NOLANS RARE EARTHS PROJECT: INNOVATIVE RECOVERY AND PURIFICATION OF PHOSPHORIC ACID BY VSEP NANOFILTRATION

By

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ABSTRACT

The Nolans Rare Earths Project (NRE) is one of the few advanced ore to oxide rare earths projects of scale in the western world to-date. The project is currently in detailed engineering phase and had started execution on enabling works in Q1 2023. It is designed to produce ~4 440 tpa NdPr oxide, 470 tpa SEG/HRE and 144,400 tpa of 54% w/w P_2O_5 MGA (Merchant Grade Phosphoric Acid) by-product on full operations.

The flowsheet involves an integrated processing of apatite and fluorapatite (major host minerals of rare earths elements) through flotation, pre-leaching, sulphation (cracking), water leaching, impurities removal, separation by solvent extraction and product recovery. Nolan's apatite and fluorapatite REE-base ore feed is the first of its kind in any known existing rare earths production plants and mature projects.

The leaching of these minerals with phosphoric and sulphuric acids produces excess phosphoric acid along with some thorium, uranium, iron, calcium, magnesium, and other cations, which are co-extracted during the process. The management of this process stream (to safely stabilise the acids and heavy metals) posed a challenge due to requirements for neutralisation, disposal, and containment.

Arafura had capitalised this challenge into an opportunity, using New Logic Research's (NLR) innovative VSEP (Vibratory Shear Enhanced Processing) nanofiltration technology. The VSEP nanofiltration membrane technology effectively recovers more than 70% of the phosphoric acid and rejects majority of the cations including thorium and uranium to meet the MGA specifications, using an acid-proof membrane. The technology is also used to control the recirculating load of impurities from recovered mixed phosphoric and sulphuric acids to the process plant. Unlike traditional (crossflow) spiral wound nanofiltration, VSEP can tolerate high suspended solids and calcium saturated feed, simplifying the circuit. Therefore, provides a more cost-effective solution than the traditional nanofiltration.

The recovery of phosphoric acid (as a saleable by-product) contributes to the economics of the project. On the other hand, the purification of the recycled mixed acid enables major operational flexibility, since it liberates the upstream pre-leach circuit from operating at a very tight range of major impurities from the fresh feed. This renders the purification circuit an integral and strategic part of the overall Nolans project flowsheet.

This paper aims to share some of the VSEP nanofiltration test work results and its translation to the engineering design of the Phosphoric Acid Recovery and Purification at Nolans. It also highlights the strategic importance of the circuit to the overall flowsheet, and therefore the technical and economic performance of the Nolans Rare Earths Project.

Keywords: Nolans Rare Earths Project, Rare earths elements (REE), NdPr, VSEP, nanofiltration, acid proof *membrane, NLR, thorium, uranium, merchant grade phosphoric acid, apatite, fluorapatite*

INTRODUCTION

The Nolans Rare Earths Project (NRE) is an advanced ore to oxide rare earths project of scale in the western world. The project is currently in detailed engineering phase and had started execution on enabling works in Q1 2023. It is designed to produce ~4 440 tpa NdPr oxide, 470 tpa SEG/HRE and 144,400 tpa of 54% w/w P_2O_5 MGA (Merchant Grade Phosphoric Acid) by-product on full operations[1].

The project is an integrated complex which includes a mine, concentrator (beneficiation plant), hydrometallurgical plant (extraction plant), refinery (separation plant and product handling), utilities (power plant, water treatment plant, sulphuric acid plant, gas separation plant) and major infrastructure (tailings dam, camp, off-site and on-site roads, bore water station etc). These facilities will be constructed and located in the Nolans site, 135 kilometres north of Alice Springs in Australia's Northern Territory. Alice Springs is well-serviced by modern existing infrastructure such as roads, rail, air, and telecommunication facilities, which largely enables the operation of the project in a single location[2].

The Nolans ore feed is a phosphate-based mineral, composed primarily of apatite and fluorapatite, which host the target rare earths elements. The ore also contains elevated amounts of thorium and uranium relative to the NORM (Naturally Occurring Radioactive Material). The processing of the ore generates mixed phosphoric acid and sulphuric acid (mixed acid), which is re-used in the plant and an excess phosphoric acid, with co-extracted impurities (including thorium and uranium) that must be managed by the project.

The Nolans Project incorporates the application of nanofiltration using New Logic Research's (NLR) innovative VSEP (Vibratory Shear Enhanced Processing) to remove majority of the impurities in these streams by membrane rejection. The VSEP nanofiltration recovers more than 90% of the phosphoric acid and mixed acid whilst rejecting more than 50% of thorium and uranium including other cations that are feeding into the flowsheet, using an acid proof membrane. This allows the Nolans project to:

(1) Generate a Merchant Grade Acid (54% w/w P_2O_5) that meets specification for sale, offsetting the requirement to neutralise the excess dirty phosphoric acid and additional volume for gypsum tailings containment and;

(2) Minimise the build-up of key impurities (Mg, Ca, Al, Fe, U, Th) from the regenerated mixed acid (particularly the phosphoric acid that is recycled upstream), in the system allowing for process flexibilities in the impurities composition of the incoming fresh ore (and concentrate) feed.

THE VSEP (VIBRATORY SHEAR ENHANCED PROCESSING) TECHNOLOGY

Membrane filtration in saltwater and wastewater have been widely demonstrated in desalination and mining industries. A key challenge to membrane filtration is fouling (scaling), which is caused by high TSS loading and increasing ion concentration build-up at the surface of the membrane. Fouling (scaling) not only leads to the decrease in flux but also in the loss of membrane's natural selectivity for rejection.

Conventional tangential or crossflow filtration reduced the solids loading to the membrane by pumping the feed at a high pressure and velocity, which creates a shear force at the surface the membrane. However, high feed velocity also results to a significant pressure drop from the inlet (high pressure) and the outlet (low pressure) end of the equipment, which increases the rate of fouling. Due to constraints in applicable forces, the conventional membrane filtration is only limited to low viscosity, low colloidal silica and low TSS feed material. Often, pre-conditioning or pre-treatment steps are necessary such as ultrafiltration and/or addition of anti-scalants / anti-fouling chemicals to meet the requirements of the downstream process[3].

In order to overcome these limitations (and provide operational flexibility and simplicity), NLR had developed an innovative vibrating membrane technology called VSEP (Vibratory Shear Enhanced Processing). In a VSEP system, the feed slurry is kept in a steady meandering flow between the parallel membrane leaf elements. Torsional vibration is induced to the membrane boundary layer via an oscillation created by the VSEP resonating drive system (Figure 1).

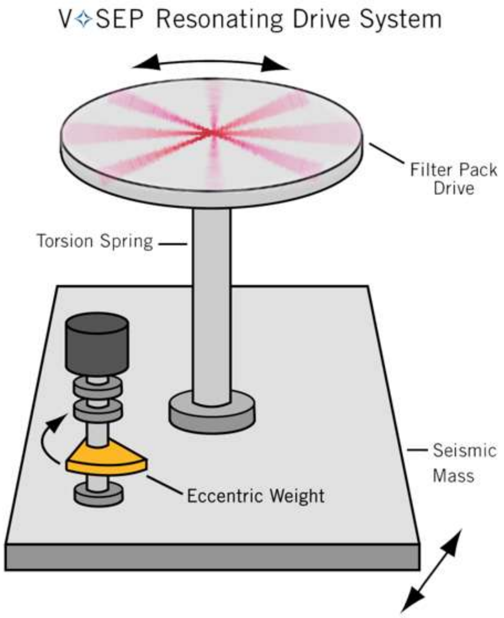

Figure 1 – VSEP Resonating Drive[4]

The membrane vibration produces shear waves that lift the solids, foulants / scales off the membrane surface and direct these to the centre of the flow (and remix with the bulk flow) in the membrane stack. This allows for the processing of high TSS and/or colloidal silica feed, eliminating the need for pre-treatments (hence, simplifying the flowsheet).

Figure 2 illustrates the comparison of fluid dynamics between a conventional crossflow and VSEP filtration.

