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APPLICATION OF NANOFILTRATION TECHNOLOGY TO IMPROVE SEA WATER NEUTRALIZATION OF BAYER PROCESS RESIDUE

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Abstract

Sea water neutralization of alkaline Bayer process solid waste residue is a sustainable solution to turn a hazardous waste material into a benign, non-hazardous material that can be re-used in some applications. The concentrations of the active neutralizing agents in sea water, calcium and magnesium, are low and large volumes of sea water are required to neutralize the alkaline residue.

Nanofiltration of sea water can produce a concentrate containing up to four times the calcium and magnesium levels in sea water, while the mono-valent ions, like sodium and chloride, are only marginally concentrated. This significantly reduces the volume of sea water required for the neutralization process and hence the size of neutralization equipment. Other advantages include improved reaction kinetics and reduced salinity of the neutralized residue, making it more suitable for certain applications. The nanofiltration permeate, with lower scaling potential, is also an improved feed for sea water reverse osmosis plants to produce potable water.

Introduction

Nanofiltration (NF) is a membrane-based separation method that is similar to reverse osmosis (RO) but the NF membranes are more selective in retaining chemical species than RO membranes. RO membranes retain >99.0% of all chemical species in solution, except for some small, polar organic molecules such as acetone and acetaldehyde. NF membranes on the other hand will allow mono-valent ions such as sodium and chloride to pass through to permeate but will retain divalent species, such as calcium and magnesium, in the retentate.

In general, the species that are retained by an NF membrane are multi-valent inorganic ions and large organic molecules. Different NF membranes have different rejection characteristics depending on their surface chemistry and surface charge density and are normally characterized by measuring the rejection of magnesium sulfate, typically in a range between 95% and 99%.

Typical applications for NF membranes are concentration of dissolved metals in acid solutions, concentration of dyes and sugars, removal of chloride from sulfate solution and removal of calcium from solution to stop scaling. Nanofiltration technology has also been applied to pre-treat sea water for desalination plants, where the NF membranes have been used to remove calcium, magnesium and sulfate prior to reverse osmosis treatment. It has been shown that the RO membranes, or thermal equipment in the case of distillation plants, operate more efficiently if NF pretreatment is applied [1].

The concentration of species retained by the NF membranes is low and hence the operating pressure is also low when compared to the operating pressures of sea water reverse osmosis (SWRO) plants. Typical sea water NF plant operating pressures are around 18 bar compared to around 60 bar for SWRO plants [2]. The capacity for concentrating calcium (Ca) and magnesium (Mg) ions is therefore likely to be limited by precipitation rather than osmotic pressure.

The production of alumina by the Bayer process results in the generation of large quantities of "red mud". Between one and two tonnes of residue are typically generated for every one tonne of alumina produced and, due to its high alkalinity and pH, the red mud generated from alumina refineries is classified internationally under the Basel Convention (Article 1, paragraph 1a) as a "hazardous waste" (classification #B2110) and its trans-boundary movement is tightly controlled [3].

The alumina industry has for many years sought ways to minimize and reduce the environmental and social impacts of red mud and alumina refineries face two basic choices in managing their red mud residue, either:

i)   store the residue indefinitely at considerable cost; or

ii)   attempt to reuse it in some form [4],[5].

Long-term storage costs and environmental liabilities are high, and it is estimated that management of residue in Australia alone costs more than AUD80 million per year [6].

Refinery waste reuse requires some form of conversion from its highly caustic state to a more environmentally benign and "user-friendly" state for both transport and reuse applications. Thus, it is desirable to neutralize refinery residues in a way that they are no longer highly caustic, irrespective of their potential or actual reusability.

Most neutralization options that attempt to address red mud residue storage and management problems do not make full use of the inherent acid neutralizing, metals and phosphate binding, and odour destroying properties of the neutralized red mud residue. One method for neutralizing alumina refinery residues, called Basecon Technology can be used to economically convert the residue and thereby imbues it with environmental and commercial value [7]-[11]. Such a technology can reduce the long-term environmental, financial and social liabilities of storing caustic residues, and has the added advantage of producing a reusable solid material, called Bauxsol Raw Material (BRM), which has favorable environmental and industrial reuse characteristics. Different reactants, including sea water, brines, concentrated brines and metal salts can be used to neutralize red mud residue.

Sea water neutralization is the most widely implemented neutralization process, due to its cost-efficiencies and readily available feedstock [12] for alumina refineries located close to the sea, and having environmental licenses that provide access to sea water. When sea water is used to neutralize red mud, the volume of sea water added is between three and ten times the volume of

red mud slurry being treated (also refer Table 4). Nanofiltration now offers an opportunity to significantly reduce the volumes required.

