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Installation and Testing of a Jorin Visual Process Analyzer

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SUMMARY

The Jorin **Vi**sual **P**rocess **A**nalyzer (ViPA) is an on-line instrument that uses video microscope imaging to detect and measure the physical characteristics of dispersed objects within a process stream or laboratory sample. Object analysis is performed by capturing an on-going sequence of single frames from the video feed and relaying the images in real time to a nearby control computer where the ViPA software then processes and transfigures the information from the images into meaningful process data. The ViPA captures and analyzes approximately 15 images per second and continuously records 17 material parameters including size, shape and optical density. The ViPA software uses the measured parameters to differentiate between different classes of objects including organic droplets, gas bubbles, and solid particles.

Procurement of this instrument provides a unique capability to support predictive modeling and further understanding of mass transfer during solvent extraction processes. Organic droplet data collected using the ViPA can be used to develop dispersion profiles of the liquid-liquid mixing and disengagements sections for each type of process equipment. These profiles will provide insight into mixing dynamics and will guide the prevention of emulsion formation that leads to system losses. Additionally, the measurement capabilities of the ViPA will provide the input needed to create new two-phase Computational Fluid Dynamics (CFD) models that characterize both mixing and separation operations in the various types of equipment. These models can then be used to improve process efficiency by optimizing operation parameters for each proposed extraction cycle.

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ACRONYMS

ACC	Annular Centrifugal Contactor
BCTC	Bonneville County Technology Center
CFD	Computational Fluid Dynamics
O/A	Organic to Aqueous Ratio
ViPA	Visual Process Analyzer

SEPARATIONS/ FUNDAMENTAL SCIENCE AND METHODS DEVELOPMENT

1. INTRODUCTION

A ViPA has been procured, installed, and tested. Initial trials were conducted in the Solvent Extraction Technology Development Laboratory located in bay 6 of the Bonneville County Technology Center (BCTC). Trials were performed by analyzing organic droplet data from a two-phase mixture sampled at the rotor inlet on an Annular Centrifugal Contactor (ACC). During initial trials, the ViPA proved capable of producing consistent results from repeated trial runs.

Although additional testing is still needed, this system has the potential to generate the data needed to develop comprehensive CFD models for the various types of solvent extraction equipment. Organic droplet data obtained from the ViPA can be used to correlate droplet size to mass transfer distribution coefficients. Additionally, the data can be used to correlate droplet size to rotor speed in ACCs, to pulse amplitude and frequency in pulse columns, and to mixing speed in mixer/settlers. These data can then be input into CFD models that characterize the mixing and separation operations in each type of equipment.

This report provides information on the ViPA system capabilities, details of installation, as well as results from initial trial runs.

2. ViPA CAPABILITIES

2.1 Equipment Overview

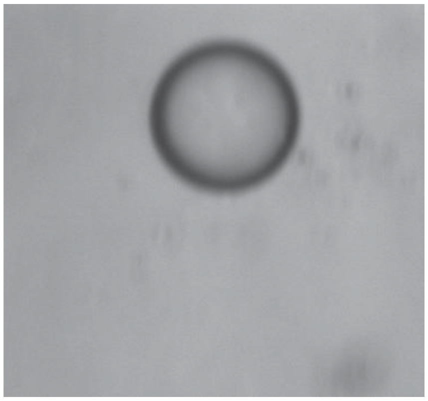
The ViPA is an on-line instrument with the capability to detect and measure the physical characteristics of dispersed objects within a process stream or laboratory sample. The ViPA unit consists of a high speed digital camera, lens, flow cell and light source. The process stream or sample is passed through the flow cell as the high speed camera captures live video. Object analysis is performed by freezing an on-going sequence of single frames from the live video feed and relaying the images in real time to the nearby control computer. The ViPA software then processes and transfigures the information from the images into meaningful process data. The ViPA captures and analyzes approximately 15 images per second and continuously records 17 material parameters including size, shape and optical density.

2.2 Object Classes

The ViPA is capable of analyzing up to eight user defined classes of objects including solids, liquid droplets, and gas bubbles. Object classes are defined based on their physical properties. Two useful properties used to segregate object classes are shape factor and optical density.

Shape factor is defined as follows:

Shape factor can be used to separate liquid droplets and gas bubbles from solids. Based on the above equation, a sphere has a shape factor of 1. Typically, droplets and bubbles are nearly spherical and have shape factors ranging from 0.8 to 1. Solids are usually irregular in shape with shape factors < 0.8. Figures 1A and 1B illustrate the use of shape factor. Figure 1A is an image of an organic droplet. The droplet has a shape factor of 0.95. Figure 1B is an image of a solid object. The object has a shape factor of 0.27.