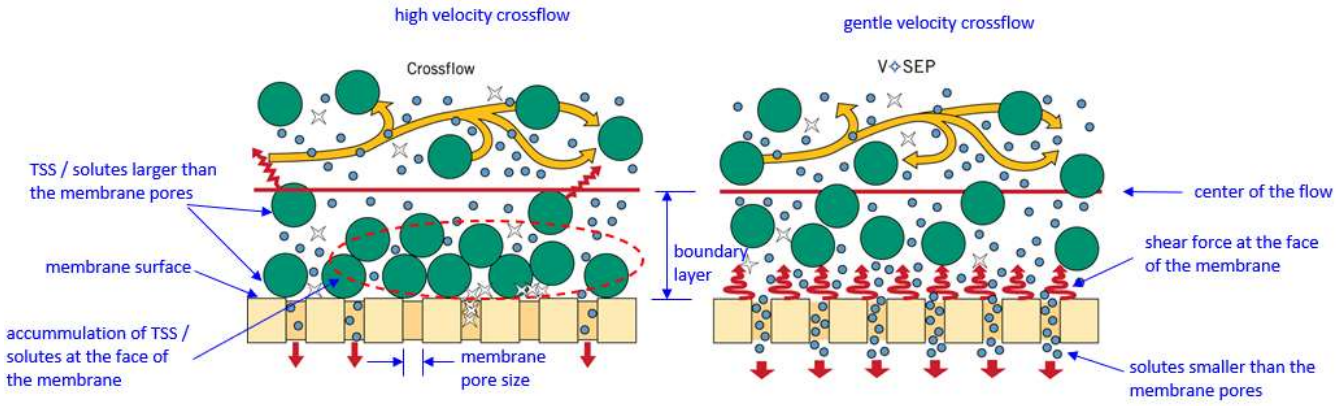

Figure 2 – Fluid Dynamics Comparison (Conventional Crossflow vs VSEP Filtration)[3]

A VSEP membrane filter pack assembly is illustrated in Figure 3. It is composed of:

- Filament-wound outer housing which encases the filter membrane elements.
- Membrane trays – which consists of a stainless-steel disc, drainage cloth and the membrane element.
- Metal spacers and rubber gaskets (o-rings) which separate the separates the tray and provide spacing for the feed to flow over the trays.

The assembly is compressed so that the gaskets form a seal to prevent process leaking. The standard modular size of a VSEP System is 9 to 139 square meters (100 to 1,500 square feet) composed of discs with a 19 inch (48 cm) active diameter[6].

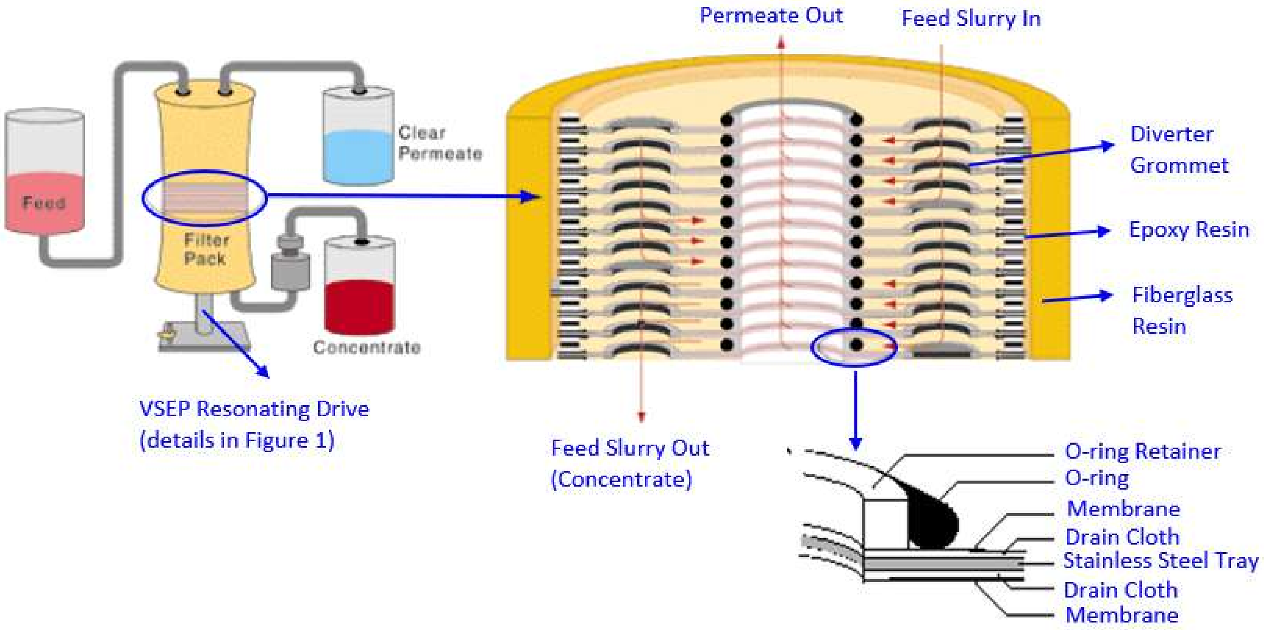

Figure 3 – VSEP Filter Pack Assembly[4]

A VSEP filter unit module is presented in Figure 4 and is made up of the following components:

- Filter Pack – as described and illustrated in Figure 3
- Drive System - as described and illustrated in Figure 1
- Supporting steel structure (frame)
- Feed, permeate and concentrate piping

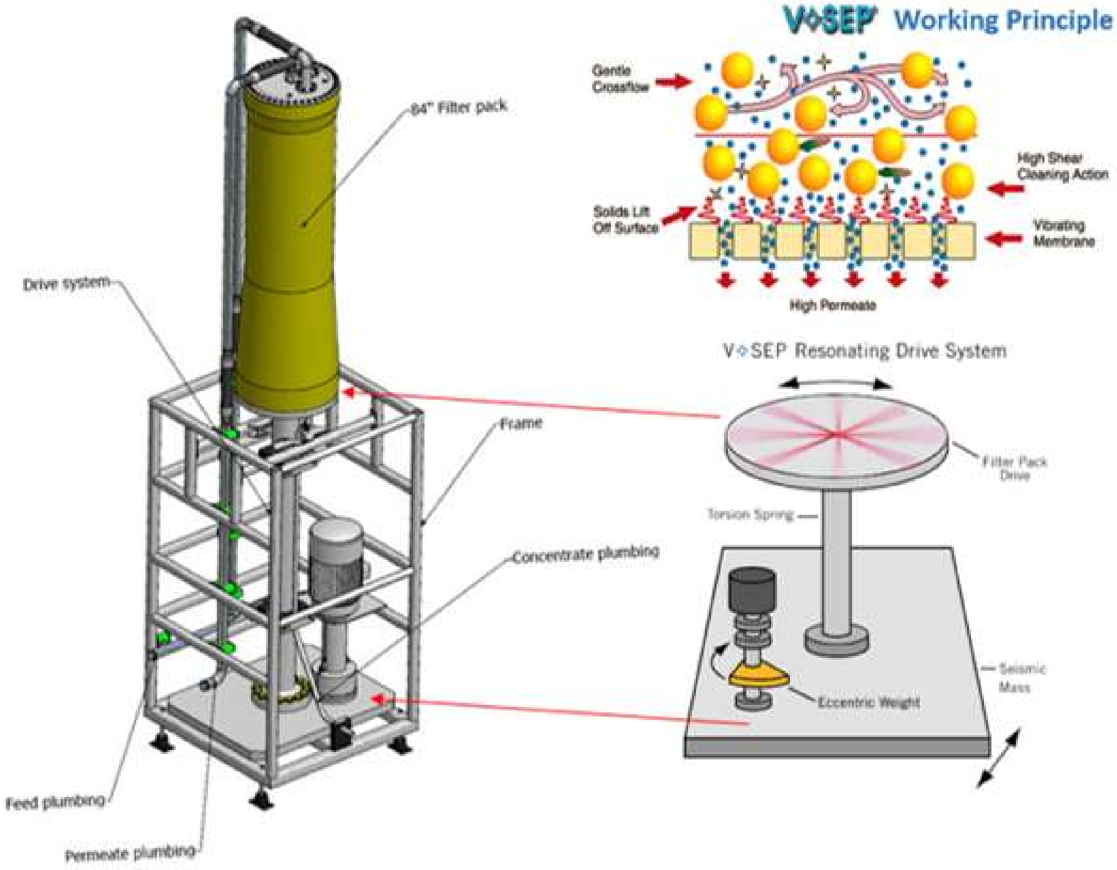

Figure 4 – VSEP Filter Unit Module[6]

NOLANS PROJECT HIGH LEVEL PROCESS DESCRIPTION

The Nolans Rare Earths Project is an integrated mine-to-metal oxide processing plant. The process plant is designed to process 1 Mtpa of ore throughput to produce 340,000 tpa of concentrate. This results to a mine life of 38 years and a nominal production of about 4 440 tpa NdPr oxide, 470 tpa SEG/HRE and 144 400 tpa of 54% w/w P_2O_5 MGA (Merchant Grade Phosphoric Acid) by-product.

A high-level schematic of the process flowsheet is presented in Figure 5.

