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SOLIDS CONTROL IN SOLVENT EXTRACTION CIRCUITS USING ELECTROSTATIC DISPERSION

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Solids Control In Solvent Extraction Circuits Using Electrostatic Particle Dispersion

Abstract: Elevation of surface charge density on colloidal particles in the presence of a high potential electrostatic field is demonstrated as a viable technique for preventing the accumulation of solids in solvent extraction circuits. The effect of the technique is manifest by relative turbidity measurements between pregnant leach solution and raffinate, by changes in the character of the sludge accumulations, by alteration of the zeta potential of particulates, and by a general reduction in the mass of solids.

Introduction

Solids accumulation in solvent extraction circuits is a problem common to all operating solvent extraction plants. Suspended solids bind with dilute extractant to form masses of sludge in the extractors, strippers, or both. The degree of severity of the accumulation depends primarily upon the leach technique and operating conditions of the circuit. Ritcey (1980) (1983), Loesch (1981) and Burniston (1991) point out that sludge is principally silicate mineral matter bound by organic from within the circuit. Most solids enter the system in suspension with the pregnant leach solution (PLS) and some components precipitate from solution. The severity of the problem of sludge accumulation varies with the level of solids in the PLS, which in turn is a function of the type of leach employed. Regardless of the circuit, the presence of accumulated sludge invariably results in increased costs from extractant losses, as well as labor and capital costs associated with control efforts.

Agitated leach operations seem to generate the most difficult to control levels of solids, but even the relatively clear PLS produced by heap and dump leach operations contain enough suspended solids to produce large masses of sludge; if at a somewhat slower rate of accumulation.

In heap leach and dump leach operations, where the leach liquor collection point is subject to receiving runoff, surges in runoff associated with storm conditions can cause severe plant upsets. Efforts to control suspended solids in these operations have focused on gravity settling in surge ponds, thickeners, or clarifiers.

Kordosky and others (1987) discuss the efforts of extractant manufacturers to formulate products that avoid the use of compounds that are known to exhibit strong affinity for bonding with solids.

Traditionally, removal of accumulated solids from mixer-settlers has been accomplished by physical separation techniques such as gravity settling and centrifugation. The solids mass is pumped from the settlers with a hand directed suction or decanted into a vessel for centrifugation and extractant recovery (Burniston, 1991). Significant amounts of system extractant are lost from circulation because they are bound up in the sludge mass. Reclaiming by centrifuging accounts for high levels of extractant loss as well as the associated expenditure of capital and labor.

The agitated leach circuit operated by Cyprus at the Twin Buttes Oxide Plant in Green Valley, Arizona is characterized by a strong propensity for sludge accumulation in the mixer-settlers of its four extractor and two stripper circuit. This study was initiated in response to the need to find a method of preventing the costly accumulations.

Electrostatic Deposit Control

For some time researchers have known that the surface charge of particles in suspension could be altered by exposure to high potential electrostatic fields (Means, 1975). Indeed, this phenomenon forms the basis for such well known applications as electrostatic precipitators, photocopiers, and certain paint spraying techniques. Means (1975) and Reimers (1980) review successful applications of electrostatic fields in boiler and cooling water treatment, as well as prevention of scale in industrial and commercial water systems.

Means (1975) discusses in detail the fundamental principals underlying the observed particle dispersion. The dispersion described is the result of elevated levels of induced like charges on the surface of sub-micron particles that result in mutual repulsion.

The electrical potential across the double layer of wetted surfaces (zeta potential) is most often discussed in conjunction with coagulation/flocculation experiments. Applications of zeta potential monitoring at present are most common in the treatment of water supply and waste water. Segments of the paper industry control the additives responsible for improved water drainage from fourdrinier paper machines through on-line monitoring of zeta potential (Penniman 1991).

Alternating current high potential electrostatic fields (electrical coalescors) are used for breaking of water-in-oil emulsions in crude oil production. Electrical coalescors are included as a key system component in experiments with emulsion membranes reported by Nilsen and Hundley (1991).

In the cases mentioned above, surface charge manipulation by various styles of electrostatic fields is being used for dispersion, coagulation, and coalescence.

In the experiment presented here we rely upon altering the surface charge of submicron particles to accomplish dispersion. In suspension, organic or inorganic particles may assume a polarity and charge density peculiar to the particular substance (Hiemenz, 1977). The work reported here is an effort to use these charged particle characteristics to reduce or eliminate sludge accumulation.

