CONTAMINATION MANAGEMENT IN SOLVENT EXTRACTION PLANTS

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ABSTRACT
Most solvent extraction plants are used to remove contaminants and to concentrate the ion species of interest for further processing. This activity can be significantly impaired by the cross contamination of the process streams with other species that can be transferred across the phases. These species can have significant impacts on the operation of the SX plant itself, operation and chemical contamination of the concentrated stream and further impacts on the process applied to the raffinate stream.

The minimisation of this cross contamination is a major driver for: PLS treatment, SX circuit design, reagent selection, internal SX plant unit operations for contamination minimisation, reagent regeneration systems, and subsequent treatment of the concentrated stream and raffinate. A wide variety of these systems is available to the plant designer and operator. The selection and use of them is not straight forward or obvious until (generally) after an upset event has occurred.

The paper describes many of the methods of contamination control as well as criteria for their selection. Management of one level of contamination often introduces a further upset condition that also has to be addressed for a successful implementation to be achieved. A hierarchy of such issues has been developed to allow practitioners to understand these interactions and to successfully implement programmes of contamination management.
INTRODUCTION

The solvent extraction (SX) process is used to purify and concentrate an ion of interest so that it can be subsequently recovered. SX operation and the purity of the product can be severely affected by contaminants transferred from the PLS, and contaminants carried over from the SX operation itself. Contamination of the raffinate and concentrate stream from the SX plant can also generate significant problems in the downstream process activities.

One of the issues with identifying that a contamination problem exists is that the symptoms often occur in a process that is remote from the contamination. This has been identified \(^{1}\) “...the perceived problems (in SX) were soon discovered to be symptoms of a problem occurring elsewhere in the circuit...”. It has also been shown \(^{2}\) that the effect of contamination was “…complex with many symptom and parameter interactions...”. It is generally a difficult process to untangle the obvious symptoms in an operation; and to follow the thread of contamination upstream to the primary source of the problem.

The other significant issue is that many plant designers are not aware of, or do not take sufficient cognisance of the effects that contamination can have on the SX and other process sections. There are a number of references covering a range of internal SX contamination caused by the PLS constituents \(^{2-10}\). The recognition that contamination can and will occur is a prime driver for the selection of the overall flow sheet and appropriate processes, unit operations and controls that are included; to minimise or prevent the subsequent effects. This paper includes only some of the available references to illustrate the range of contamination and the potential effects that ensue.

Table 1 summarises the overall structure of the contamination issues with:

- The contaminant and its source
- The direct effect on the process
- Effects on subsequent process stages
- Direct treatment / prevention
- Treatment of indirect or subsequent symptoms that develop

SOURCES OF CONTAMINATION

The major sources of contamination are: the PLS constituents, the transfer of PLS constituent(s) to the SX concentrate stream and the transfer of the SX reagent to the raffinate and the concentrate streams.

PLS contamination

The PLS constituents are impacted by the upstream process and operations. The actual SX operation itself determines if a PLS characteristic is a contaminant or not. Amphoteric ions such as Mo, Bi, Zr can have dramatic effects on U SX\(^{1, 5, 6, 9}\) but have none on Cu SX. Whereas silica can affect all SX plants when it is in a polymerisable form \(^{1, 6, 8, 9}\). Many contaminants cause issues in SX plants independent of the SX process used. The most significant of these are:

- Suspended solids including biomass
- Scale forming ions especially calcium
- Surfactants
  - Emulsifiers
  - Flocculant
  - Biomass \(^{11}\)
  - Oils from gear box failures and mining equipment maintenance
- High TDS and /or solution viscosity can be the cause of poor SX performance that leads to downstream contamination issues.
- Humates that can transfer deleterious ions \(^{6}\) (acting much like carboxylic acids e.g. V10 to transfer via the organic phase)
- Highly oxidizing conditions from nitrates or high Eh in the leach
- Organic and extractant from upstream SX operations
- PLS ions when present in sufficient concentration
  - Amphoteric ions
- Chloride in tertiary amine USX circuits that co-extract with the uranium

The PLS ions themselves can be contaminants not only to the SX plant, but to downstream processes in the concentrated stream. The wide range of ‘contaminants’ is so broad that their potential effects are not always properly considered. The identification of the main ones is obvious (solids, surfactants, known poisons for each extractant); but the more subtle ones are often overlooked until such time as adverse operation is evident; when remedial measures are more urgent and costly. The prime example of this is the presence of cruds(1) in the early uranium SX plants that had not been evident in the laboratory or pilot plants.