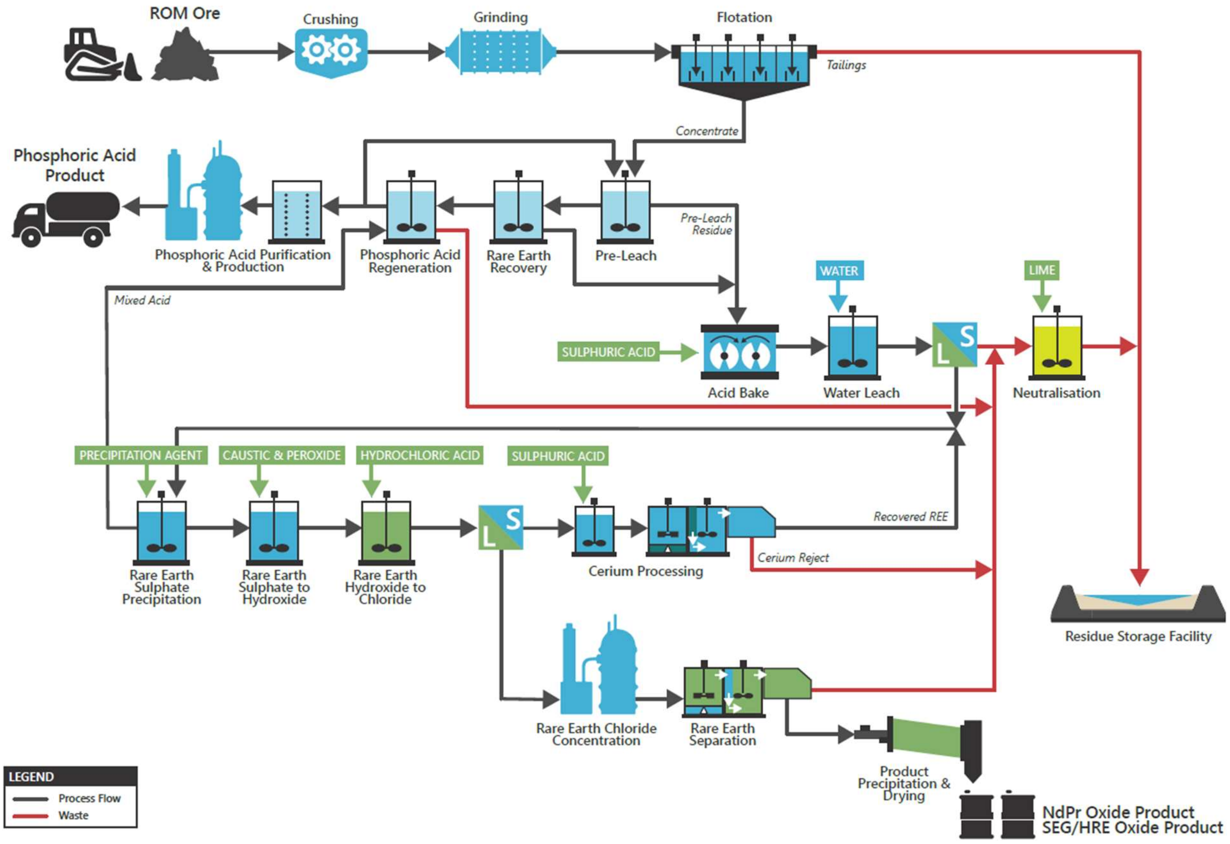

Figure 5 – Nolans Rare Earths Project High Level Flowsheet [1]

The Nolans ore feed is composed mainly of apatite and fluorapatite (REE host minerals), which is the first of its kind in REE processing. The ore is extracted from the deposit using open pit mining method. The ROM ore is transported to the processing plant (8.5 kilometres away from the mine) via haul trucks, where it is crushed, milled and beneficiated using flotation. The beneficiation plant separates majority of the gangue minerals (clays, non-phosphate minerals) from the REE-carrying phosphate minerals, increasing the REE contents from ~2.9% TREO (Total Rare Earths Oxide) in the ore to ~ 6% TREO in the concentrate. The ROM feed also contains elevated thorium (~2 800 ppm w/w%) and uranium (~200 ppm w/w%), of which ~30% is rejected in the beneficiation plant tailings.

The concentrate is pre-leached with regenerated phosphoric acid to selectively remove most of the calcium impurities in apatite / fluorapatite and reduce the solids mass for downstream sulphation. The leached calcium is solubilised as phosphate along with co-extracted REE and impurities such as Al, Mg, Ca, Fe, Th and U. The leach liquor is heated to an elevated temperature to selectively precipitate and recover the REE phosphates leaving behind most of the co-extracted impurities in the solution.

The solubilised calcium, which exists as monocalcium phosphate (MCP) is reacted with a mixed acid regenerated from the downstream water leach process. The mixed acid is composed of 26% w/w H_2SO_4 and 6% w/w H_3PO_4. The addition of sulphuric acid (from the mixed acid) to the MCP - rich liquor regenerates the phosphoric acid and precipitates the calcium as dihydrate gypsum. More than 90% of the phosphoric acid from this process is recycled back to pre-leach to leach the incoming fresh concentrate. The remaining excess regenerated acid is sent to the Phosphoric Acid Purification Area to remove the heavy metal impurities (particularly thorium and uranium) and reduce to trace levels. The gypsum precipitate is one of the final wastes of the process plant. This is treated and subsequently disposed to the Tailings Storage Facility (TSF).

The pre-leached residue and precipitated rare earths phosphates are then fed to the sulphation area, where the combined material is mixed with concentrated sulphuric acid (98% w/w H2SO4) and held for a period of time at 250^{o}C. The baking process effectively “cracks” the occluded refractory rare earths, so that it will be amenable to the subsequent water leaching. In water leaching, the baked material is mixed with water to solubilise the rare earths as sulphates. Phosphoric acid is also generated (from sulphuric acid leaching of the remaining apatite), along with the free sulphuric acid.

The rare earths sulphates are recovered from the liquor as sulphate precipitate through the addition of methanol before sending to impurity removal stages. The post-purification precipitate, virtually free of major impurities is dissolved with hydrochloric acid to selectively dissolve the target non-cerium rare earths (SEG/HRE, Nd, Pr), leaving the cerium behind in solids. The resulting solution is a target rare earths - rich chloride liquor ready for separation in the succeeding solvent extraction stages. The cerium which remains in the precipitate, is dissolved separately with sulphuric acid.

The resulting cerium-rich sulphate solution is sent to its dedicated solvent extraction to separate the cerium from the residual SEG/HRE, NdPr rare earths. The cerium, which is loaded in the organic is stripped with sulphuric acid and treated prior to tailings disposal. The target rare earths contained in the raffinate is recycled back to the water leach.

The SEG/HRE is separated from the NdPr through the first solvent extraction, where the former is loaded to the organic and stripped with hydrochloric acid. The raffinate, which contains the NdPr-rich solution is forwarded to the second solvent extraction where the NdPr are loaded into the organic and stripped with hydrochloric acid.

The different high purity product liquors (SEG/HRE and NdPr chloride solutions) are subsequently recovered as oxalate precipitates in their respective trains. The individual precipitates are calcined to convert the solids to SEG/HRE oxide and NdPr oxide final products.

PHOSPHORIC ACID PURIFICATION AREA IN DETAIL

In the Nolans flowsheet, phosphoric acid purification (PA Purification) is achieved by using nanofiltration technology, where molecules with large ionic size such as thorium and uranium are rejected by the membrane while small neutral molecules pass through the membrane. Nanofiltration technology is an established process for removing uranium and thorium in leach liquors and recovery of water in bitterns (RO concentrate).

The PA purification flowsheet employs VSEP nanofiltration (Vibratory Shear Enhanced Processing), where vibratory shear waves is directly applied in the membrane surface (as described in the previous section). In this manner, the VSEP is able to process raw acid feed from the PA regeneration and mixed acid circuits with relatively high TSS, high TDS and high acidity.

The VSEP nanofiltration has been proven in an industrial application of phosphoric acid recovery in a fertiliser plant in the US. Arafura had done a number of laboratory and pilot testworks including feed and membrane variability tests to establish its technical viability to the Nolans flowsheet.

