An investigation into sub-optimal control on the downstream processing of a large scale industrial process

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Abstract—**Industrial processes can be divided into two main areas： upstream processing; involving the manufacture of product and downstream processing; product separation and purification. Although both of these operations are necessary to obtain the final product; optimisation and improvement efforts are generally biased towards upstream processing. The importance of downstream processing can often be neglected to concentrate on the "more important"aspect of upstream processing. However, if the purification and separation steps aren't controlled effectively it can result in a significant reduction in the overall process yield. This investigation focuses on the control of a two stage counter current liquid-liquid extraction unit downstream of a batch process. A reduced separation capacity of this process is observed due to a flow oscillation in the solvent stream. The cause of this oscillation is investigated by analysing process variable behaviour, paying particular attention to the interplay between the control strategy and disturbances of the process. The effect on separation efficiency is shown through high frequency analysis of product concentrate in the output stream.**

The aim of this work is to highlight the importance of the process control strategy. Reviewing the linkages between the process controls, product yield and quality are essential as part of a continuous improvement strategy. Such reviews can highlight opportunities for significant increases in yield that are often masked out through infrequent sampling procedures.

Fig. 1.   Schematic of liquid-liquid extraction unit operation

I. INTRODUCTION

Downstream processing is an integral and essential aspect of many industrial processes. Its application can be varied from industry to industry but the goal remains the same: to separate and purify a product. The downstream processing operation of concern here is liquid-liquid extraction. This is used across a large range of industries; including the biotechnology sector to separate out enzymes [1] and antibotics [2], removal of toxic metals from waste water systems [3], and in the reprocessing of nuclear fuel waste [4]. Generally liquid-liquid extraction processes involve the extraction of a product from an aqueous phase into a solvent phase. This is achieved through the physical contact between the two liquids. There are many different types of liquid-liquid extraction equipment; but the centrifuge extractor will be the focus here. This work will look at the current control strategy implemented on a liquid-liquid extraction process on a large scale manufacturing plant, depicted in Fig. 1. The unit operation is a batchwise continuous process with each batch lasting approximately 15 hours. The aqueous stream flows in the direction of Pod-A to Pod-B
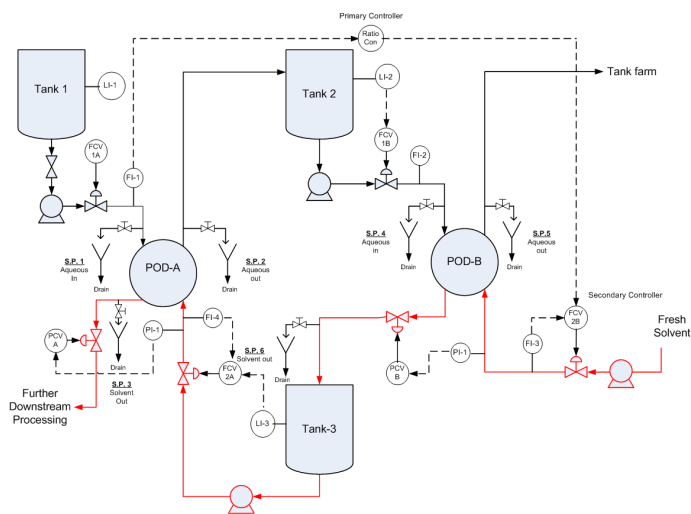
and the solvent phase flows counter currently in the direction of Pod-B to Pod-A. The product from the aqueous phase is extracted through physical contact with the solvent phase. Separation of the product is promoted by a chemical adjustment in the aqueous phase. Pod-A is the primary extractor; removing approximately 85% of the product and Pod-B is the secondary extractor; removing approximately 13%. Improving the separation capacity of Pod-A is the primary goal of this work.