The expected advantages of using NF concentrate to neutralize red mud are:

- Reduction in reacting volume and therefore tanks, pumps, piping and the capital and operating costs associated with this equipment.

- Reduction in salinity of the neutralized product, thus making it more useable for soil remediation and other similar applications.

- Production of an NF permeate stream that will perform better in SWRO plants than untreated sea water.

An R&D program was initiated to determine the extent to which sea water could be concentrated using NF membranes before scaling commenced, or before the osmotic pressure became excessive. In addition the comparative reactivity of the sea water NF concentrate and untreated sea water with red mud was investigated in order to gain an understanding of the equipment capacities required and capital reduction benefits that could reasonably be anticipated by alumina refiners.

Experimental

Membrane tests were conducted in two phases: laboratory bench tests (2L start volume), and pilot scale (1kL start volume). Cross flow filtration over a membrane surface under pressure was used in all tests to effect the chemical separations, the aim being to concentrate magnesium and calcium by rejecting it to the retentate, while passing sodium chloride to the permeate. All tests were run using a sample of sea water collected from the Western Australian coast near Perth, and pre-filtered using 5 μm cartridge filters.

Two types of tests were conducted. Perturbation tests (P-tests) determined the relationship between rejection, pressure and flux for a given membrane and solution type, while recovery tests (R-tests) provided the relationship between operating pressure and flux as the volume recovery and feed analyte concentration increased. The results were used to:

i. Determine flux behavior over a range of operating pressures for a given solution matrix and assist in determining which membrane type is most suitable for a given application.

ii. At pilot scale, provide quantitative data that facilitates an engineering design process.

During R-testing, the retentate is recycled to the feed tank such that the concentration of rejected species increases, causing the osmotic pressure to increase toward a maximum value. A schematic diagram showing the layout of the equipment for both the laboratory and pilot scale tests is shown in Figure 1.

Laboratory scale tests were conducted using 21.5 cm² flat sheet membranes that represented three commercially available spiral wound NF membranes. The capacity of each membrane to concentrate Ca and Mg into the retentate at suitable NF operating pressures (<4000 kPa) and flux rates (>20 liters per square meter per hour - LMH) were compared and the best performing membrane was finally tested in the pilot scale program.
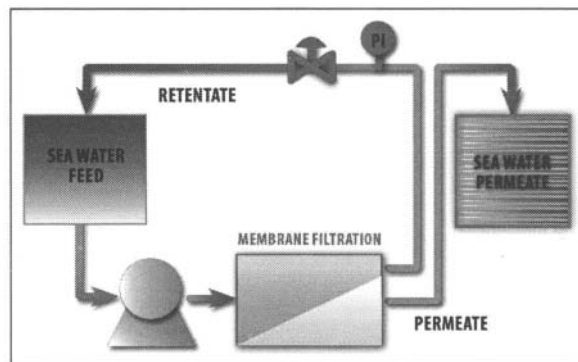

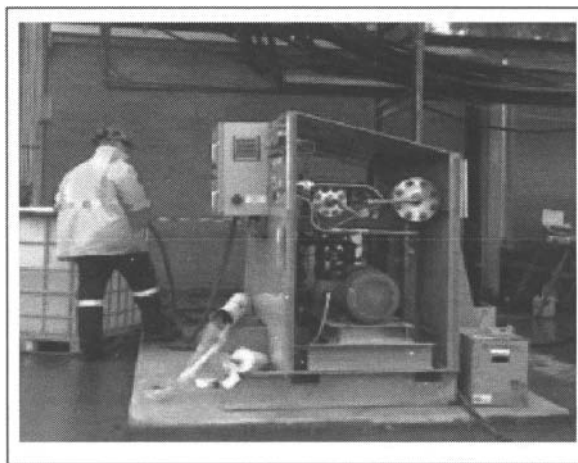**Figure 1: Schematic Diagram of Test Arrangement**

Figure 2: Pilot Test Rig

The first pilot scale test run was performed at a constant pressure of 3200 kPa. The flux was allowed to decline from an initial flux rate of 45 LMH as the osmotic pressure increased and conductivity, temperature and pH measurements were taken at increments of 10% recovery up to a maximum recovery level. The maximum recovery level was characterized by a significant rate of flux decline.