The PA purification is comprised of two stage nanofiltration described as follows (refer to Figure 6 below):

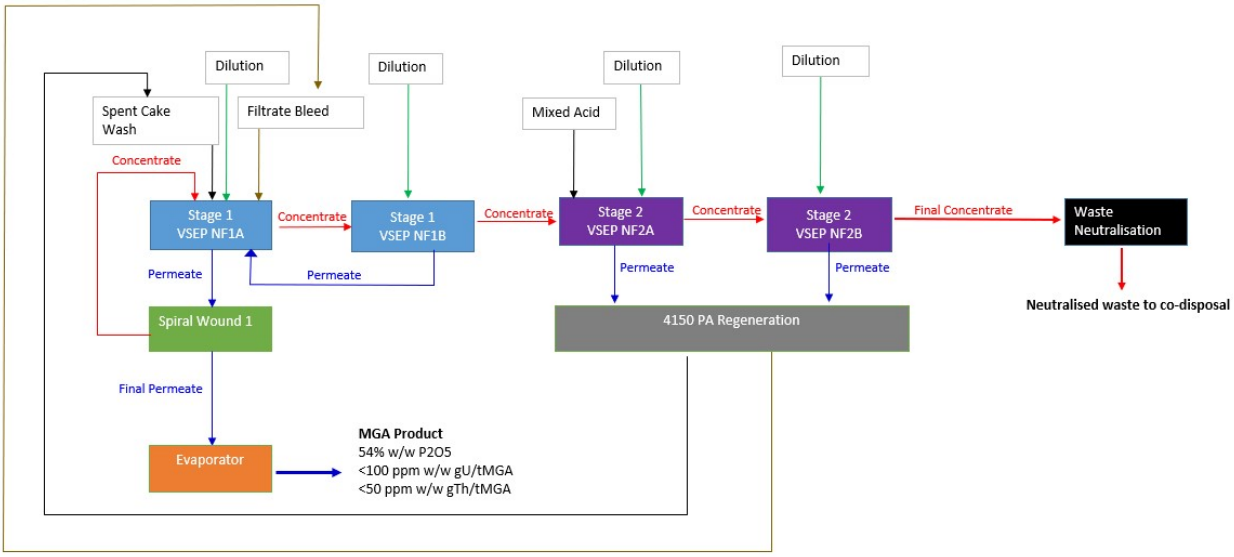

Figure 6 – Nolans Rare Earths Project Phosphoric Acid Purification Flowsheet

Stage 1 Nanofiltration - processes the excess phosphoric acid feed from the PA regeneration area, where the membrane rejects dissolved impurities, including U and Th to meet the phosphoric acid product specifications. Stage 1 uses two passes of nanofiltration for the permeate to maximise the phosphoric acid recovery within the constraints of impurity concentration. The concentrate (retentate) produced in Stage 1 flows in series to Stage 2 for further recovery of residual phosphoric acid.

Stage 2 Nanofiltration - processes the mixed acid recovered from the extraction area and Stage 1 concentrate. The membrane rejects dissolved impurities, including Al, Mg, Ca, U and Th to minimise the build-up of these impurities in the upstream circuit. The permeate collected from each pass (containing high concentrations of sulphuric acid and some phosphoric acid) is combined and recycled to the PA regeneration area for gypsum precipitation. The concentrate (retentate) produced in Stage 2 flows in series, with the final concentrate produced after the second pass.

Three parameters are key to the nanofiltration of the Nolans acid feeds:

- **Flux rate** – refers to the flowrate of the permeate and dictates the sizing or number of unit modules required. The Stage 1 VSEPs have flux rates ranging at 9-15 GFD (gallons of permeate per square feet/day) and Stage 2 have higher flux rates at 14-22 GFD

- **Rejection rate** – refers to the efficiency of molecule rejection by the membrane, which is typically around 15% phosphoric acid, >50 – 70 % uranium and thorium rejection in both stages.

- **Membrane life** – degradation resistance of the membrane from high acidity and high-pressure exposure

The final permeate leaving the polishing spiral wound nanofiltration contains about 15% w/w P_2O_5. This is sent to an evaporator, where the water is driven-off to achieve the 54% w/w P2O5 concentration for the merchant grade phosphoric acid (MGA) specification.

The final concentrate generated at the end of Stage 2 nanofiltration contains elevated levels of thorium and uranium (following the series of concentration). This is sent to waste neutralisation to stabilise and immobilise the dissolved metals (as hydroxide precipitate) prior to disposal to the RSF. Figure 7 illustrates the PA Purification circuit, with VSEP unit modules installation in the Nolans hydrometallurgical processing facility.

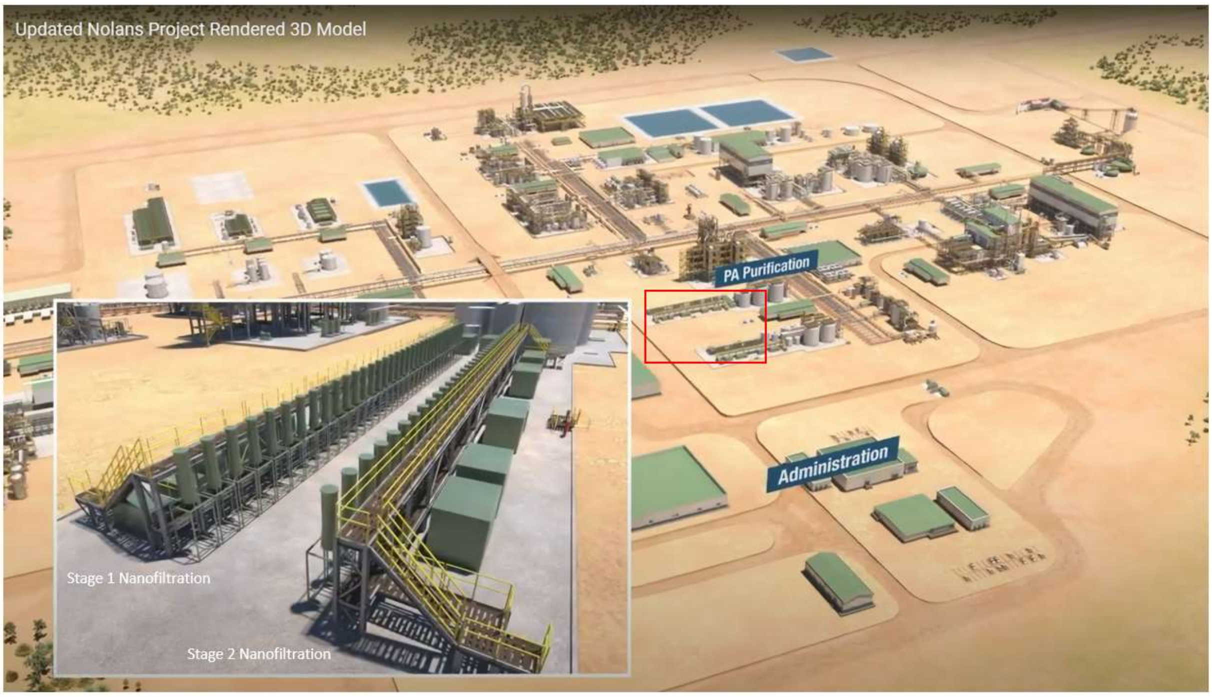

Figure 7 – PA Purification Circuit at Nolans Hydrometallurgical Processing Facility [7]

LABORATORY SCALE TESTWORK RESULTS

Overview

NLR and Arafura had begun working for the laboratory scale test work on Q1 2022. The testing was performed on synthetic solution of simulated acid streams with compositions nominated by Arafura. Initial work involved a thorough analysis of various nanofiltration membranes to ascertain instantaneous flux and rejection rates on the simulated acid streams. The most effective membranes were used to concentrate the simulated acid streams to produce clean permeated acid and concentrated reject acid streams. These concentration tests also allowed for the determination of average flux and clean permeated acid recovery rates. Simultaneously, long-term soak testing was performed on the entire suite of possible nanofiltration membranes to ensure that no significant loss flux or solute rejection would occur from continued exposure of the membranes to the acid solution.

The VSEP LP (Lab & Pilot) unit was used for the laboratory scale test work. The unit was set up in L-mode (Lab Mode) and utilized a single sheet of membrane surface area at 0.5 square feet (0.046 m^2). The surface area of a the full-scale industrial VSEP unit is 2,400 times the surface area of this unit, and thus would have 2,400 times the volume throughput. Figure 8 shows a typical set up for a VSEP LP in L-mode.

Figure 8 – VSEP LP in L-mode (left) with pump (center) and feed tank (right).

The system was operated in either recirculation mode (membrane selection) or batch mode (concentration studies), the only difference between the two being that in batch mode, the VSEP permeate is removed from the system as the acid concentrates, while in recirculation mode, the VSEP permeate is returned to the system. Recirculation mode is only used during the initial membrane selection, whereas batch mode is used in all other testing simulating the full-scale operation.

In batch mode, a finite volume of feed is processed. The concentrate, or reject, is sent back to the feed tank and the permeate, or filtrate, is sent to a separate destination. As the permeate is removed, the feed tank volume is decreased and becomes concentrated until an end point based on flow or other desired set point. The concentrate flow is held constant to maximize crossflow, and, in combination with vibration, will reduce the overall rate of fouling. The advantages of batch mode are slightly higher flux rates and slightly lower cleaning frequency. Figure 9 below shows a diagram of batch mode operation.