Before the process trends are considered, it is important to understand the fundamentals of liquid-liquid extraction. One of the most important variables to control during operation is the back pressure on the solvent phase. This back pressure is controlled by manipulating the two pressure control valves PCV-A for Pod-A and PCV-B for Pod-B. Fig. 2 illustrates the steady state operation of one of the centrifuges/pods during normal operation. The heavy liquid in (HLI: aqueous phase) enters near the shaft, the light liquid in (LLI: solvent) enters near the rim. Due to the centrifugal forces and the differences in specific gravity of the two phases, the heavy liquid is forced towards the rim and the light liquid is forced towards the centre. This results in the formation of three phases:
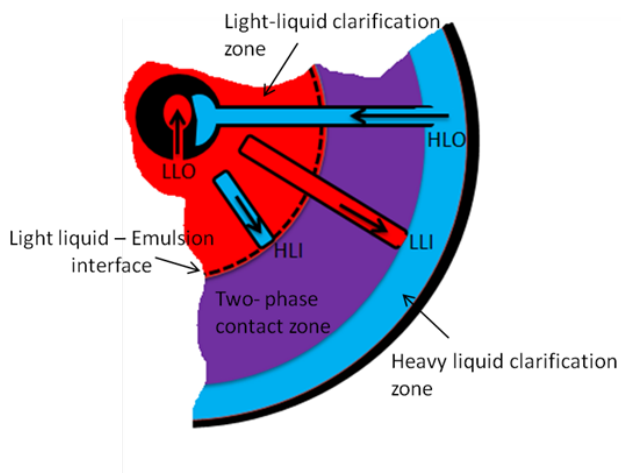
Fig. 2.   Schematic of Pod internals

1) Heavy liquid phase towards the rim.
2) An emulsion of both heavy and light liquids mixed together (contact zone).
3) Light liquid phase towards the shaft.

The position of the interface between the emulsion phase and the light liquid out (LLO) is primarily controlled by imposing a back pressure on the LLO. The position of the interface is also affected by the ratios of the two phases entering the pod. Controlling this interface is key and it is important to ensure adequate separation of the two liquids. If the back pressure is too low, heavy liquid can become entrained in the light liquid out; known as shaft flooding. And if the back pressure is too high, light liquid can't leave the shaft and can become entrained in the heavy liquid out; known as rim flooding. As shown on Fig. 1, the back-pressure of the solvent exiting the pods is controlled using a pressure control valve which is manipulated using the output from the pressure indicator on the solvent flow in. The outlet pressure is adjusted to ensure the ratio of the pressure in to pressure out is kept at 65%.

II. DISCUSSION

The overall ratio of the streams entering the unit is controlled through feed forward ratio control. The aqueous flow entering Pod-A produces the set point for the primary controller; the output of this is used by the secondary controller to manipulate the valve position of FCV-2B to ensure the aqueous to solvent ratio of 6.75:1 is kept as constant as possible. Fig. 3 shows a typical representation of the trends associated with this cascade control loop. The flow of aqueous entering Pod-A is shown to be relatively constant at 12,000 L/hr, changing by a maximum of 2.3%. The solvent flow entering Pod B is controlled by the secondary flow controller FCV-2B. The valve position of this secondary loop oscillates from 75% to 67% every 2-3 minutes. These small changes in the valve position result in the solvent stream fluctuating repeatedly from 1600 L/hr to 2000 L/hr; a 22.5% change . Although the output of controller FCV-2B only tries to make small adjustments to
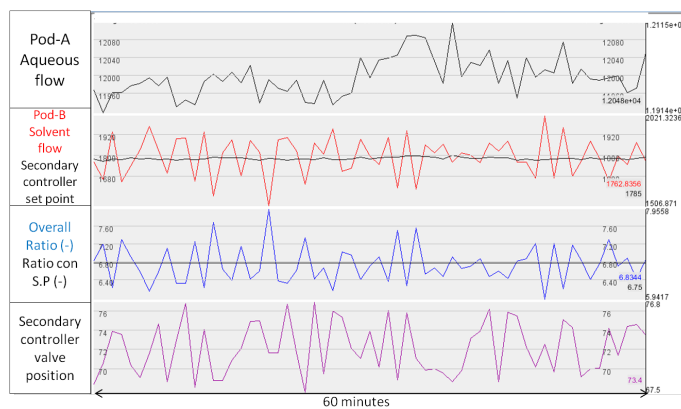