Setting the Hypothesis

If the assumption that organic and inorganic surface charge is the responsible mechanism promoting particle agglomeration and aggregation of sludge mass, then it follows that anything done to alter the charge differentials that promote bonding of suspended particles with organic compounds would serve to establish a stable dispersion of solids in the PLS. If the dispersion were sufficiently stable, then the flocculent-style bonding process would be inhibited and particles of suspended solids entering the system would transit through the circuit without forming sludge.

An hypothesis was thus postulated that the dispersive effect of an electrostatic field would be effective as a method of controlling or eliminating sludge formation in solvent extraction circuits.

Evaluation of the hypothesis could be performed by observing the turbidity of the PLS as it passed through the system to be discharged as raffinate. If the solids exiting the system could be made to equal or exceed the solids entering the system, over a significant period of time, then the hypothesis would be supported.

Goals and procedures to evaluate the experiment were developed into a three part program. The initial phase of the test was observation and measurement of induced charge density changes within the system. The second phase of the test was a six month observation period to record relative treatment effects under varying operating conditions for comparison with data from the previous year. The third phase involved econometric evaluation of the data to formally establish that treatment had in fact induced a change in suspended solids transport.

Charging units manufactured by York Energy of Ontario, Canada under the trade name "Ion Stick" were installed April 15, 1991 and data was accumulated until Oct 10, 1991. Because no information relevant to electrostatics as applied to solvent extraction solids control was available, the choice of the number of units to be installed was extrapolated from guidelines used in designing applications in closed recirculating cooling water systems. Based on the prevailing PLS flow rate, four charging units were installed at Twin Buttes, peripheral to the suction of the PLS feed pump.

Characterizing Zeta Potential

For the first stage of test work, an instrument for measurement of zeta potential was obtained from Komline-Sanderson, Peapack, New Jersey, along with specific recommendations for sample handling and preparation.

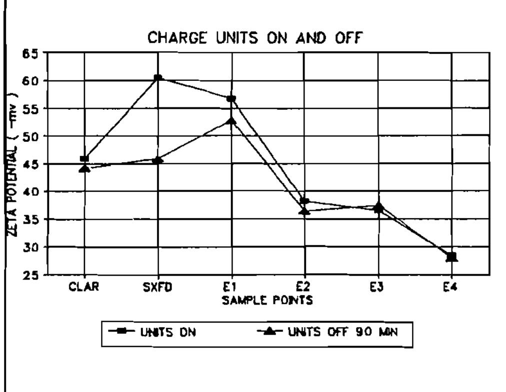

Figure 1

Sets of zeta potential measurements were taken to measure the charge density of the suspended solids before and after exposure to the electrostatic field. Zeta potential relative to turbidity, time and existing sludge deposits was determined, as well as the magnitude of the zeta potential level resulting from the number of electrostatic units employed.

Figure 1 illustrates sets of zeta potential readings obtained across the system before and after disconnecting the charging units for a period of 90 minutes. The transit time through the extractors is approximately 30 minutes. Because the charging units were installed around the periphery of the suction of the PLS feed pump, the clarifier sample is untreated. With removal of the electrostatic field, the PLS feed sample charge density falls to near that of the clarifier.

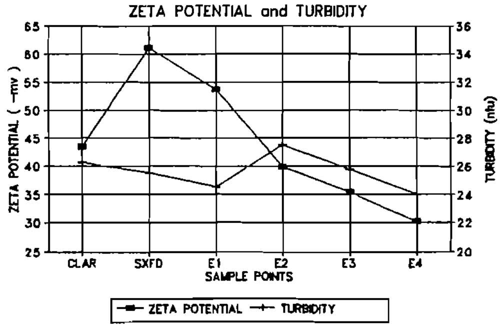

Figure 2

The downstream loss of zeta potential is pronounced, reflecting the effect of contact with the existing positively charged solids mass. This pattern of charge elevation and decline was highly reproducible and prevailed throughout the various test series.

Figure 2 plots the average relationship between observed charge density and observed turbidity over a five day period. Elevated turbidity readings are indicative of PLS that contains higher levels of suspended solids. A decline in turbidity associated with a decline in zeta potential is in line with the hypothesized relationship wherein particles, having lost their mutual repelling charge density, will bond together and settle, or become attached to existing sludge deposits.

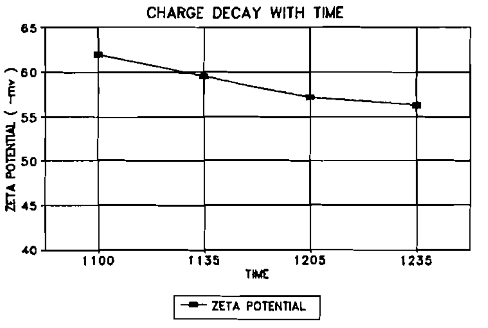

Figure 3

Of additional importance was determination of the way time effects charge decay. If the charge decay over transit time through the circuit was significant, then the test data would need to be evaluated with respect to this decline. Figure 3 illustrates the charge decay of an individual sample, taken from the discharge side of the pump, at various intervals over a 95 minute period; a period longer than the average transit time through the circuit. The degree of charge dissipation over time was insufficient to materially alter any of the observed relationships.