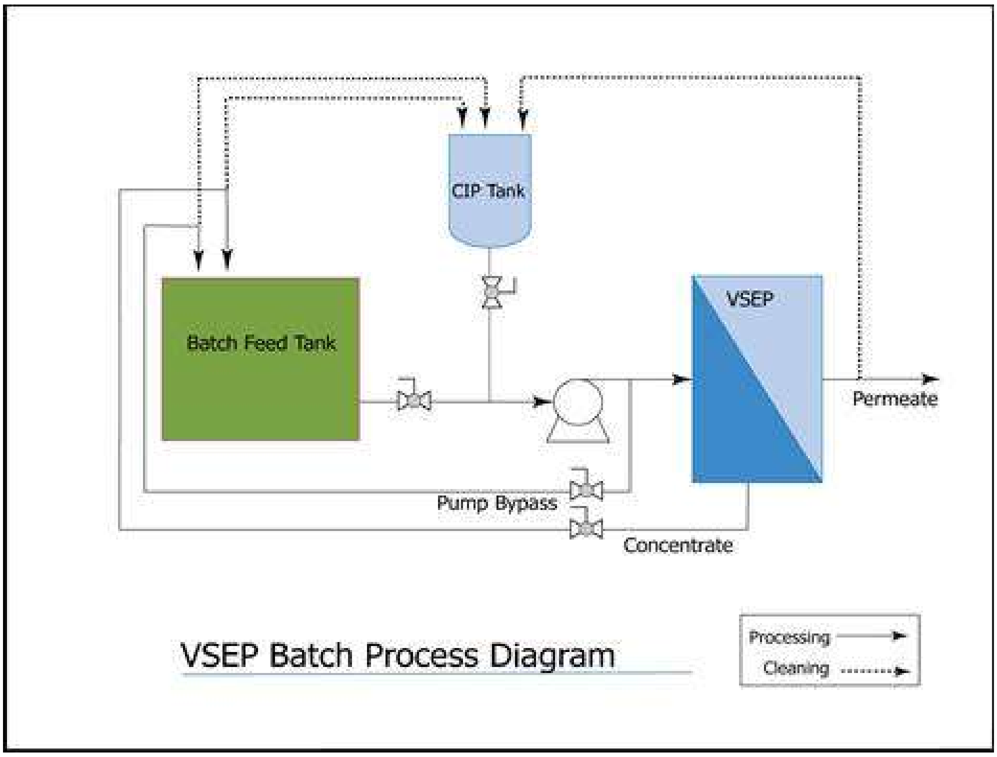

Figure 9 – VSEP Batch Mode Operation

Membrane Selection

A suite of nanofiltration membranes was tested with the simulated acid streams to determine which membranes had the highest permeate flux and solute rejection. The pore size of the membranes tested ranged from a molecular weight cut off (MWCO) of 100 to 800. A lower MWCO indicates higher solute rejection, while a higher MWCO indicates lower solute rejection. Typically, magnesium sulphate is the standard used to evaluate rejection for nanofiltration membranes. As uranium and thorium are the key contaminants in the acid streams, surrogates for tracking their partitioning were needed. Uranium rejection tracks relatively close to magnesium rejection, while thorium rejection tracks relatively close to lanthanum rejection, so these were the main solutes followed in the simulated acid streams. Each membrane was tested on an identical acid solution in the VSEP system at 40°C and the results were compared.

Table 1 shows the standard manufacturer information for the membranes tested.

Table 1 – Standard Manufacturer Membrane Data

Membrane	Pore Size	Max Temp	pH Tolerance	Water Flux[1,2]	Material
NF-1	100 da	80°C	0 to 12	13 GFD	Thin-film Non-polyamide
NF-2	200 da	80°C	0 to 12	17 GFD	Thin-film Non-polyamide
NF-3	300 da	70°C	1 to 14	62 GFD	Thin-film Non-polyamide
NF-4	300 da	90°C	1 to 14	18 GFD	Polyethersulfone on PP
NF-5	500 da	70°C	1 to 14	95 GFD	Thin-film Non-polyamide
NF-6	800 da	90°C	1 to 14	107 GFD	Polyethersulfone on PP

1. Average Batch Cell Test Results in GFD on new membrane @ 25ºC
2. GFD = Gallons of permeate produced per square foot of membrane per day. 1 GFD = 1.7 LMH (liters per square meters per day).

The membranes were compared using the permeate flux and the solute rejection. In general, the flux rates and rejection rates tracked well with the pore size (MWCO) of the membranes. Membranes with lower MWCO had lower flux rates and higher rejection, while membranes with higher MWCO had higher flux rates and lower rejection. To ensure that the quality of the permeate was satisfactory with respect to lanthanum and magnesium, only NF-1, NF-2, and NF-3 were progressed for further testing.

Table 2 shows the instantaneous flux and rejection data for the membranes tested.

Table 2 – Instantaneous Flux and Rejection Data

Membrane	Permeate Flux[1]	Pressure	Conductivity	pH	%Solids[2]	Lanthanum	Magnesium
Feed	-	-	108,000 µS/cm	1.9	18.79%	164 ppm	473 ppm
NF-1	1.0	500 psi	76,800 µS/cm	2.0	12.77%	4.6 ppm	11.6 ppm
NF-2	2.1	500 psi	78,800 µS/cm	1.9	13.16%	12.8 ppm	26.7 ppm
NF-3	1.2	500 psi	98,800 µS/cm	1.7	17.86%	62.5 ppm	184 ppm
NF-4	1.2	500 psi	96,800 µS/cm	1.7	17.27%	72.7 ppm	256 ppm
NF-5	3.3	500 psi	96,000 µS/cm	1.8	17.65%	118 ppm	337 ppm
NF-6	8.2	500 psi	91,700 µS/cm	1.8	18.51%	155 ppm	447 ppm

1. Permeate Flux is given as a ratio of permeate flux of the membrane to the lowest observed flux.
2. % Solids means total TSS + TDS

Membrane Soak Testing

In parallel to the membrane selection testing, soak testing of the membranes was taking place. The membranes were soaked in identical phosphoric acid solutions (same to that of the test concentration) and periodically removed from the solution and tested on clean water to track changes in flux and magnesium sulphate rejection over time. Within a few months it was observed that only NF-3 and NF-5 membranes were capable of being exposed to the acid solutions and not have significant changes in flux or rejection. Notably, the best rejecting membranes, NF-1 and NF-2, quickly lost rejection and gained flux, indicating that damage to the membranes from the acid was significant. As NF-5 membrane did not satisfy permeate quality limitations during the membrane selection, NF-3 membrane was the only membrane that was acceptable for long-term acid filtration. The NF-3 membrane was used for the remainder of the testing.

Figure 10 below shows the flux rates for each of the membranes over time, soaked in the phosphoric acid solution. Figure 11 shows the magnesium sulphate rejection for each of the membranes over time soaked in the phosphoric acid solution. After it was determined that only NF-3 would be appropriate to use, the NF-3 membrane was also put through a soak test with a sulfuric acid-based solution simulating the mixed acid feed. Figure 12 below shows the flux rates and magnesium sulphate rejection rates over time for the NF-3 membrane soaked in the sulfuric acid-based solution.