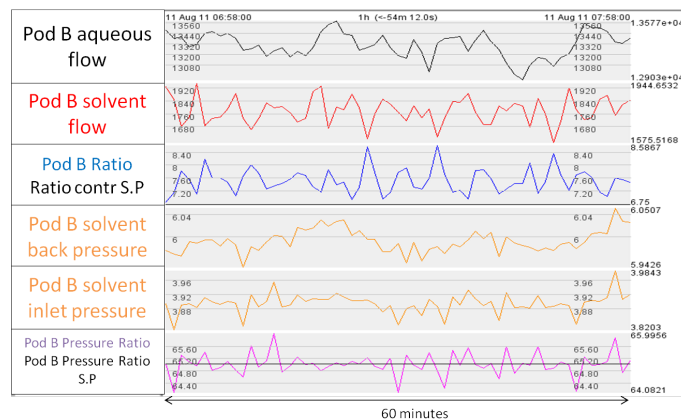Fig. 3.   Trends for overall flow ratio control

Fig. 4.   Trends of flow ratio and pressures across Pod B

the flow, the indicator FI-3 shows that the resulting changes in the flow are significant. The difference between the flow set-point and the actual flow may be the result of numerous reasons including a high back pressure on solvent entering the process, poor tuning of the control loop or a result of a faulty or oversized valve.

Fig. 4, looks at the actual ratio of the two streams entering Pod B. As the process is a continuous one, the above mentioned trends would be expected to be similar as long as the levels of the tanks 2 and 3 are kept constant. However after comparing figures 3 and 4, the flow ratio of the streams entering Pod-B is shown to be consistently higher than its set-point of 6.75:1. The pressures of the solvent entering and exiting the pods are shown to fluctuate around their set point of 65%. Some of the larger fluctuations are shown to correlate with the peaks shown by the flow ratio of Pod-B, however since both signals are quite noisy it is difficult to show a direct relationship.

Fig. 5, shows the process trends for Pod-A; a large fluctuation is seen by indicator FI-4 on the solvent stream. The ratio of the aqueous to solvent phase entering the pod is shown to periodically oscillate around its set point of 6.75, ranging from 5.45 to 7.54. This oscillation is shown to repeat throughout each batch and has a complete cycle of approximately 22
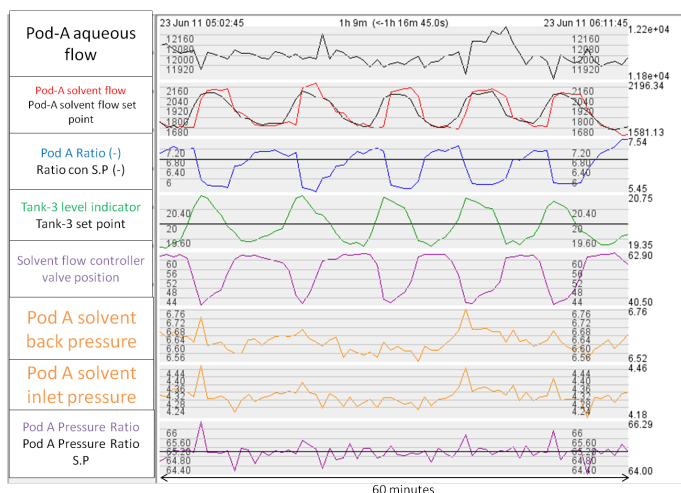
Fig. 5.   Trends of flow ratio, pressures and levels associated for Pod A

Fig. 6.   High frequency sampling campaign modelling the pod efficiency during normal operation

minutes. In order to examine the root cause of this fluctuation the level controller of Tank-3 (LI-3) is examined.