In an effort to define the high potential electrostatic field necessary to sustain an effective level of particle charge induction, measurements of PLS zeta potential were recorded five minutes after the sequential activation of each of the four Ion Sticks. Figure 4 describes the curve generated as the charging units were sequentially turned on.

The Nature of the Data

Turbidity readings are part of normal system monitoring procedures at Cyprus Twin Buttes and have proven to be an effective indicator of the relative activity of sludge accumulation in the four extraction stages.

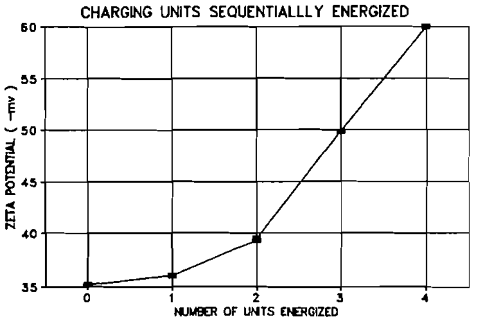

Figure 4

The background data used in this study was collected principally during the last half of 1990 and was accumulated from a routine that produced four to six sets of samples for turbidity observation each week. Background turbidity data was therefore available for clarifier overflow, PLS feed, and raffinate as well as PLS flow rates and organic flow rates. Clarifier turbidity and organic flow rates were not usable in the regression analysis because they are perfect linear combinations of PLS turbidity and aqueous flow rate respectively.

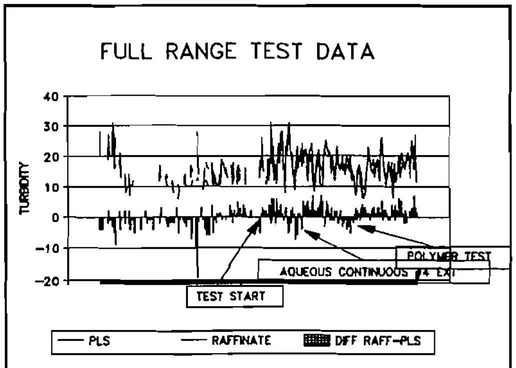

Figure 5

Turbidity readings from the test period are an average of samples obtained twice daily. They include samples from the clarifier, PLS feed pump, extractor #1 PLS underflow, and raffinate as well as PLS flow rate.

If the treatment performed as hypothesized, the raffinate turbidity would remain approximately equal to the PLS feed turbidity as solids transport through the system improved. Raffinate turbidity higher than the PLS feed would indicate erosion of the existing solids mass.

The earliest observed effect on the system was the nearly complete disappearance, within four weeks, of a heavy layer of floating sludge that had previously covered more than half the surface of the settlers. Additionally, sediment on the bottom of a unit that was taken down for cleaning was found to have become more fluid, thus making the cleanout an easier task. These were encouraging signs early in the study, as sufficient turbidity samples and data had not been collected to begin constructing the econometric models necessary for evaluation of the data.

There were two periods of system upset during the treatment period that would be considered non-normal operating events. A period of aqueous continuous operation and a period of flocculent overfeed are factored into the regression models by introduction of appropriate "dummy" variables. Shortly after start up of the experiment, operating requirements necessitated an increase in the PLS flow rate. The flow rate increased from 13.63 M^3/min (3600 gpm) and generally ranged close to 18.94 M^3/min (5000 gpm) for the remainder of the test. The increased hydraulic throughput was coincident with an increase in suspended solids from the thickeners. Intuitively one would surmise that there would be a strong correlation between flow rate through the thickeners and suspended solids levels in the PLS. However, modelling later revealed that the suspected correlation in this case was quite low.

Dramatic near-term improvements in the condition of the system were not likely in the face of such a sizable increase in the control task, regardless of the source of the increased loading. Regression modelling provides a way to demonstrate both quantitative and qualitative relationships among variables, allowing comparison of data across time and under different treatment and/or operating conditions.

The most significant variables that covered both the non-treatment and the treatment period were raffinate turbidity and PLS turbidity. Figures 6, 7 and 8 represent the results of the regression of these variables over the shorter time frames of the background period and the treatment period. These data were later pooled to demonstrate the overall treatment effect and the influence of operational upsets.