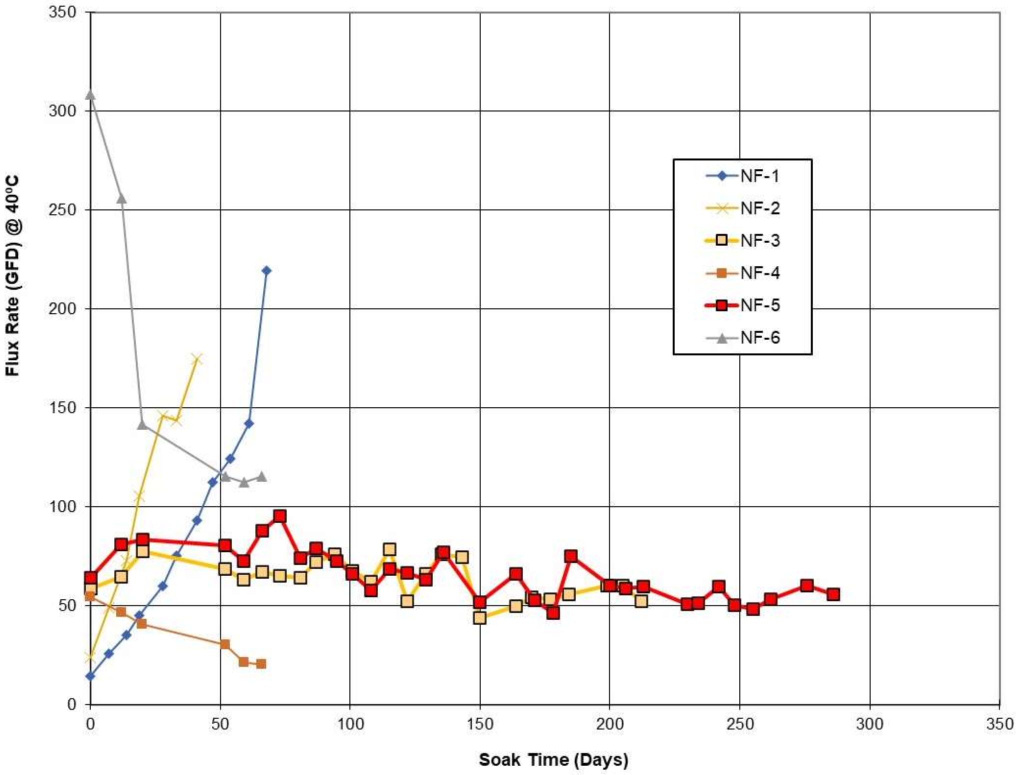

Figure 10 – Phosphoric Acid Soak Test Flux Rates

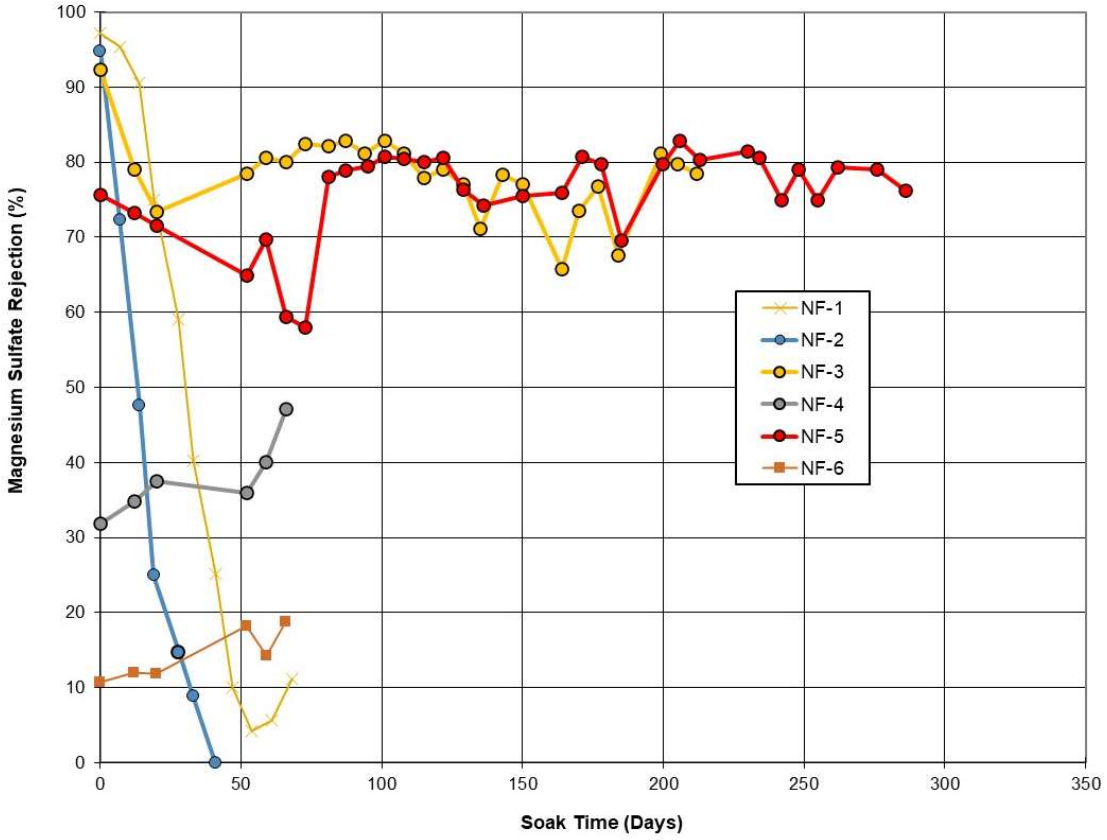

Figure 11 – Phosphoric Acid Soak Test Magnesium Sulphate Rejection Rates

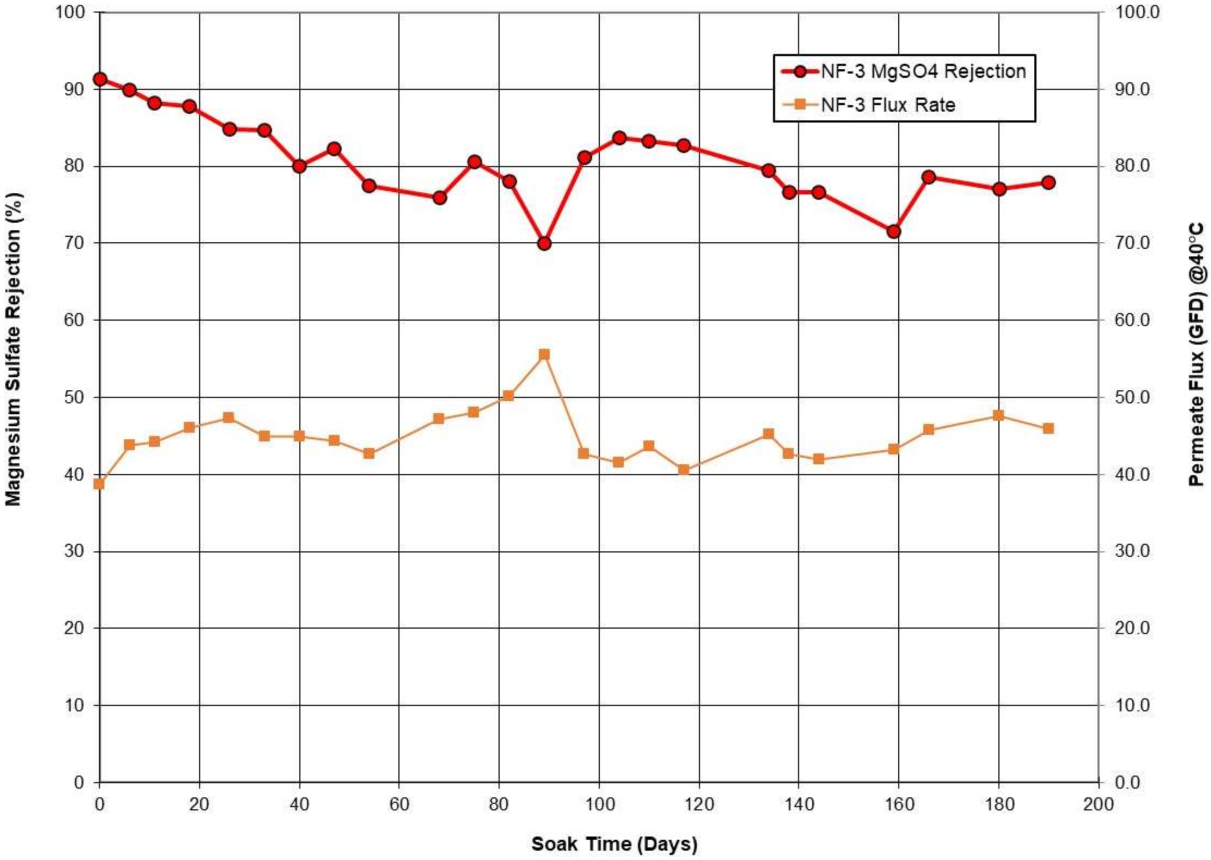

Figure 12 – Sulfuric Acid NF-3 Soak Test Flux and Magnesium Sulphate Rejection Rates

NF-3 L-Mode Concentration Testing

The NF-3 membrane was used to concentrate the phosphoric and sulphuric acid-based streams in the overall acid purification process flow sheet. The flow sheet is shown in Figure 13 below. Incoming phosphoric acid-based material was diluted with water and spent flush water and fed into NF1A. The concentrate from this stage is diluted with water and fed into NF1B. The permeate from these two stages feeds the evaporator to produce saleable MGA product.

The concentrate from NF1B is blended with sulphuric acid based mixed acid, diluted with water, and fed to NF2A. The concentrate from NF2A is diluted with water and fed to NF2B. The permeate from NF2A and NF2B is recycled back into the refining process, while the concentrate from NF2B is neutralized for disposal. Dilution rates at the beginning of each stage targets 180 g/L phosphorus plus sulphur (180 g/L P+S) to keep viscosity at a reasonable level for adequate filtration rates. Volumetric recovery rates were targeted at 80% +/- 5% for each stage as higher recovery with nanofiltration membranes can lead to significant bleed of undesirable contaminants to the permeate. In addition, unnecessarily high permeate recovery can also cause decreasing acidity and lead to thorium phosphate precipitation in NF1B, and excessive gypsum precipitation in NF2A and NF2B due to the presence of sulphuric acid mixed with the monocalcium phosphate-rich concentrate from NF1B. While the vibration of the VSEP system can minimize the effects of precipitation on the membrane system itself, excessive precipitation could eventually cause pumping issues and damage to downstream equipment.

