically related to the respective sand and phosphate concentrations. Then, R and C for slurry can be estimated using the following model:

$$\begin{cases} C(S,P) = C_0 + C_s(S) + C_p(P) \\ R(S,P) = R_0 + R_s(S) + R_p(P) \end{cases}$$
(12)

where *S* and *P* are the concentrations of sand and phosphate respectively, while R_0 and C_0 are the respective R, C values of the carrier fluid. $R_s(S)$, $C_s(S)$ are the contributions to R and C from the sand concentration with no phosphate, and $R_p(P)$, $C_p(P)$ are the contributions from phosphate to R and C with no sand.

Information on the functions $R_s(S)$, $C_s(S)$, $R_p(P)$ and $C_p(P)$ can be acquired through calibration. For slurry loaded with only sand at a known concentration, the impedance spectrometer data are collected and the corresponding R and C values are extracted. Then the slurry concentration is increased in steps, and the measurement procedure is repeated for each step. By interpolating values between steps, it is possible to get two mappings of $R_s(S)$ versus S and $C_s(S)$ versus S. Using this procedure and Equation (12), it is possible to determine the sand contribution to the overall impedance character of the slurry. A similar calibration is done for slurry loaded only with phosphate. In the same way, mapping of $R_p(P)$ versus P and $C_p(P)$ versus P is carried out.

In order to determine the respective sand and phosphate concentrations for slurry with unknown composition, the following procedure is employed. Impedance data are collect on the unknown slurry, and R* and C* are extracted using the equivalent circuit analysis. Then an estimate the sand concentration s and phosphate concentration p is made by solving the following optimization problem:

$$(s, p) = Arg \min\{\left(\frac{R^* - R(S, P)}{R^*}\right)^2 + \left(\frac{C^* - C(S, P)}{C^*}\right)^2\}$$

$$= Arg \min\{\left(\frac{R^* - R_0 - R_s(S) - R_p(P)}{R^*}\right)^2 + \left(\frac{C^* - C_0 - C_s(S) - C_p(P)}{C^*}\right)^2\}$$
(13)

 R^* and C^* in the denominator are used to normalize the errors from R and C, so that errors in R and C will have equal contributions to the target function. Otherwise, since values of R are usually much greater than values of C, differences between R^* and R(S,P) will overwhelm the difference from C^* and C(S,P). Physically, this mathematical procedure identifies the values of sand concentration s and phosphate concentration p which lead to the best match between the estimated values of R,C using Equation (12) and those measured values, R^* , C^* .

Temperature Compensation

In actual deployment of an impedance sensor for phosphate mining applications, the temperature of the slurry will vary over time. It is well known that R and C are temperature-dependent, and thus it is necessary to introduce a correction for temperature into the calibration. To consider the temperature effects, Equation (12) is re-written as:

$$\begin{cases} C(S, P, t) = C_0(t) + C_s(S, t) + C_p(P, t) \\ R(S, P, t) = R_0(t) + R_s(S, t) + R_p(P, t) \end{cases}$$
(14)

For this stage of calibration, all impedance calibration measurements are repeated for different temperature conditions. This includes measuring the impedance characteristics of only the carrier fluid at different temperatures in order to construct a mapping between R_0 , C_0 and t. Also, by measuring the impedance characteristics of known slurry concentrations of sand or phosphate at different temperatures, mappings are constructed for $R_s(S_i, t)$ versus t, $C_s(S_i,t)$ versus t, $R_p(P_i,t)$ versus t and $C_p(P_i,t)$ versus t, i=1,2,...,n. When changes of concentration are considered, calibrations of $R_s(S, t)$ versus $(S, t), C_s(S,t)$ versus $(S, t), R_p(P, t)$ versus (P, t) and $C_p(P, t)$ versus (P, t) are required. An example is shown in Figure 15 below.



Figure 15. An Example Showing the Mapping Between R, C and S, P, t.

Here different lines denote different sand or phosphate concentrations. The slope of each line delineates the variation of R and C with changes in the slurry temperature at uniform concentration.

When measuring an unknown slurry at known temperature t^* , through interpolation, the above mappings can be degenerated to lower dimensional mappings $R_s(S, t^*)$ versus S, $C_s(S,t^*)$ versus S, $R_p(P_i,t^*)$ versus P and $C_p(P_i,t^*)$ versus P. Then the same procedure as described in the previous section is adopted for estimating the slurry sand and phosphate concentrations respectively. The whole calibration system is shifted to the same temperature basis as the unknown slurry to essentially eliminate the temperature influence.