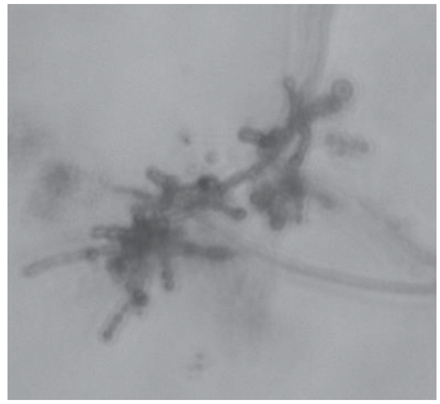


Figure 1A. Image of an Organic Droplet Figure 1B. Image of a Solid Object

Optical density can be used to separate liquid droplets from gas bubbles. Relative to bubbles, droplets typically have a lower refractive index difference from the continuous phase; therefore, they have a lower optical density. Additionally, the liquid inside a droplet will tend to magnify light. The result is that liquid droplets usually appear brighter than air/gas bubbles. Because optical density depends both on the object type and the continuous phase, it must be determined empirically for each set of experimental conditions.

2.3 Threshold Value and Edge Strength

In addition to defining object classes, the ViPA software allows the user to define which objects are in focus and should be included in the analysis based on the parameters of threshold value and edge strength. As with optical density, determination of threshold value and edge strength depends on the object type and the continuous phase and must be determined for each set of experimental conditions.

The threshold value defines the lowest acceptable difference between the background and the boundary of an object. Figure 2 shows an example of the use of threshold value. In this image, object 1 meets the threshold value, while object 2 does not. Only object 1 will be analyzed by the ViPA.

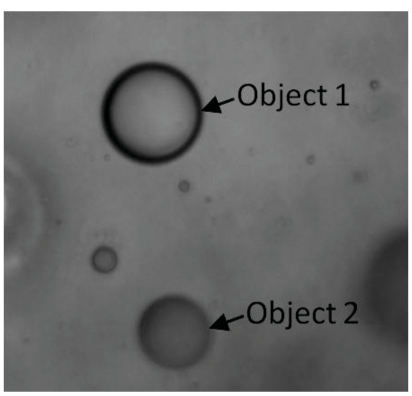


Figure 2. Determination of Threshold Value

Edge strength is a measurement of the rate at which the dark grey of the object turns to the light grey at the edge of the object. The edge strength parameter is used to filter out objects that meet the threshold value but are out of focus. Figure 3 illustrates the use of edge strength. The object in this image meets the threshold value, but is excluded from measurement based on edge strength.

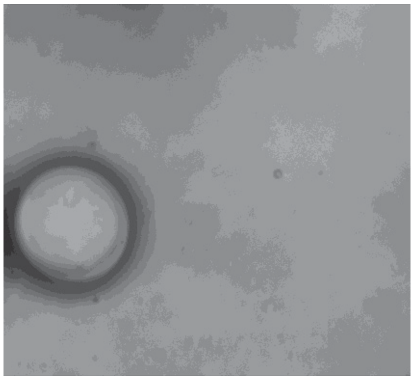


Figure 3. Determination of Edge Strength

2.4 Data Collection and Reporting

The ViPA has two data collection modes, batch data collection and periodic data collection. The periodic data collection feature is designed for use when the ViPA is permanently installed on a process line and used for continuous monitoring. While this is a useful feature for manufacturing facilities, this feature is not very useful in a research setting; and therefore, will not be discussed in detail. The batch data collection feature allows collection of discrete batches of data. Data can be collected in batch sizes ranging from 1 to 50 thousand objects in increments of 1000. Alternatively, data collection can be stopped at any time during a batch run once it has been determined that sufficient data has been gathered.

Data collected from batch runs are reported in two data files. The first data file includes the measured values for all 17 material parameters for each object. The second file contains a statistical summary for each parameter. The summary includes information such as the mean, minimum, maximum, median, mode, and standard deviation. Information is summarized for each object class as well as for all objects.

Data can also be reported graphically. The ViPA software includes graphing functions that allow the user to select the parameter to plot (usually size) as well as the graph type and weight. Data collected in batch mode can be presented in three types of graphs; integral curve, histogram, and temporal sequence. Graphs are either weighted based on count (count/total) or volume (volume/total). Figure 4 shows an example of a graph generated using the ViPA software. This graph is a histogram that represents size distribution of organic droplets. The graph is weighted by count.