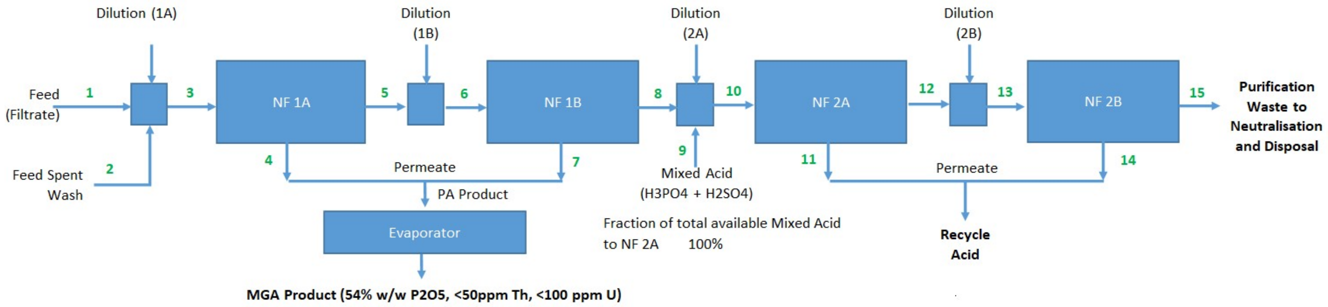

Figure 13 – Laboratory Scale Testwork Flowsheet

With so many recirculating streams, isolation of each stage was necessary. Thus, the testing was run in an open-loop fashion, in which each stage was independent of the other stages. Iterative modelling based on the initial rejection from the membrane selection informed the composition of the solutions for the concentration studies performed on each stage. The results of the four concentrations are shown in Table 3 below. Images of the samples from each stage are shown in Figures 14 through 17.

Table 3 – NF-3 L-Mode Concentration Testing Average Flux, Permeate Recovery & Analytical

Sample	Average Flux[1]	%Recovery	Conductivity	pH	%Total Solids[2]	Lanthanum	Magnesium
NF1A Feed	-	100%	81100 µS/cm	1.5	16.82%	127 ppm	388 ppm
Composite Perm.	1.6	74.3%	86100 µS/cm	2.1	14.19%	30.8 ppm	142 ppm
Final Concentrate	-	25.7%	88100 µS/cm	2.2	23.77%	459 ppm	1260 ppm
NF1B Feed	-	100%	55400 µS/cm	2.2	16.69%	135 ppm	694 ppm
Composite Perm.	2.0	79.9%	56800 µS/cm	1.9	11.92%	81.4 ppm	438 ppm
Final Concentrate	-	20.1%	57900 µS/cm	2.0	27.06%	186 ppm	1510 ppm
NF2A Feed	-	100%	443000 µS/cm	1.3	16.92%	259 ppm	43.8 ppm
Composite Perm.	2.5	77.5%	395000 µS/cm	1.5	14.20%	57.5 ppm	15.7 ppm
Final Concentrate	-	22.5%	422000 µS/cm	1.4	17.59%	788 ppm	121 ppm
NF2B Feed	-	100%	425000 µS/cm	1.7	17.17%	885 ppm	625 ppm
Composite Perm.	1.0	79.7%	383000 µS/cm	1.5	12.31%	423 ppm	322 ppm
Final Concentrate	-	20.3%	437000 µS/cm	1.2	19.60%	2360 ppm	1670 ppm

1. Permeate Flux is given as a ratio of permeate flux of the membrane to the lowest observed flux.
2. % Solids means total TSS + TDS

Figure 14 – NF-3 L-Mode NF1A Feed, Permeate, and Concentrate (left to right)

Figure 15 – NF-3 L-Mode NF1B Feed, Concentrate, and Permeate (left to right)

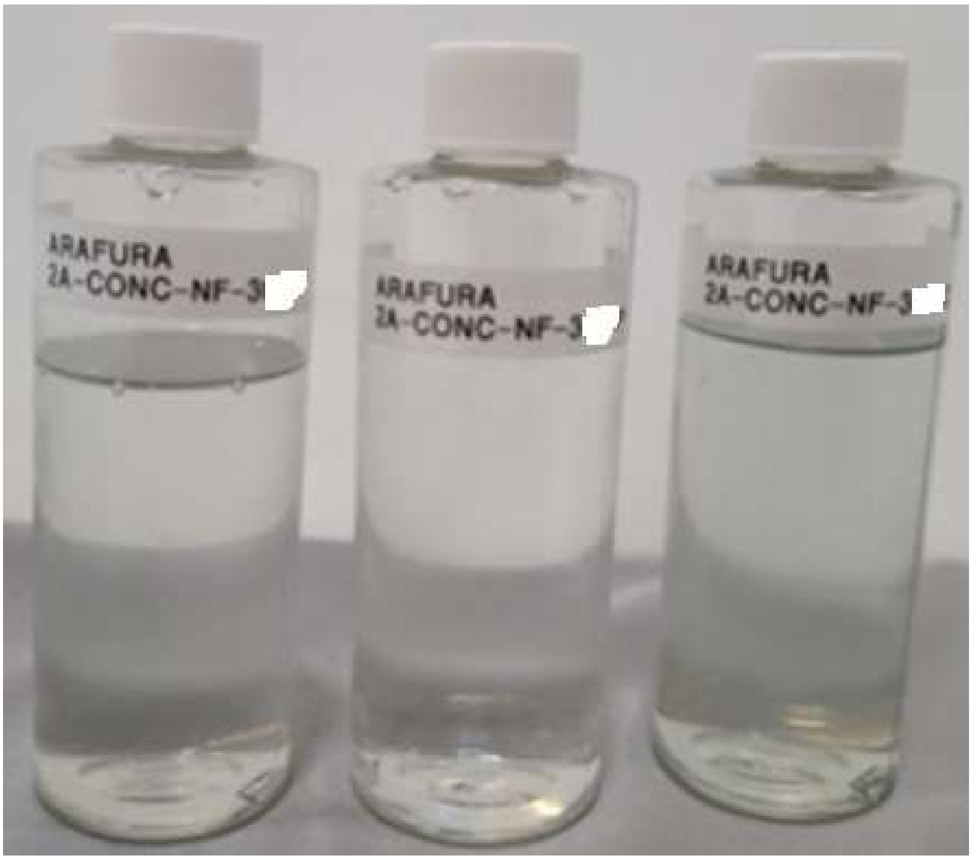

Figure 16 – NF-3 L-Mode NF2A Feed, Permeate, and Concentrate

Figure 17 – NF-3 L-Mode NF2B Feed, Concentrate and Permeate (left to right)

PILOT SCALE TESTWORK RESULTS

Overview

Laboratory scale test work was completed in October of 2022, the results of which informed the optimum flowsheet for pilot scale test work (refer to Figure 6 above). The optimised flowsheet incorporates both VSEP and traditional spiral-wound nanofiltration (both utilizing the NF-3 membrane). Testing began in January 2023 and is ongoing. Testing was performed with simulated acid streams with compositions designated by Arafura based on the optimum flowsheet.

The VSEP LP (Lab & Pilot) unit was used for the pilot scale test work. The unit was set up in P-mode (Pilot Mode) and utilized 19 double-sided trays of membrane surface area at 16.7 square feet (1.55 m^2). The surface area of a the full-scale industrial VSEP unit is 72 times the surface area of this unit, and thus would have approximately 72 times the volume throughput. Figure 18 shows a typical set up for a VSEP LP in P-mode.

Figure 18 – VSEP LP in P-mode (left) with pump (center) and feed tank (right)

NF-3 P-Mode Concentration Testing

The NF-3 membrane was used to concentrate the phosphoric and sulfuric acid-based streams in the pilot flowsheet (Figure 6). Extensive modelling was performed to allow for Stage 1 and Stage 2 to be run independent of each other, but unlike the L-Mode testing, the products within each stage were carried over to the next step within the stage to better simulate actual operation.

Multiple repeat runs were performed on different NF-3 membrane stacks to ensure proper flux and analytical averages are covered. The results of the VSEP and spiral concentrations are shown in Table 4 below. Images of the samples from each stage are shown in Figures 19 through 23 below. The significant amounts of precipitated salts in the photos highlights the need for VSEP's vibration to ensure no plugging and higher throughput compared to traditional spiral wound systems.

Table 4 – NF-3 P-Mode Concentration Testing Average Flux, Permeate Recovery & Analytical

Sample	Average Flux[1]	%Recovery	%Total Solids	pH	Lanthanum	Magnesium
NF1A Feed	-	100%	26.39%	1.3	240 ppm	1218 ppm
Composite Perm.	3.1	48.7%	24.60%	1.3	92 ppm	445 ppm
Final Concentrate	-	51.3%	29.61%	1.3	397 ppm	1855 ppm
NF1B Feed	-	100%	28.75%	1.3	373 ppm	1741 ppm
Composite Perm.	2.2	77.8%	26.32%	1.3	205 ppm	982 ppm
Final Concentrate	-	22.2%	39.79%	1.3	1124 ppm	5195 ppm
NF1A Spiral Feed	-	100%	24.60%	1.3	92 ppm	445 ppm
Composite Perm.	1.0	79.1%	22.10%	1.3	21.3 ppm	111 ppm
Final Concentrate	-	20.9%	26.75%	1.3	284 ppm	1328 ppm
NF2A Feed	-	100%	33.60%	0.5	3058 ppm	1217 ppm
Composite Perm.	3.5	78.6%	30.92%	0.5	1057 ppm	572 ppm
Final Concentrate	-	21.4%	43.47%	0.5	8844 ppm	4384 ppm
NF2B Feed	-	100%	32.51%	0.4	6688 ppm	2886 ppm
Composite Perm.	3.7	77.9%	32.14%	0.4	3582 ppm	2063 ppm
Final Concentrate	-	22.1%	41.93%	0.5	9721 ppm	5669 ppm

1. Permeate Flux is given as a ratio of permeate flux of the membrane to the lowest observed flux.
2. % Solids means total TSS + TDS

While the pilot testwork is still in progress, it was observed that key rejection rates are well within the range of the averages of the laboratory results. Additionally, the average flux at increased feed concentration in the pilot testwork (150% more than the L-mode feed concentration) is also within the range of predicted limits. By concentrating the feed and pushing the limits the membrane to an optimum point, dilution water requirement for each stage is minimised. This results to the reduction of water make-up to the circuit and reduction of evaporative requirements in the phosphoric acid evaporator. By far, the optimised conditions of the pilot testing established the flexibility of the PA purification flowsheet to manage feed variabilities and room for further optimisation during actual operations.