The flow of solvent entering Pod-A is manipulated based on the level in Tank-3. The level in this tank is shown to oscillate around its set point of 20% , peaking at a high of 21.07% and a low of 19.23%. Although this deviation from set point is very minor, it causes the output of the valve FCV-2A to change significantly. The valve position of FCV-2A is shown to change by 20% in direct response to the small changes in the level of Tank-3. This consistent opening and closing of the valve results in the solvent stream entering Pod-A to oscillate from 1600 to 2300 L/hr with the same frequency as the level controller.The actual solvent flow to Pod-A matches the cycling set point closely showing that the root cause of the cycle to be the level controller output of Tank-3. Although the pressure ratio of the two pods are shown to be reasonably well controlled to their set points, some spikes on the flow ratio are shown to align up with the valve positions of FCV-2A. This implies that the solvent flow ratio is also having an effect on the position of the light liquid-emulsion interface which is of primary importance to the separation efficiency of the pods.

Although the above mentioned flow oscillation has been an inherent problem on this plant for many years, its effect on the separation capacity of the pods had never been quantified. The current sampling procedure is too infrequent to capture the effect of this flow oscillation on the separating efficiency of the process. The current sampling procedure takes three process samples from the sampling points 1, 3 and 5 outlined in Fig. 1 every two hours. These sample points allow one to calculate the overall efficiency of the process by performing a simple mass balance but it is too infrequent to capture the effect of the solvent flow fluctuation.

In order to quantify the effect of the solvent flow oscillation on the process, a high frequency sampling campaign was undertaken. This campaign concentrated on Pod-A, as this pod accounts for approximately 85% of the product separation. It was carried out through two separate sampling campaigns. The
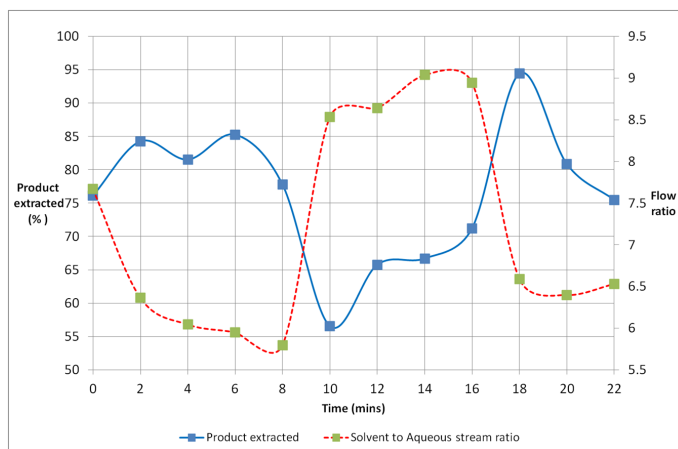
first campaign involved sampling the process over a 24 minute period during normal operating conditions, with the aim to model the effect of the solvent flow oscillation. The second involved fixing the ratio of the two streams entering the pod with the aim to model the pod's behaviour when the ratio of the two streams entering the pod is held constant.

The high frequency sampling campaign involved taking process sample every two minutes from the sample points 1, 2, 3 and 4 shown in Fig. 1. Once the process samples were collected, the product concentration was measured in these samples using off line analysis techniques. Fig. 6 shows the effect of the oscillating flow ratio on the separating efficiency of Pod-A, based on the product concentrations measured during the high frequency sampling campaign. The percentage of product extracted is calculated using the mass flow rate of product extracted into the solvent phase in Pod-A divided by the total mass flow rate of product entering the process.

The second high frequency sampling campaign is shown by Fig. 7, it shows the separation capacity of Pod-A when the flow ratios entering the unit are kept constant. This shows that by controlling the ratio of the two fluids to set point, the separation capacity of the pod also remains constant.

Comparing the results shown in Fig. 6 with the process trends shown in Fig. 5, an approximate average concentration for the entire batch can be calculated to be just above 75%. This can be compared against the average separation efficiency of 85% approximated by Fig. 7 when the ratio of the flows entering the pods is held constant. This highlights the significant effect of the flow fluctuation on the separation capacity of Pod-A, and is summarised by Fig. 8.