**Contamination from the SX operation**

**SX Internal Contamination**

Internal contamination from the SX operation is related to additional issues that arise within the plant, that contribute to the contamination seen within the exit streams or as upset conditions in the SX – poor phase disengagement, poor transfer capacity or poor transfer kinetics being typical outcomes. The main causes of internal contamination are associated with chemical changes in either the aqueous or organic phases; or internal generated addition of other components.

Chemical changes in the aqueous stream
- Scale formation from increases in calcium content (especially in V10 nickel SX extraction)
- Chemical changes during SX that can create precipitates (13,14) that in turn can
  - Cause blockage of the plant and poor utilisation
  - Form crud with the precipitate
  - Create stable emulsions
  - Create a stable ‘third phase’ that interferes with the phase separation activity
- Polymerisation of silica (and other amphoteric ions) creating voluminous floating crud

Chemical changes in the organic stream
- Degradation of alcohol modifier to carboxylic acids
- Oxidation of organic to surface active species, an inactive components (nitration reactions).
- High loading leading to third phase formation
- Poisoning by extracted ions that can lead to major reductions in transfer capacity and kinetics
  - Cu on Cyanex 301
  - Fe on D2EHPA
  - Cl on tertiary amines
  - Rare Earths on D2EHPA for Zn extraction
  - Mo & Cr for tertiary amines
  - Cr for Cyanex 272

Addition of other components is in interesting area that can have some unusual input vectors.
- Air entrainment during mixing leading to:
  - Stable emulsions
  - Low density emulsions that float rather than sit at the interface
  - Air disengagement that can upset the settler flow patterns and increase entrainments
  - Increased entrainment of aqueous attached to the air bubbles
- Oil contamination from gear box seal failures
- Glycerine contamination from failure of peristaltic pumps
- Massive insect infestation with addition of proteins, humic acids and other organic species that can interfere with all aspects of organic health. This particular aspect has been a feature of arid areas in Australia after significant rain events; with Bogong moths at GCC and grasshopper at Whim Creek as examples.

**SX Output Contamination**

The contamination from the output of SX plant (raffinate, wash/scrub effluent and concentrate streams) is related to the transfer of a deleterious species from the PLS or the organic itself to the exit stream(s). This contamination then affects the SX operation directly or has downstream effects
The more common (and simpler) contaminations involve:

- The entrainment of PLS species into the concentrate stream
- The chemical transfer of non-target ions to the concentrate stream
- The entrainment of the SX organic into the raffinate and concentrate stream(s)
- Transfer of any one phase into another part of the SX circuit via crud formation and movement (a “crud run”) \(^{(12)}\)
- Other physical methods of passing one phase to another part of the plant where it is a liability (such as leaking baffles and partitions, valves that do not close properly etc). These can be very difficult to diagnose and then find.

More complex transfer mechanisms can involve closed loops \(^{(8)}\) and chemical positive feedback. The most well known is the manganese degradation of copper SX organic, in the absence of Eh control in the electrowinning (EW) plant.

- Manganese in the PLS is transferred by entrainment (or more often silica based floating crud) into the copper electrolyte.
- It is oxidized to Mn\(^{7+}\) in the EW operation
- The Mn\(^{7+}\) returns in the spent electrolyte sent to the SX and oxidizes the organic
- The oxidized organic has slower phase separation times leading to more entrainment of the PLS to the electrolyte
- The higher concentration of oxidized manganese in the electrolyte gives a faster degradation of the SX organic finally leading to a catastrophic collapse of the SX operation.

Another involving a closed loop is that experienced at the Olympic Dam Operation which has a sequential Cu SX and U SX circuit. The problem experienced was chloride transfer into the EW with the stainless steel cathodes being corroded with the copper sticking and not being stripped \(^{(4)}\). The eventual culprit was found to be the USX reagent that was entrained in the raffinate.

- The tertiary ammine can transfer chloride
- It was being lost to the raffinate in such quantities that it was returning to the copper PLS via the leach and CCD circuit.
- In the Cu SX it was transferring the chloride to the EW electrolyte with disastrous results for the stainless steel cathodes.
- The cause of the high losses was not purely entrainment but physical transfer during start and stop transients; especially the extraction system that released much of the contactor organic contents to the raffinate tank on shut down.