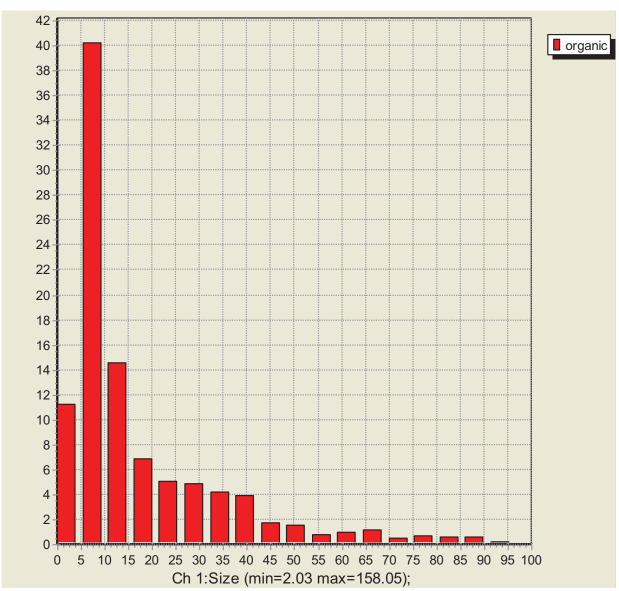


Figure 4. ViPA Graphing Capabilities

3. SYSTEM TESTING

3.1 Installation

To test the capabilities of the system, the ViPA was set up to analyze a two phase mixture at the rotor inlet on an ACC. Figure 5 shows a schematic of the ACC. A slip stream sample was taken from the contactor drain before the mixture entered the rotor for disengagement. A valve located on the drain was used to provide flow to the ViPA, while a peristaltic pump located downstream of the ViPA controlled the flow rate through the ViPA unit. A process flow diagram of the set-up is presented in Figure 6, and a photograph is shown in Figure 7. Initial trials were performed using an ACC with a 5 cm rotor. Lamp oil was used for the organic supply and tap water was the aqueous (continuous) phase.

While the current setup is sufficient for initial testing, improvements are still needed. A challenge with using the ViPA to measure droplet size in an ACC is that the ViPA cannot directly measure the droplets in the ACC mixing zone. Instead the mixture must be transported from the ACC to the ViPA. This leads to the possibility of coalescence of droplets in the transfer line. A method for transporting the solution with minimal disruption should be further investigated.