Feed and permeate samples were collected at various recovery points and analyzed for Ca, Mg and S by ICP-OES. The concentrated feed sample produced at the maximum recovery level of 80% during the first pilot run was then used for comparisons of neutralization characteristics with untreated sea water by titration of the free alkalinity of a generic Bayer residue water (refer Figure 3). Analytical results from this first pilot run were also used as the input to modeling of the neutralization using Basecon Technology (refer Table 4).

A repeat of the previous R-test was performed at a reduced starting pressure of between 1000 and 2400 kPa and then gradually increased to achieve a constant flux rate of 35 LMH. As recovery increased beyond 60%, samples were collected at recovery increments of 5%. P-tests were conducted concurrently during the test at a range of recovery levels to better determine the ideal recovery recommended for a full scale process.

Results and Discussions

Table 1 shows that, of the three membranes P-tested in the laboratory, the Dow DK membrane demonstrated superior magnesium rejection performance at relatively low operating pressures and it was selected for use in the pilot scale tests. The TS80 membrane achieved similar magnesium concentrations, however the flux decline was more significant and the applied pressures required were much higher. The TS50 achieved the lowest magnesium rejections.

Table 1 : Membrane Screening Results Summary

Membrane Type	Applied Pressure [kPa]	Initial Flux [LMH]	Final Flux [LMH]	Recovery achieved [%]	Mg Rejection [%]
TS80	2820	35	9.5	60	98
TS50	1700	35.4	22.5	75	92
DK	1425	35.4	18.6	78	97

The sea water neutralization reaction was simulated by titration of 250 ml of a generic Bayer residue water with both untreated sea water, and sea water concentrate (NF retentate). The aim of the titration experiments was to confirm that the increase in magnesium and calcium concentration did not change the reactions from those typically observed during sea water neutralization and the pH was allowed to equilibrate after each addition. Samples were also collected at the start, at the perceived inflection point (~11.5 pH for sea water and ~10.3 pH for NF concentrate) and at the end of the titration (~pH 9.4) and details of analytical data are shown in Table 2.

Table 2: Chemical Analysis of Titration Solutions

	Bicarbonate	Carbonate	Hydroxide	Total Alkalinity	Aluminium	Calcium	Magnesium
		[mg Na₂CO₃/l]			[mg Al/l]	[mg Ca/l]	[mg Mg/l]
Bayer Residue Water	<1	8200	46000	54200	3100	0.5	<0.1
Sea Water	-	-	-	-	0.34	410	1200
Residue & Sea Water at Inflection	<1	5300	160	5460	620	4	8.4
Residue & Sea Water at end	2800	480	<1	3280	2.4	110	220
Sea Water NF Concentrate	-	-	-	-	0.55	760	6600
Residue & Concentrate at Inflection	6700	4500	<1	11200	40	12	18
Residue & Concentrate at end	9000	720	<1	9720	0.71	240	1900

The titration curves (Figure 3) show similar symmetry with two inflection points when the added reagents are normalized to magnesium addition, suggesting that similar reactions are taking place.

The causticity in a Bayer residue water can exist as both free hydroxide (OH⁻) and as the aluminate ion (Al(OH)₄⁻). Further alkalinity is provided by the carbonate ion (CO₃²⁻). When this Bayer residue water is reacted with sea water, or sea water concentrate, there are a multitude of reactions that can take place. The initial reactions precipitate magnesium and calcium hydroxides (brucite and portlandite) thereby lowering the pH.

$$3Mg^{2+} + 6OH^- \rightarrow Mg_3(OH)_6 \qquad (1)$$

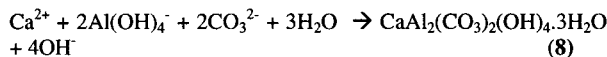$$Ca^{2+} + 2OH^- \rightarrow Ca(OH)_2 \qquad (2)$$

As the hydroxide concentration reduces, the aluminate ion precipitates, releasing hydroxide ions which then form more magnesium and calcium hydroxides and reactions with carbonate ions also start to occur.