Fig. 8 highlights the opportunity to increase the overall efficiency of Pod-A by approximately 10%. This is a significant improvement to the overall efficiency of the process as the product quality extracted by Pod-A is of higher quality than that extracted from Pod-B.
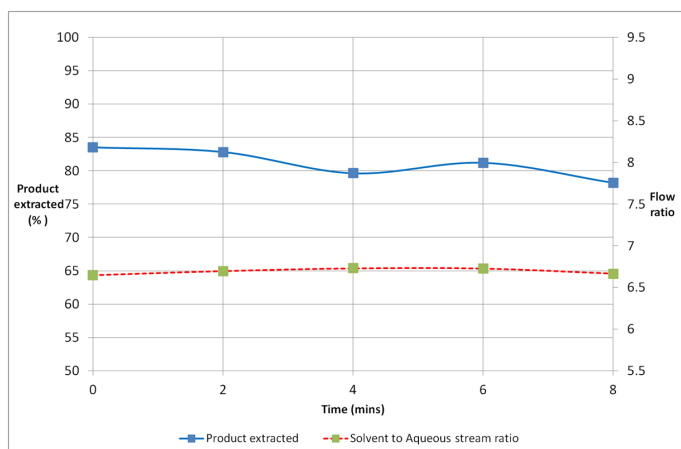
Fig. 7. High frequency sampling campaign modelling the pod efficiency during normal operation
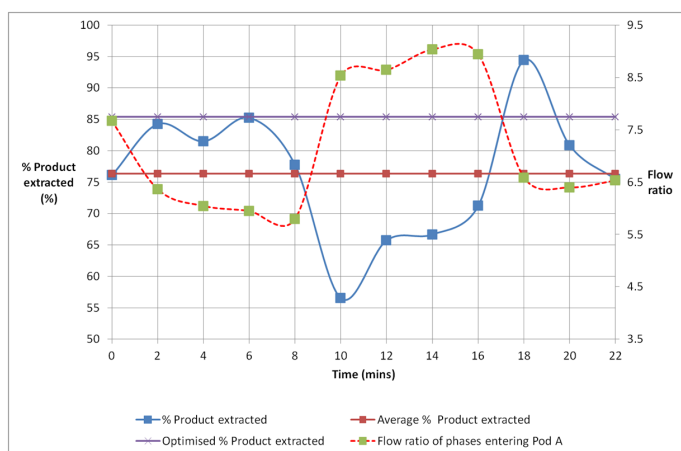

Fig. 8. Approximated average and optimised % product extracted

III. FUTURE WORK

Investigate some of the possible ways to improve the flow ratio across the two pods. One possible solution is seen by analyzing the ranks of the individual control loops. The overall ratio of flows entering the unit operation and the levels of the tanks, are shown to take precedence over the actual ratio of flows entering the pods. This ranking of the control loops does not reflect the key process parameters of the operation. This could be overcome by installing individual cascade control loops across the two pods; FCV-2A cascaded against FI-1 for Pod A and FCV-2B cascaded against FI-2 for Pod B. Although the levels in the tanks 2 and 3 will fluctuate this control strategy will ensure that the stability of the flow ratios across the pods will take priority. Once the solvent flow fluctuation have been fixed, a design of experiment will be conducted on the process with the aim of finding the optimum flow ratio set point and back pressure for the individual pods.

IV. CONCLUSION

This work highlights the importance of regularly reviewing the process control strategy. These reviews must consider the interplay between the different control loops; failure to do so may result in an undesired interaction and may lead to unwanted disturbances. The disturbance highlighted here between the level controller and the flow controller was shown to have a significant effect on the separating capacity of this liquid-liquid extraction process. The work also highlights the importance of considering the key performance parameters of the process when designing the control strategy. These should take preference in the control strategy allowing the variability to be absorbed by the less important parameters such as tank levels.

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