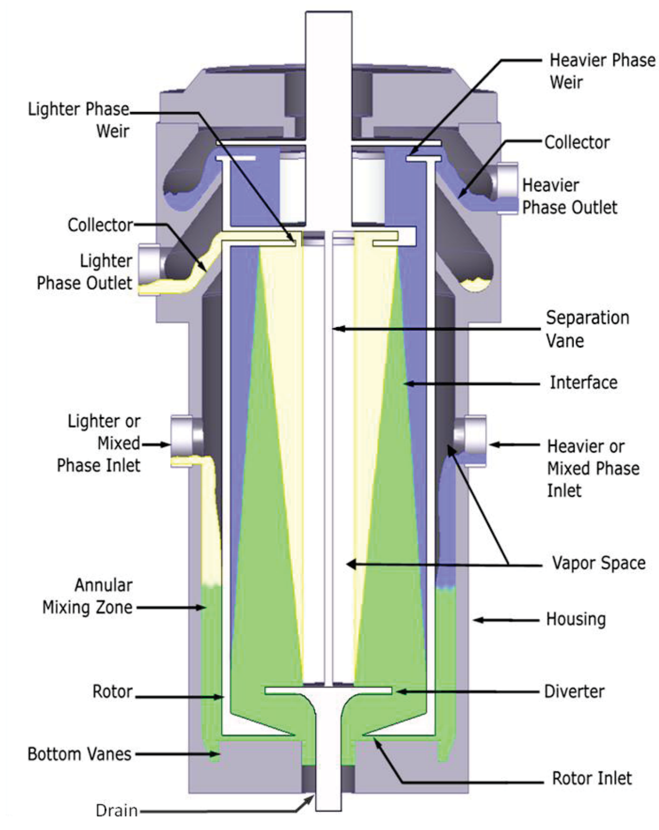


Figure 5. Annular Centrifugal Contactor

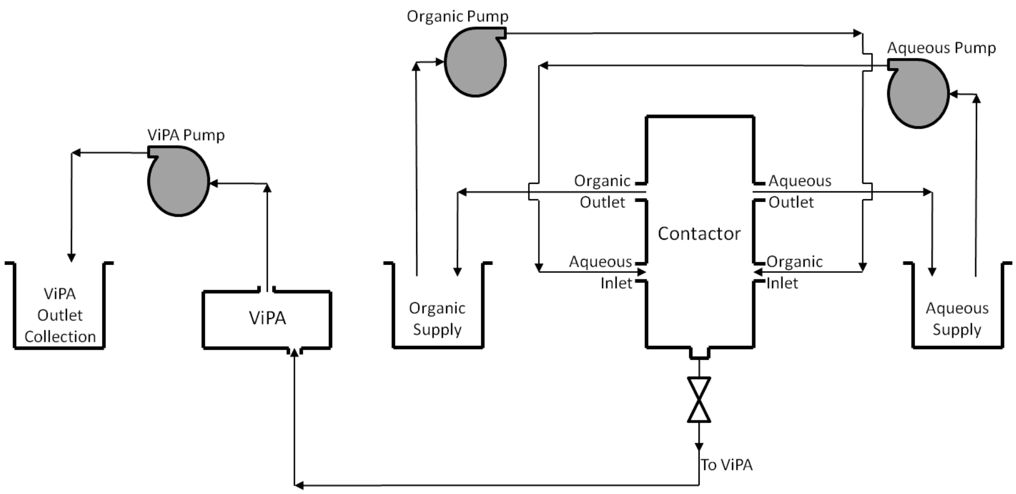


Figure 6. Process Flow Diagram of ViPA Set Up

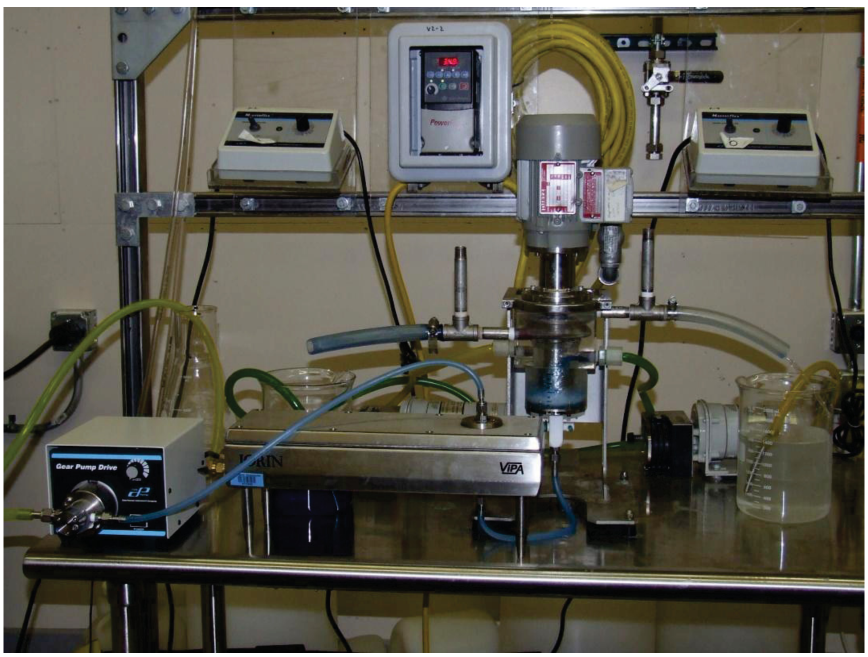


Figure 7. Photograph of ViPA Setup

3.2 Parameters for Defining Organic Droplets

By defining the parameters of the organic droplet class, the ViPA software was able to distinguish organic droplets from other types of objects such as air bubbles and solids. The parameters used to define organic droplets are provided in Table 1. Based on an evaluation of the captured images, objects smaller than 2.0 µm did not appear to be organic droplets, so the minimum size was set to 2.0 µm. As discussed previously, organic droplets are nearly spherical with shape factors ranging from 0.8 to 1. The optical density range was evaluated by capturing several images of organic droplets and recording the optical density measurements reported by the ViPA software. It was determined that most droplets fell into the optical density range of 0-0.5.