Evaluating the Fidelity of the Concentration Estimation Algorithm

To test the measurement algorithm, some test samples of sand and phosphate slurry mixtures with different concentrations were prepared: (S,P) {(3%, 3%), (6%, 3%), (9%, 3%), (12%, 3%), (3%, 6%), (6%, 6%), (9%, 6%), (3%, 9%), (6%, 9%) and (3%, 12%)}.

Then the impedance spectrometer was used to collect data and estimate R^* and C^* . From these the concentrations S and P were determined using the algorithm described above. The results are shown in Figure 16.



Figure 16. Comparison of the Known and Estimated Concentrations.

The agreement between the known and estimated concentrations is satisfactory. As is observed, there is slightly better agreement for the phosphate concentration than that of the sand. It is also necessary to point out that the calibrations are made based on the mass fraction of sand and phosphate flowing through the test section. However, the impedance is theoretically dependent of the volume fraction of different constituents. So, in order to get a more precise calibration, it would be necessary to calibrate the system with volume fraction as opposed to mass fraction. Nevertheless, the procedure which has been presented is satisfactory for demonstrating the potential of using impedance spectroscopy to make online measurements of mineral constituents.

MEASURING SCHEME INDEPENDENT OF CARRIER FLUID

The main shortcoming with the mineral concentration measuring algorithm described in the previous section is that the impedance characteristics of the carrier fluid, R_0 , C_0 must be known. However, it is well known that the impedance characteristics of the

carrier water used for phosphate mining operations change with time. In order to make use of the measuring scheme described in the precious section, it is necessary to either continuously measure R_0 , C_0 in a separate flow circuit or deduce a method for electronically compensating for R_0 , C_0 . The latter approach is pursued here, and a description of its implementation is described next.

Through extensive impedance spectroscopy testing, it has been observed that the presence of solid particles in the slurry influence the impedance characteristics much more significantly at low frequency than at high frequency. The reason for this is that at high frequency the reactance is significantly diminished as suggested by Equation (4). Thus, electrical charge can readily pass through the solid materials. This is pictorially illustrated in the Appendix. Thus, the starting point for identifying a calibration scheme for the carrier fluid is to discriminate between the high and low frequency impedance signals. However, the electrode configuration employed does not provide a sufficiently strong signal to get good low frequency measurements. Thus it was necessary to develop of an improved electrode arrangement that yields a better quality signal at low measuring frequencies. Different materials (iron, copper and aluminum), shapes (rectangular and circular) and wiring (solid wire, multiple wires, shielded and unshielded) were tested. The tests showed that the factor which has the greatest influence on the measurement is the separation distance to area ratio, d/A, where d is the distance between two electrodes and A is the area of the electrode surface. Small values of d/A are usually preferred for impedance spectroscopy measurements. It is especially important for the dynamic environment and the low frequency range. When the value of d/A is large, the measurement at low frequency is less stable due to the motion of particles and ions. A large surface area can better average out the difference from particle distributions. A small distance creates a shorter path for ions to travel when polarization occurs, which is better for low frequency measurements. Since the influence from temperature is easy to be compensated with careful calibration, all the experiments here are run at 26°C.

Construction of New Test Section

The slurry flow loop configuration was maintained, and a new test section was fabricated for the slurry flow loop. The new test section was constructed with a pair of parallel rectangular copper sheet electrodes. A cross sectional view of the new test section is shown in Figure 17 and a photograph of the actual test section is shown in Figure 18.



Figure 17. Cross Sectional View of the New Test Section.



Figure 18. Photograph of the New Test Section.

The wiring configuration for the new test section is shown in Figure 19. The wires connecting to the impedance analyzer are fixed to two copper electrodes and are adhered with solder. Figure 20 shows the impedance characteristics of tap water measured with the new test section and different wiring configurations. The new electrode configuration has a very large area to separation ratio, 117mm×31mm/8mm, which is good for tracking the low frequency behavior of the slurry. As seen in Figure 20, different terminal configurations

and wirings do not significantly influence the measurement. The large tail that goes to the negative y phase is modeled using an inductor and resistor combination. It is due to the inductance of the wires at very high frequency.