**EFFECTS OF CONTAMINATION**

All of the sources of contamination have some level of effect within the SX plant, or on downstream operations. Generally the effects are obvious with formation of crud, transfer of unwanted species to the concentrate stream, poor internal operation of the SX (slow phase disengagement, reduced chemical transfer capacity and formation of stable emulsion) and adverse operation of the downstream process. Large scale examples of these effects are experienced in the copper industry, with organic contamination of the advance electrolyte. The organic causes changes in the copper growth morphology which entrains the organic in the copper (“organic burn”). This is a major economic loss to the operation that must be controlled. Similarly the transfer of iron to the electrolyte lowers the current efficiency and the productivity of the EW plant.

Most contamination transfer events have economic and plant utility effects, that need to be minimised. Organic in the SX raffinate is a direct loss while it can also have major consequences on the downstream process plant \(^{(4, 9, 13)}\). The Bulong plant had a sequential cobalt and Nickel SX with Cyanex 272 and Versatic 10 as the extractants. Only 0.5% of C272 in the nickel circuit gave massive calcium transfer in extraction and uncontrolled gypsum precipitation in E1, E2 and the scrub stages. They replaced their entire nickel SX organic inventory due to the contamination.

Silica and solids based crud make the C272 in the cobalt raffinate partially hydrophilic and the air flotation column efficiency was badly affected. This passed C272 to the V10 nickel circuit. However one of the main causes of the transfer were the shared facilities between the two plants – crud treatment and extraction clean up sumps and pumps. Cross contamination was readily
If the raffinate is recycled in the plant then any elastomers need to be resistant to the organic constituents or premature failure will result. The direct second stage SX treatment of SX raffinate can have severe consequences from organic entrapment.

The downstream operation in turn can have an effect on the SX plant when these are in closed loops with each other. A well documented case is that of Olympic Dam. The Cu SX reagent in the U SX feed (Cu SX raffinate) causes poor phase separation and significant transfer of deleterious species to the ADU product.

The poisoning of the SX reagent by extraction, interference or adsorption has many manifestations that can be seen as secondary affects:

- Reduced transfer capacity from sites occupied by ions that do not strip
  - Fe in D2EHPA
  - Cu in C301
  - Cr, Mo, etc in USX
  - Cr in C272
  - Rare Earths in D2EHPA in Zn SX
- Reduced transfer kinetics from reduced affective interfacial area – generally involving some sort of surfactant
- Reduced phase separation time due to interference with the surface tension and contact mechanisms – again surfactants or amphoteric ions
- Production of stable emulsions and third phases that do not separate in the time available from reagent degradation, amphoteric ions, silica etc
- Poor selectivity as other ions are transferred by the contaminants particularly humic and other organic acids from plant and animal sources.

Oxidation of the organic can occur in a number of mechanisms:

- Permanganate oxidation of Cu SX via EW electrolyte
- Nitrate oxidation / nitration of Cu SX reagents in Chile
- Air / oxygen oxidation of Cyanex 301

The oxidation products themselves are the vector for the reduction in SX performance as many are either polar or surfactants or both.

The most widely experienced contamination is that of suspended solids or precipitates within the SX plant. This is the seed for crud formation and all the ills that are associated with this stable three phase emulsion.
It is the effects of internal SX contamination that can generate primary, secondary and sometime tertiary symptoms that can be very difficult to unravel symptoms from underlying causes \(^1,9\). The listing in Table 1 provides some of the more usual effects but is not sufficiently detailed to try to unravel all the potential outcomes, interactions and symptoms seen. Once a significant event has occurred it is a site specific activity to understand the sequence of events, the closed loops and the interactions of the plant sections in the development of the site of symptoms that are being experienced.

**METHODS OF CONTAMINATION CONTROL**

The methods of contamination control are based on a hierarchical approach. This approach has shown its strength in the ability to differentiate between the observed symptoms and the real underlying cause of a plant upset condition. It starts with the plant process and mechanical design with the recognition that contamination will occur often. The subsequent steps in the control hierarchy are:

- Eliminate the contamination route
- Prevent the contamination from occurring, which itself may create further effects or subsequent process upsets
- Minimise or control the contamination effects by other methods and hardware. This is the most commonly used method and has generated the widest range of methods and approaches as shown in Table 1.
- Treat the resultant symptoms and outcomes to minimise their economic and process impacts. This approach has also generated a myriad of methods and techniques to address the symptoms.