Figures 6 and 7 present a visual comparison of the change between the background period and the treatment test period. Introduction of the electrostatic charging units tightened the scatter of datapoints, increased the slope of the regression plot and moved the intercept toward zero.

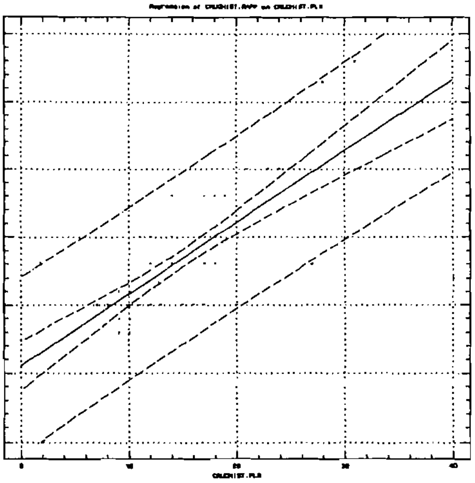

Figure 6 Untreated system. Raffinate turbidity regressed on PLS turbidity.

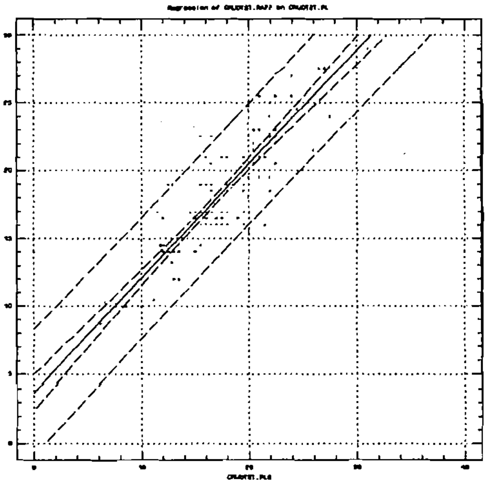

Figure 7. Treated system. Raffinate regressed on PLS.

Figure 7 and 8, together, present a view of the change that takes place across the extractors as the elevated zeta potential is lost through contact with existing sludge deposits. Hypothesis testing would later reveal that there is a horizontal shift in the regression plot caused by charge decay, along with an increase in scatter of data points between the first and last extractor. The installation of additional Ion Sticks between the second and third mixer-settlers should offset this loss and provide a more positive removal mechanism for sludge in inventory. In treating a system with fewer stages, concentration of the total treatment at a single point in the PLS feed line would likely yield the best results.

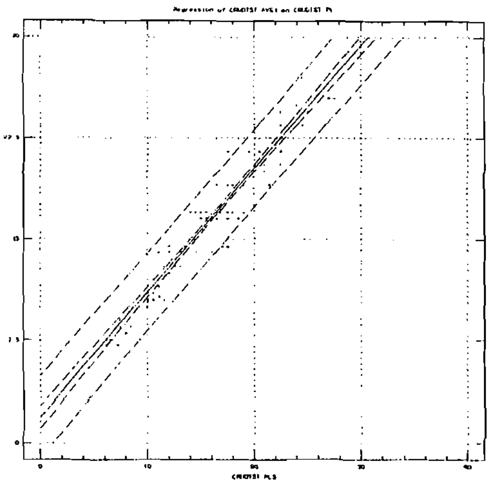

Figure 8. Treated System. Underflow turbidity from last extractor regressed against PLS feed turbidity.

Testing of The Hypothesis

Evaluation of the results of the experiment was accomplished by hypothesis testing of the parameters from models developed with multiple regression techniques. Econometric models were developed using background and test period data. The data was pooled for a test of all variables and all parameters across time (Equation 1). Unpooled data set usage (Equations 2 and 3) was necessary principally because there was no background corollary to the first extractor turbidity readings.

Multiple regression modelling allows the simultaneous evaluation of the treatment effect along with the impact of system upsets and operational changes. This method of simultaneously evaluating test data gauges the interrelationships of dependent and independent variables, providing insight into the interactions of system components and has revealed other subjects for future investigation.

Reliable hypothesis testing requires that the regression model be applied to data that is free of certain problems that if left uncorrected will violate the basic assumptions behind regression analysis, causing lost precision and thus inaccurate test results. Chief among these problems to be avoided are multicollinearity, serial correlation of error terms, and the problem of non-constant variance known as heteroscedasity.

As mentioned earlier, the avoidance of perfect multicollinearity among variables negated the use of clarifier turbidity and organic flow rate readings. Imperfect multicollinearity is not evident in the data. When present, it can be recognized by the combination of a high coefficient of multiple correlation (R^2) and statistically insignificant coefficients of the variables.