$$Al(OH)_4^- \rightarrow Al(OH)_3 + OH^- \qquad (3)$$

$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3 \qquad (4)$$

$$Mg^{2+} + CO_3^{2-} \rightarrow MgCO_3 \qquad (5)$$

Once the hydroxide concentration has sufficiently reduced, the hydrotalcite-like compounds precipitate at suitable nucleation sites, typically on the previous precipitates. These compounds are layered double hydroxide compounds with the general formula $[M(II)]_{1-z}[M(III)]_z(OH)_2[A]z.mH_2O$ where M(II) and M(III) are divalent and trivalent metal ions and A is an anion e.g. CO_3^{2-}, SO_4^{2-}, Cl^-, OH^-. Typically hydrotalcite, hydrocalumite and para-aluminohydrocalcite can be produced as shown in the reactions below:

$$6Mg^{2+} + 2Al(OH)_4^- + 8OH^- + CO_3^{2-} + 4H_2O \rightarrow Mg_6Al_2CO_3(OH)_{16}.4H_2O \qquad (6)$$

$$2Ca^{2+} + Al(OH)_4^- + 3OH^- + 3H_2O \rightarrow Ca_2Al(OH)_7.3H_2O \qquad (7)$$

$$Ca^{2+} + 2Al(OH)_4^- + 2CO_3^{2-} + 3H_2O \rightarrow CaAl_2(CO_3)_2(OH)_4.3H_2O + 4OH^- \qquad (8)$$

Determination of the exact stoichiometry is impossible due to the number and complexity of the reactions taking place.

Titration of Bayer Residue Water :
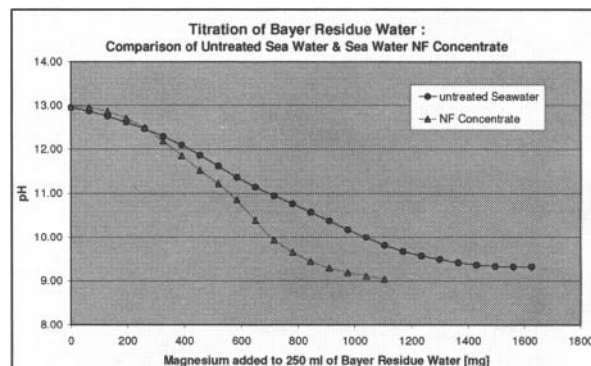Comparison of Untreated Sea Water & Sea Water NF Concentrate

Figure 3: Mass Mg Added vs pH for Concentrated Sea Water in Residue Water

Inspection of the neutralization curves (Figure 3) shows that there are two areas where a small inflection is observed: around pH 11.5 and pH 10.3. The first inflection point corresponds to the pKsp of magnesium hydroxide of 11.3. This is expected because magnesium is present at three times the concentration of calcium in sea water and in sea water NF concentrate. The second inflection corresponds to the carbonate/bicarbonate equilibrium as the hydrotalcite-type reactions (equations 6, 7 and 8) remove carbonate from solution. Analysis showed that while some hydroxide and aluminate was still present at the upper inflection point they had all been consumed by the lower inflection point. The alkalinity remaining at the lower inflection and the end point was due to the presence of carbonate and bicarbonate ions. These observations were similar, regardless whether sea water or sea water NF concentrate was used for neutralization.

Two points of difference between the sea water and sea water NF concentrate neutralization curves are evident: i) at the end point

where a lower pH was reached using sea water NF concentrate (9.0 cf 9.4 pH), and ii) that the endpoint was achieved much sooner. This is explained by the higher concentration of reactants in the sea water NF concentrate pushing the reaction equilibrium further to the right (equations 1 to 5), thus removing more hydroxide, carbonate and aluminate ions from solution thereby reducing the pH. There is also less dilution if the sea water NF concentrate is applied, which reduces the total amount of un-reacted calcium and magnesium in solution and hence improves the magnesium and calcium reaction efficiency.

Although a faster reaction with concentrate was observed, in good agreement with Virotec's experience, this was not investigated further. Alumina refinery residues have different properties and reaction kinetics need to be specifically evaluated with on-site pilot tests which will determine actual reaction rates and equipment sizing.

The results for the first pilot scale test are shown in Figure 4. In addition to observing the deportment of magnesium in this test, the behavior of calcium was also considered of high importance as the propensity of calcium sulfate to form a scale on the surface of the membrane will lead to rapid flux decline. A highly concentrated layer of calcium and sulfate ions at the membrane surface can form as a function of increasing bulk concentration of each ion in the feed stream and as a function of flux. The formation of this layer is called concentration polarization and was identified in the tests by a sharp decline in flux for a given pressure (Figure 4). The determination of the conditions at which this significant flux reduction occurs is of considerable importance in determining the achievable recovery level.
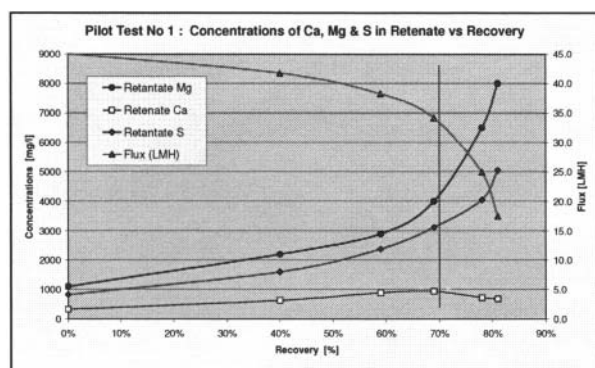