Table 1 contains a short list of the more usual methods of control, minimisation and treatment that have been used in the SX industry. Many others exist that are less well known or have a narrower range of application. Many of the methods are graded on how much of the contamination that they actually remove. For instance dual media filters can remove down to < 5 ppm of entrained organic in the stream. This is acceptable for Cu EW but is not enough for Ni EW. In this case < 1 ppm total (entrained and dissolved) is required and a second stage of organic removal using activated carbon is required.

**Elimination**

Elimination is not often an option or is costly in hardware or reagents. It is unusual to change leach conditions, but relatively easy to control flocculant addition. More usual is the treatment of PLS before SX to prevent contaminants such as: solids, polymerisable silica, surfactants and other organic contaminating materials. Some strategies in the category include:

- oil control by containment,
- elimination of treated sewage water from the process water supply (humic and adipic acids)
- alternate reagent selection that is not affected by the contaminant \(^12\). This is particularly important for systems containing nitrate in the PLS where organic nitration can be an issue
- Diluent wash of the PLS to remove organic contaminants.
- Use of an anti-scalant to either prevent precipitation or to make the precipitate less likely to form crud and to pass out of the SX in the raffinate.
- Precipitation of deleterious species esp – Fe, Cr, Mn. Many of these ions that can be precipitated at modest pH can be eliminated in the iron removal step. A two stage process, with recycle of the second stage solids to leach, as used at Bulong and Tati \(^10,13\); was found be to allow higher pH operation with acceptable losses of Ni and Co.
- A diluent scrub of the Ni PLS from a C272 cobalt SX was effective in elimination of the entrained C272 at Bulong (13). A unique saponification method of recovery of the C272 from the scrub diluent meant that it could be returned to the circuit without upsetting the diluent balance.
Prevention

The prevention strategies are seldom totally effective and minimisation is rather the goal. Physical removal methods fall into this category as does chemical modification of the PLS.

- The major PLS treatment is for removal of suspended solids to ppm levels as a means of crud control.
- The other major element to be prevented from entering the SX is silica in a polymerisable form. This can be precipitated when an iron removal stage is used; or sequestered with long chain poly-ethylene-glycols (PEG)\(^6\). This is a significant issue in uranium SX where the aggressive leach conditions often result in high silica levels in the PLS.
- Filtration, coalescing of organics, activated clay treatment (for organic and Eh control) and PLS diluent and peroxide wash\(^9\) are all methods of managing the PLS constituents.
- Adjustments to upstream PLS preparation can also be made to minimise the chemical risk: iron removal by precipitation can minimise the amount of D2EHPA that needs ferric removal by scrubbing. Similarly the removal by precipitation can minimise the presence of other amphoteric ions.

One of the most difficult things is to continue the prevention strategy throughout the life of the operation. As plant management changes so much of the history can be lost and plant operation revert back to less difficult methods. In some cases this has led to a recurrence of the problems that had been sorted out by the elimination strategy.

Minimisation and Control

This strategy is aimed at minimising the effects inside the SX plant and trying to control the level of contamination effects that result. It uses very similar strategies to the symptom treatment protocols. As a result it is often difficult to separate the control aspect from the symptom treatment aspect. Some of the more obvious minimisation methods are:

- Ensuring maximum loading of the desired species on the organic to crowd off unwanted ions. This is an especially effected method in Cu SX where iron crowding can be achieved.
- Use of synergistic reagent mixture that can all but eliminate the transfer of an unwanted ion\(^{13}\). Recent developments in Ni SX have shown that the calcium transfer in extraction can be managed to eliminate gypsum precipitation in the circuit.
- Use of higher aromatic diluent (20%) can minimise crud production while enhancing
• Selection of unmodified reagents in Cu SX can minimise crud production\(^{[12]}\). Alternatively a different modifier can be used that has less of an effect on crud production.
• In U SX the organic can be pre-protonated to minimise the co-extraction of other ions \(^{[4]}\). This in turn minimises the organic poisoning by the other ions.
• Provision of an inert gas atmosphere in some operations is required to minimise the organic oxidation by atmospheric air. The Ga SX in WA and Goro Nickel are two recent examples of this technique.

Figure 3: Gallium SX Plant with Nitrogen Atmosphere.

• Use of VSD’s on pump mixers can:
  - Minimise crud production
  - Reduce air entrainment
  - Improved phase break times
  - Minimise entrainment
• Planning tools for manganese control in Cu EW can provide good guidance on the strategy to be adopted in minimisation of this ion transfer\(^{[2]}\). By understanding the transfer mechanisms and the methods of management a coherent plan can be developed.