Figure 19 – NF-3 P-Mode NF1A Feed, Concentrate and Permeate (left to right)

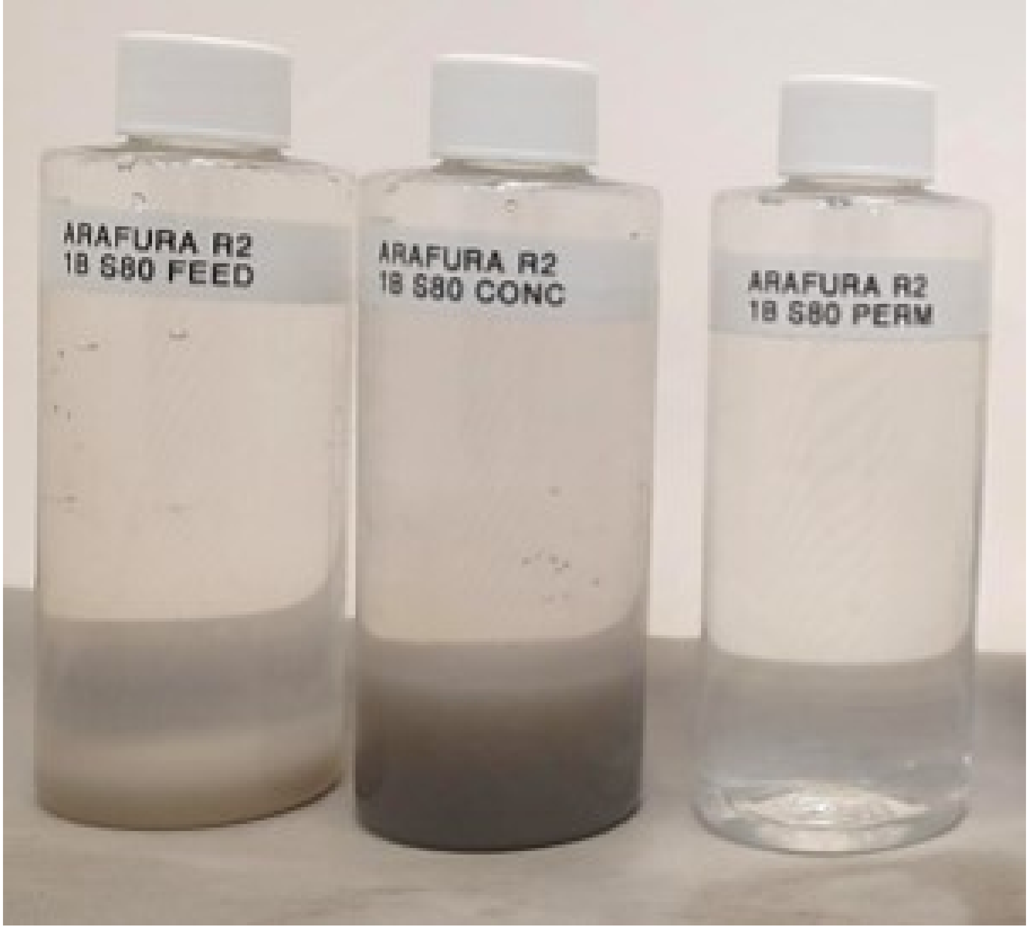

Figure 20 – NF-3 P-Mode NF1B Feed, Concentrate and Permeate (left to right)

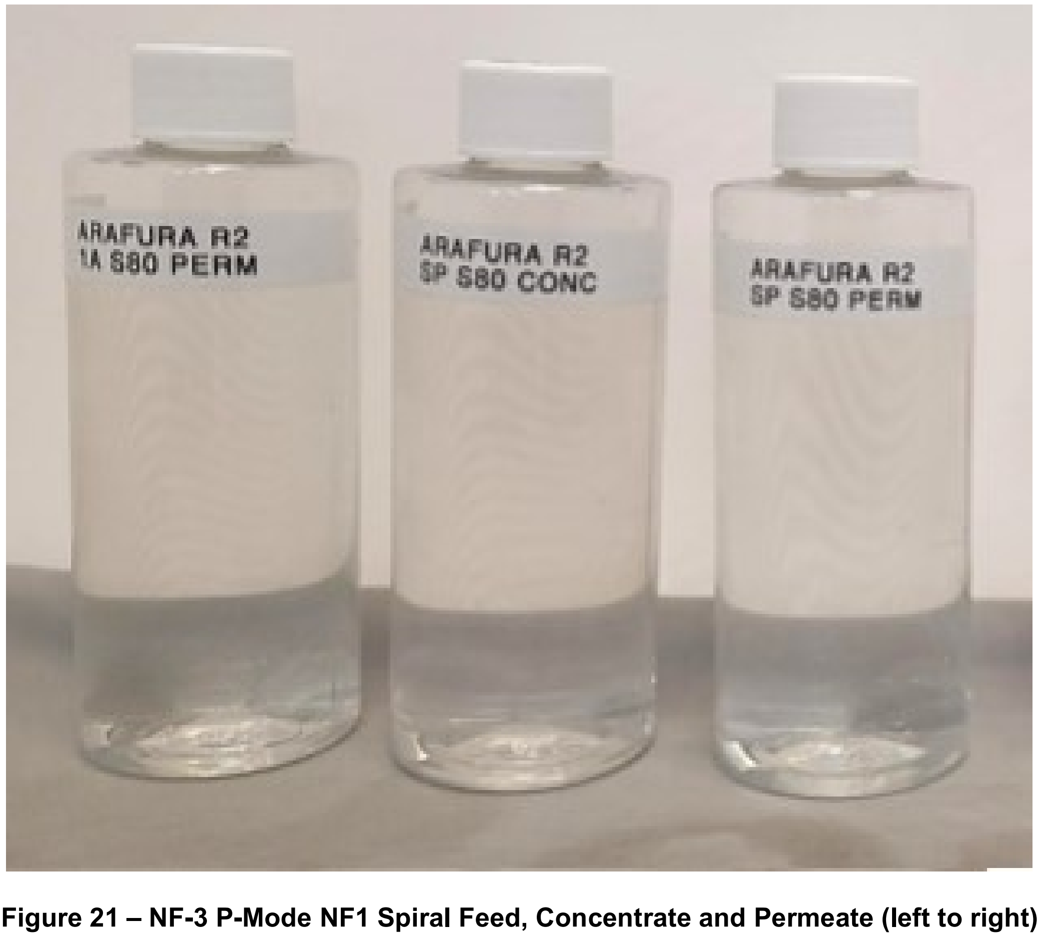

Figure 21 – NF-3 P-Mode NF1 Spiral Feed, Concentrate and Permeate (left to right)

Figure 22 – NF-3 P-Mode NF2A Feed, Concentrate and Permeate (left to right)

Figure 23 – NF-3 P-Mode NF2B Feed, Concentrate and Permeate (left to right)

CONCLUSIONS

The PA Purification circuit is a strategic and integral part of the overall Nolans Rare Earths flowsheet. Following the results of laboratory and pilot testworks, it was established that nanofiltration process offers a viable technical solution to recover and purify the excess phosphoric acid and mixed acid (sulphuric + phosphoric acid) generated from the Nolans process. The wealth of data collected and analysed from the testworks established the ability of the flowsheet to manage variabilities and thus provides both Arafura and NLR a key confidence in the solution.

The application of NLR's innovative VSEP (Vibratory Shear Enhanced Processing) to the Nolans nanofiltration process provides operational flexibility and economic viability of the solution to the project.

The PA Purification circuit is now entering the detailed design engineering phase. As the project progresses, it is envisaged that the collaborative work of NLR and Arafura will go beyond the delivery of the Nolans project. The aim is to continuously improve the efficiency of the circuit with feed variability, along with the evolution in membrane filtration technology (i.e. new membrane, feed conditioning etc.).

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