Figure 19. Terminal Configuration. DUT Denotes Device Under Test.



Figure 20. Sample Plots of the Impedance Characteristics of Tap Water Measured in the New Test Section with Different Wiring Configurations.

Parametric Scheme

A physical phenomenon can usually be modeled mathematically with a specified set of parameters. The method that analyzes the phenomenon in the parameter space instead of raw data space is called a parametric scheme. The method described in the previous section for analyzing the slurry constituent concentration is a type of parametric method since it is based on a RC parallel equivalent circuit and estimates the concentrations based on the parameter values of R and C.

In order to develop a measuring scheme that is independent of the carrier fluid impedance characteristics, both high and low frequency measurements need to be modeled. An updated equivalent model is shown in Figure 21 below, and is adopted to represent the measurement with abstracted parameters shown in lieu of hundreds of values.



Figure 21. Updated Equivalent Circuit Model for Slurry Impedance Characteristics.

In this model, the constant E is the measurement bias from the impedance analyzer, L and R3 are used to model the influence from connecting wires at very high frequency, R1 and C1 are used to model the bulk slurry impedance characteristics at low frequency and R2, C2, and Z_w are used to model the double layer capacitance for high frequency. The Warburg impedance is computed as follows, $Z_w=1/[y_0(j\omega)^{0.5}]$. Figure 22 compares the measured impedance characteristics of 2% sand slurry with those predicted using the equivalent circuit model shown in Figure 21. The parameters of the model are acquired through a least squares fitting procedure. As observed, the equivalent circuit model fits the data very well.

In Figure 22, it is obvious that at different positions of the measured Nyquist plot, the density of data collected near a given frequency varies greatly over the entire range of frequency. If all the data readings are given the same importance, the parts with high point density, such as the part around the corner, will dominate the fitting algorithm and lead to a large bias error at other places, such as the low frequency portion. To solve this problem, a weighted curve fitting is used. A weight is assigned to each data reading according to the distance from its neighbor. The larger distance means the less density so the larger weight is assigned.

Using the circuit model parameters obtained from the fitted plot shown in Figure 22, the contribution to the impedance characteristics from different model parameters is easily illustrated as shown in Figures 23, 24, and 25.



Figure 22. Measured Impedance Characteristics of 2% Sand Slurry Compared with the Equivalent Circuit Simulation.



Figure 23. Contribution of (R3, L) to the Model.



Figure 24. Contribution of (R2, C2, Zw) to the Model.



Figure 25. Contribution of (R1, C1) to the Model.

The impedance from these three components is additive, as shown in the circuit diagram, Figure 21. Thus the summation of these three plots will reproduce the original Nyquist plot shown in Figure 22.

Some experimental data were collected on slurries flowing through the experimental facility consisting of tap water, tap water with sand (1%, 2%, 3% and 4%), and phosphate dissolved carrier, PDC (tap water mixed with 5% phosphate and then filtered, leaving behind only the water carrier), PDC with sand (2% and 5%), tap water with K_2SO_4 (different concentrations) and tap water with KH_2PO_4 . The resulting Nyquist plots are shown in Figure 26 below.



Figure 26. Measured Impedance Characteristics for Different Slurry Samples.

All the concentrations reported are the volumetric concentration of material added to the system. These were determined based on the total volume of the circulation system V_t which is a constant, 896 mL, the density of the rougher tail (sand) d_s , the density of the amine concentrate (phosphate) d_p , and the measured weight of sand and phosphate added w_s , w_p . The volumetric concentration of sand and phosphate added to the system are S and P, respectively. They are calculated as:

$$\begin{cases} S = \frac{w_s}{d_s V_t} \\ P = \frac{w_p}{d_p V_t} \end{cases}$$
(15)

where $d_s = 2.65$ g/mL and $d_p = 2.72$ g/mL. It should be noted that these volumetric concentrations are not necessarily the volume fractions flowing through the test section. The actual volume fractions flowing through the test section depend on the respective velocity differences between water, sand, and phosphate.