**Symptom Treatment**

Symptom treatment has received a lot of attention especially for control of PLS transferred to the concentrated strip solutions. Physical methods of entrainment control include:

• Settler enhancements and improved internal designs to improve phase separation \(^{[15]}\)
• Coalescing of PLS from the loaded organic \(^{[16]}\)
• Washing the loaded organic to dilute the entrainment \(^{[10]}\)
• Coalescing, filtration, diluent scrubbing, of organic from the raffinate and concentrate streams
• Change of the organic mix to enhance phase separation such as an alternate reagent or addition of the third phase modifier \(^{[6, 9, 12, 15]}\)
• Regular removal and separation of crud to lower the risk of a crud run \(^{[16]}\).
Chemical control relates to both the transfer of ions to the concentrate stream as well as removal of poisons from the organic phase. Transfer controls include scrubbing of the ion from the organic as well as reductive stripping for multivalent ions. Organic rehabilitation methods are quite SX system specific but are widely practiced:

- Activated bentonite clay treatment of copper oxime/ketoamine extractants \(^{(18)}\)
- Caustic scrubbing of U SX reagents to remove amphoteric ions and humic acid \(^{(1, 9)}\)
- Ethanol regeneration of U SX organic to remove Cu SX organic entrained in the PLS \(^{(4)}\)

Many of these activities have led to the development of specific hardware and methods to undertake the symtom treatment.
- Dual media filters with and without internal coalescing systems
• Organic recovery from ponds with rope mops and floating skimmers

Figure 6: Organic Recovery from Raffinate Pond with a Rope Mop

• Operator friendly crud removal systems that eliminate much of drudgery associated with this activity.
• Easier crud treatment with three phase centrifuges of robust construction and relatively high capacity.

Figure 7: Permanent Crud Removal Piping in a Settler

• Clay treatment of Cu SX reagents using pressure filters and more recently three phase centrifuges.
• On line bypassing (and a spare mixer-settler) in systems with high scaling and gypsum precipitation risks.
• Provision of both organic and aqueous recycles to allow operation of all stages in either continuity as required by the presence of silica, crud, precipitates etc.
• Use of pre-saponification of C272 and D2EHPA reagents to eliminate the risk of precipitation when using NaOH for pH adjustment.
Subsequent symptoms treatment follows the same path of physical and chemical controls. The best known of these is the combination of primary controls and secondary symptom treatment for controlling manganese oxidation in Cu EW transferred as a result of silica based floating crud. The

- All stages of SX are run in organic continuity to form interfacial compacting crud
- The high (and even extreme) entrainment of PLS in the loaded organic is managed by improved settler performance and stream specific coalescing of the loaded organic
- A wash stage is included to wash the entrained PLS Fe and Mn but to retain the Fe chemically transferred on the organic
- Regular removal of interfacial crud to limit the risk of a “crud run” transferring high volumes of PLS
- Regular clay treatment of the organic to remove the degradation products of the residual levels of manganese oxidation
- Maintenance of a high Fe:Mn ratio in electrolyte by either increasing Fe SX transfer (less selective reagent, more reagent, altering the wash conditions) or addition of ferrous sulphate to the electrolyte
- Potential use of Eh management in electrolyte with options such as: copper metal reduction tower, SO2 gas reduction, iron addition via ferrous sulphate or iron shavings and high concentration of cobalt in electrolyte.

This particularly complex solution set is not untypical of the lengths to which process and equipment need to be added, to change the operating conditions; and to address the contamination management and the secondary after effects of the primary programme adopted.

CONCLUSION

Contamination management is specific to the SX system; as to what contaminants are important and what techniques to use- U SX is very different to Cu SX.

Some PLS contaminants are universal – surfactants, silica, humic acids etc and their management is also reasonably straight forward. Others are not as widely encountered and their management is very site specific.

Often the management of a specific contaminant will entail the management of secondary and tertiary effects of the primary activity. These can become complex and interactive.

Many tools are available, from which to select an appropriate set, to address each specific contaminant.

ACKNOWLEDGEMENTS

I would like to acknowledge the many workers who have published material in the field. They have provided a wealth of information that can be used to understand and implement contamination control. This short paper cannot reference all of them as they should be.

REFERENCES


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Table 1: Contamination Sources, Effects and Treatments