A review of the Durbin-Watson Statistic revealed serial correlation of error terms which was corrected by application of the Durbin-Watson Statistic Method. The Goldfeld-Quant Test for heteroscedasity indicated that the variance of the error terms was a constant.

The model for the full range data set was developed as follows:

Equation 1:

$$R = \underset{(0.44)}{0.57} + \underset{(21.5)}{0.82\,T} + \underset{(4.15)}{1.88\,TMT}$$

$$- \underset{(-2.14)}{1.11\,AC} - \underset{(-3.40)}{1.70\,PM} + \underset{(0.59)}{0.0002\,FR}$$

Values in parenthesis are t ratios.

$N = 227 \quad R^2\,(adj.) = .71$

Durbin-Watson statistic: (original/corrected) = (1.25, 2.11)

Durbin-Watson (upper limit/lower limit) = (1.51/1.72)

R = Raffinate turbidity units
T = PLS feed turbidity units
TMT = Electrostatic treatment effect (dummy)
AC = Aqueous continuous upset effect (dummy)
PM = Polymer trial overfeed effect (dummy)
FR = PLS flow rate
N = Number of observations

Testing for absence of treatment effect sets the hypothesis:

$$H_0: \; b_{TMT} = 0;$$
$$H_1: \; b_{TMT} > 0.$$

With the t value of b_{TMT} equal to 4.15 the treatment effect is clearly significant at the 5 percent level and the null hypothesis is rejected. The alternate hypothesis is accepted that there is a significant treatment effect.

The model correctly assigns negative values to the two periods of upset. The magnitude of the coefficients indicate that either period carried sufficient impact to negate the treatment effect accomplished with the initial treatment installation.

The insignificance of PLS flow rate in explaining the turbidity of raffinate was surprising, but the lack of correlation was both strong and persistent in other tests. PLS flow rate was therefore excluded from Equations 2 and 3 to direct the focus toward the significant parameters.

Comparing the treatment effect between the first and last extractors produced the following models:

Equation 2:

$$R_{E1} = \underset{(5.62)}{2.66} + \underset{(32.42)}{0.89}\ T - \underset{(-2.21)}{0.78}\ AC$$

$$\underset{(-0.95)}{-0.32}\ PM$$

Durbin-Watson test (uncorrected/corrected): (1.431/1.904)

Durbin-Watson (lower limit/upper limit): (1.53/1.70)

R_{E1} = First extractor underflow turbidity.

R^2 (adj.) = 0.85 N = 190

Equation 3:

$$R_{RAFF} = \underset{(6.33)}{4.21} + \underset{(22.3)}{0.87}\ T - \underset{(-3.33)}{1.66}\ AC$$

$$\underset{(-4.62)}{-2.23}\ PM$$

Durbin-Watson test (uncorrected/corrected): (1.442/1.904)

Durbin-Watson (lower limit/upper limit): (1.53/1.70)

R_{RAFF} = Raffinate turbidity.

R^2 (adj.) = 0.73 N = 190

The test to determine that the first extractor treatment effect is significantly greater than the treatment effect exhibited at extractor 4 (raffinate), proposes:

where:

a is the constant (intercept) of the equation.

$$H_o: a_{(T\ E1)} < a_{RAFF};$$
$$H_1: a_{(T\ E1)} \text{ not } < a_{RAFF}$$

The difference between the two models represents a horizontal shift in the plot of the regression line with a subsequent difference in the value of the respective intercepts. The value of the intercept of Equation 1 representing the first extractor turbidity is 0.57 which lies well below the lower 95% confidence limit of 3.27 associated with raffinate turbidity. The hypothesis is thus accepted at the 5% level of significance that the first extractor underflow exhibits a greater treatment level. The shift to a lower value for the intercept conforms with theory that the plot of the intercept of an adequately treated system will approach zero. In addition, the relative effect of system upsets as exhibited in the coefficients of AC and PM is shown to be much less than at the more elevated treatment level in spite of the circulating load of existing sludge which is delivered to the unit with the organic.

Conclusion

The application of a high intensity electrostatic field can improve solids transport through mixer-settlers in solvent extraction circuits. Design of sufficient surge capacity into installation designs promises to aid in moderating upsets caused by abnormal operating conditions or abnormal loads of suspended solids, and contribute to erosion of existing sludge deposits.

Electrostatic dispersion therefore provides a means of reducing the operating costs associated with sludge accumulation in mixer-settlers. Functioning on the most elemental level, by amending the charge densities of the sludge components, this technique performs without introducing compounds that potentially interfere with kinetics or with phase separation.

Acknowledgement

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