The goal here is to estimate the impedance characteristics of the carrier portion of the slurry. As already mentioned, the carrier is the liquid portion of the slurry and the remaining part is solid particles. As shown in Figure 26, the solid particles affect the impedance characteristics of the slurry mainly in the low frequency range, while the dissolved ions in the carrier maintain their influence over the whole frequency range. Then, it is reasonable to make 3 assumptions as described below:

- (a) The high frequency impedance characteristics are determined by the carrier (ion concentration, ion type, temperature, etc.)
- (b) The low frequency impedance characteristics are determined by the carrier plus the solid particles (permittivity, size, concentration, etc.)
- (c) Similar carriers have similar have similar relationships between their contribution to the high frequency and low frequency range measurements.

The very high frequency portion of the signal (the negative tail) can be ignored because it is due to the impedance characteristics of the connecting wires, which should not change with different slurries. To further simplify the analysis, the focus will initially consider only one solid constituent in the slurry. For assumption c), the term similar carriers refer to the same type of liquid with similar chemical ions dissolved in solution at the same temperature. In actual practice, a temperature compensation scheme can be implemented. Also, the requirement for the carrier to hold the same chemical ions should be satisfied since water feeding the matrix or tailing lines is recirculated.

With all these assumptions, the impedance measurement of slurry should only differ from the measurement of its carrier in the low frequency range. In addition, the high frequency range measurement of the carrier impedance characteristics can be used to estimates the carrier contribution to impedance in the low frequency range. Referring to the equivalent circuit model in Figure 21, the problem becomes one of estimating the (R1', C1') parameter of the carrier from the (R2, C2, Zw) parameter of the slurry. Denote (R1,C1,R2,C2,Zw,R3,L,E) as slurry parameters and (R1',C1',R2',C2',Zw',R3',L',E') as parameters of the carrier. According to previous assumptions, L=L', E=E', R3=R3', Zw=Zw', R2=R2', C2=C2'. The problem can be analyzed on parameter planes such as R1 versus R2 space as shown in Figure 27.



Figure 27. Model Fitted Estimations of R1 and R2.

First it is assumed that the relationship between the parameter R1 and R2 of similar carriers is fixed, that is R1'=f (R2'). And from the previous assumptions we know that R2'=R2. Then given an unknown slurry, the value of R2' can be obtained from a given point in the plane. Accordingly, for R1'= f (R2), the value of R1' is estimated. If only one kind of solid particle is considered, its concentration could be estimated based on the value of R1-R1'. If two kinds of solid particles are considered, it could still be solved when we do the same estimation on C1' and get the value of C1-C1'.

Unfortunately, Figure 27 shows that there is no obvious pattern for the behavior of carriers or slurries. This could be due to the inaccuracy of the equivalent circuit model, improper combination of parameters, or independence among parameters. Thus, another scheme is required.

Non-Parametric Scheme

The non-parametric method tries to solve a problem based on the raw data. No assumptions regarding a model or parameter are required. It is typically more robust than the parametric method, but the parametric method could achieve a better solution when the model is physically accurate.

Instead of focusing on a complicated equivalent circuit model, an attempt has been made to use a non-parametric analysis on the raw data, and an appealing solution has been identified. It is described next.

This scheme is also based on the fact that solid particles influence the impedance of the slurry more significantly at low frequency than that at high frequency as illustrated in the Appendix. However, instead of fitting the collected data to a model and doing analysis on the parameters assigned to high and low frequency segments, one direct impedance reading at a given high frequency point and another direct reading at a given low frequency point are extracted as the representations of the high and low frequency electrical characteristics of the slurry. The two frequency points are chosen to be 25.56 MHz and 1 MHz. The values are chosen so that the imaginary parts of the corresponding impedances are very small. The two real parts are plotted as points in a plane of one versus the other.

A series of experiments are done on different carriers mixed by tap water (TW) and rougher circuit water (RCW) with volume percentage of RCW changing from 10% to 100%. The step size is 10%. For each carrier sample, two concentrations (2% and 5% by volume) of sand are added and measured. Figure 28 shows the Nyquist plots for the data collected.



Figure 28. Impedance Characteristics of Different Carriers and Sand Concentrations.