Table 1. Organic Object Class Parameters

Parameter	Range
Size	2.0-1000 μm
Shape Factor	0.8-1
Optical Density	0-0.5

3.3 Operating Conditions

Table 2 shows the operating conditions for the trial runs. Aqueous flow rate, organic flow rate, and rotor speed were set to optimize the images captured by the ViPA. The flow rate through the ViPA was set to 45 mL/min based on the manufacture's recommendation of 30-80 mL/min. Threshold value and edge strength were determined by capturing still images of organic droplets and adjusting the values, as necessary. The batch size was set to collect sufficient data to represent the process within a reasonable run time. All data were collected during steady state operations. In an ACC, operational equilibrium is typically reached in < 1 min. To ensure steady state, the ACC was operated for a minimum of 5 minutes prior to the start of each run.

Table 2. Trial Run Operating Conditions

Parameter	Setting
Aqueous Flow Rate	1.1 L/min
Organic Flow Rate	0.36 L/min
Rotor Speed	2080 rpm
Flow Rate Through the ViPA	45 ml/min
Threshold Value	20
Edge Strength	2
Batch Size	5000 objects

3.4 Results

The ViPA produced consistent results from three trial runs. A statistical summary of the data for organic droplet size is presented in Table 3. The size of organic droplets ranged from 2.0 μm to approximately 170 μm, with an average size of approximately 21 μm. Figure 7 represents the size distribution of the organic droplets. This figure shows that most organic droplets fell into the 0-10 μm size range (approximately 50%), then were fairly evenly distributed throughout the 10-20, 20-30, 30-40, and 40-50 μm size ranges. Although the accuracy of the data has not been verified, the data were very consistent between the three trial runs, demonstrating the reliability of the ViPA.

Table 3. Trial Run Data Summary

Parameter[1]	Trial 1	Trial 2	Trial 3
Average Size	23.4	23.2	16.4
Standard Deviation	22.8	26.8	19.9
Maximum	157.5	168.4	158.1
Minimum	2.0	2.0	2.0

[1]All values are in units of μm.

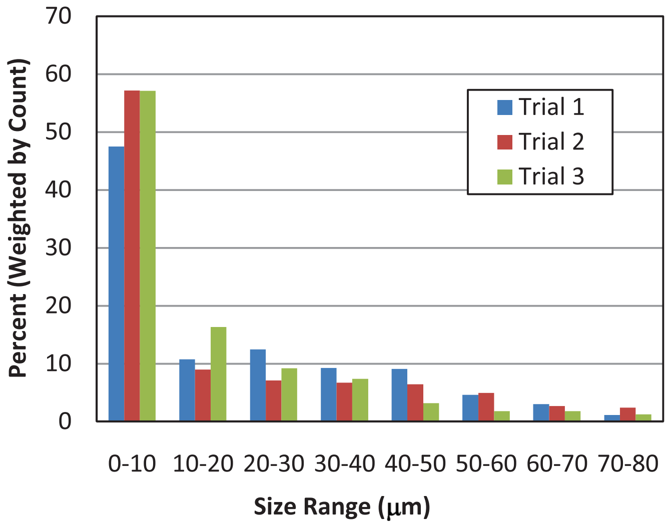


Figure 7. Size Distribution of Organic Droplets

4. CONCLUSION

A ViPA has been procured, installed, and used to characterize a two-phase mixture from an ACC. Consistent results obtained from three trial runs demonstrate the reliability of the ViPA. The ability to distinguish organic droplets from other object classes based on physical properties was evaluated, and the use of threshold value and edge strength to define object analysis was explored.

While the results of initial trials are promising, additional method development is needed. The ViPA does not allow direct measurement in the mixing zone of an ACC. Instead solution must be transferred from the ACC to the ViPA. This may result in droplet coalescence in the transfer line. The same issue will be present in pulse columns and mixer/settlers. In order to obtain representative data, methods for transporting solution to the ViPA with minimal disruption need to be developed.

Once methods are developed, additional testing will be performed. Proposed future tests include examining different operating conditions in the ACC, such as varying rotor speeds, evaluating different types of extraction solutions, and testing a range of organic to aqueous (O/A) ratios. Once ACC testing is complete, similar tests will be performed using mixer/settlers and pulse columns.

Although additional development is needed, the ViPA shows promise in providing a unique capability in the characterization of liquid-liquid extraction processes. The ViPA may be capable of providing the organic droplet data needed to support CFD models for the various types of solvent extraction equipment. These models could then be used to improve process efficiency by optimizing operation parameters for each proposed extraction cycle. If additional method development and testing is successful, the data generated from this instrument will prove invaluable in the understanding of mass transfer during solvent extraction processes.