Then the real parts of impedances at 1 MHz and 25.56 MHz for each sample are plotted in Figure 29. The data on the blue line correspond to different carriers without sand added. As observed, all of the carrier data represented by (Z'_L, Z'_H) follow the same pattern,

where Z'_L is the real part of impedance at 1 MHz, and Z'_H is the real part of impedance at 25.56 MHz. The slurry data with 2% sand and 5% sand follow their own patterns, respectively. This implies that carriers of the same components but different concentrations follow the same pattern in the plane of Z'_H vs Z'_L . Adding sand to this type of carrier will make the corresponding points of the slurry biased from the base curve by a discreet distance. With enough calibration data, this base curve can be accurately constructed, and the relation between the concentration of sand and the distance of (Z'_L, Z'_H) from the base can be determined. After calibration, for any unknown slurry, measurements of only the real parts of impedances (Z'_L, Z'_H) at the two given frequencies can be used to determine the distance of (Z'_L, Z'_H) to the base curve, and the concentration of sand can be estimated. There is no need to know the carrier information at the testing stage to determine the sand concentration using this scheme.



Figure 29. Plots Showing the Pattern of Carriers.

In Figure 29, another line with rectangular points shows the (Z'_L, Z'_H) data for carriers with tap water and different concentrations of K₂SO₄. The amounts of K₂SO₄ added to 930 mL of tap water are 0g, 0.1g, 0.2g, 0.3g, 0.4g and 0.5g. It is observed that even with a completely different carrier family, the relationship in the (Z'_L, Z'_H) plane still holds. This result is very appealing because it suggests that for all kinds of carriers a simple field calibration will allow the determination of the solids concentration from a low and high frequency measurement of the real part of the impedance.



Figure 30. Impedance Pattern for the Carriers Consisting of TW+RCW.

Figure 30 shows that the base curve of carriers of different concentrations of tap water plus rougher circuit water can be approximated by a polynomial as:

$$\mathbf{Z'}_{H} = 0.0072 \mathbf{Z'}_{L}^{2} - 0.4782 \mathbf{Z'}_{L} + 16.7120$$
(16)

For an unknown slurry sample corresponding to the point marked by " \mathbf{x} ", the concentration of sand can be estimated according to its distance, d, to the curve represented in Equation (16). If all the solid particles in the slurry are of high capacitance and low conductance, the estimation of sand concentration can be generalized to the estimation of solid particles.

SUGGESTED FUTURE RESEARCH

In order to carry the current investigation forward to develop a solids concentration measurement instrument suitable for field operations, the following should be considered.

- It is important to consider practical implementation of test section so that it can survive in the erosive slurry flow environment. Key issues include determination of the size and distribution of electrodes, as well as the circuit design and operating frequencies.
- An algorithm has been developed to determine the total solids concentration without knowing the impedance characteristics of the carrier water. The next challenge is to combine this algorithm with the one used to discriminate the concentration of different constituents in the slurry.
- It will be quite useful to design and fabricate a solids concentration sensor for field operations and test its performance over a wide range of operating variables.

CONCLUSIONS

Considering all the experiments and data analyses presented, the following conclusions are made: (1) when the impedance characteristics of the carrier are known, an equivalent circuit analysis can be used to estimate the solids concentrations of sand and phosphate respectively; (2) a non-parametric scheme has been identified which may be used to estimate total solids concentration in real time without measuring the impedance characteristics of the carrier; (3) real-time carrier-independent estimation of sand and phosphate concentrations is possible, but further experiments are needed for verification. With some additional effort is should be feasible to design and fabricate a solids concentration sensor that can be used for both phosphate mining and beneficiation applications.

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Appendix

INFLUENCE OF SOLID PARTICLES ON IMPEDANCE CHARACTERISTICS OF SLURRY

Grimnes and Martinsen (2000) have reported on the electrical properties of slurries. Consider a simple case of a volume containing many particles in water. Some particles are considered to have a high capacitance and a low conductance. At low frequencies (or DC), current must pass around these particles. At higher frequencies, the particle capacitance lets AC current pass. This is illustrated in Figure A-1 below.



Figure A-1. Electrical Properties of Slurries.

Sand and phosphate rock particles have a high capacitance and low conductance. At low frequency, these particles block the current and decrease the conductivity of the slurry, i.e. the resistance of the slurry increases. At high frequency, the blocking effect is less notable because the current can pass through the particles. This explains why the plot of slurry with sand is above the plot with water only at low frequency but merges with that at high frequency. But for amine concentrate, the plot is below the plot of water, which means the slurry has lower resistance and higher conductivity. The reason may be the amine concentrate sample can provide multiple types of ions, which significantly increases the conductivity and decreases the resistance. And according to the previous properties of the slurry, the ions affect the character of the slurry at both low and high frequency.