Figure 4: Pilot Test 1 - Analytical Trends

Figure 4 also shows the concentration of each analyte in the feed at each recovery level and the flux at each recovery level. The rejection of chemical species was very high and resembled those achieved in the screening tests recorded in Table 1. At 80% recovery the magnesium rejection was 95% and the sulfur rejection was up to 100% (the limit of detection for these analyses was 100 mg/L). The calcium concentration at 80% recovery was less than that at 70% recovery. This implied that some calcium precipitation occurred at about the 70% recovery level. It can also be seen that the decrease in calcium concentration coincides with a significant increase in the rate of decline in flux and this supports the hypothesis that concentration polarization of calcium sulfate was occurring. As a result recovery levels were reducing because of the resultant rapid rate of flux decline at the applied pressure of 3200 kPa.

The suitability of using sea water NF concentrate with Virotec's Basecon Technology was also considered. Untreated sea water and NF concentrate were compared as the feedstocks for neutralization of three different alumina refinery red mud residues in different parts of the world. The sources were:

i)   a refinery in Australia, processing bauxite from North Queensland using the Bayer Process;

ii) a refinery in North America, processing bauxite from Jamaica using the Bayer Process; and

iii) a refinery in China, processing diasporic bauxite from China using the Lime Sinter Process.

The chemical composition of neutralizing streams used in the kinetic model was based on the world average sea water, and the chemical composition of NF concentrate at 70% recovery as analyzed in the first pilot trial. This was the point at which scaling became apparent and therefore the maximum condition for operation. Details of the chemical composition of the two neutralization reagents are shown in Table 3.

Table 3: Chemical Analysis of Reagents used for Basecon Technology Computer Model of Sea Water Neutralization Kinetics

Chemical Analysis	Sea Water	NF Concentrate
Ca^{2+} [mg/l]	412	940
Mg^{2+} [mg/l]	1290	4000
SO_4^{2-} [mg/l]	910	9400
K^+ [mg/l]	400	645
Na^+ [mg/l]	10400	13000
Cl^- [mg/l]	19400	27100
pH	8.3	8.0

The Basecon Technology neutralization kinetics for each of these three refinery residues was modeled using Virotec's computer model. Table 4 presents the pre- and post-neutralization properties and data of the three alumina refinery residues investigated.

Table 4: Basecon Technology Kinetic Model Outputs for Three Different Refinery Residues

Refinery Bauxite Source Process	North America Jamaica Bayer	Australia Nth Qld (Aus) Bayer	China Central China Lime Sinter
Properties : pH of residue before neutralization	12	12.4	11.6
pH of residue after neutralization	9.0-9.5	9.0-9.5	9.0-9.5
Total alkalinity of residue before neutralization [mg Na₂CO₃/L]	19,000	11,370	44,000
Total alkalinity of residue after neutralization [mg Na2CO3/L]	<500	<500	<500
Treatment ratio of Seawater to refinery residue [kL/kL]	5 : 1	3 : 1	10 : 1
Treatment ratio of NF Conc. to refinery residue [kL/kL]	2 : 1	0.9 : 1	3 : 1
Ratio of Sea Water to NF Concentrate	2.5	3.3	3.3

The model supported pilot testing results showing the end point is reached sooner and the volume of neutralizing reagents can be reduced approximately three fold and hence there is significant potential to reduce pumping, piping and size of major equipment if an NF concentrate is applied.

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The impact of concentration polarization can potentially be reduced by lowering the operating pressure and therefore flux rate, leading to greater recovery levels. This hypothesis was assessed in the second pilot scale test.

The applied pressure was varied as required to maintain the flux at a lower constant target flux rate of 35 LMH. The test incorporated a number of P-tests at various recovery points. The operational data and the species transmissions are summarized in Figure 5, and the relationship between flux, pressure and recovery is shown in Figure 6. By comparing with Figure 5 it can be seen that controlling flux and minimizing concentration polarization improves permeate recovery, and therefore calcium and magnesium concentration. It is apparent in Figure 6 that the pressure / flux curve at 75% recovery is almost identical to those at lower recoveries, and the onset of scaling has been delayed by reducing flux.
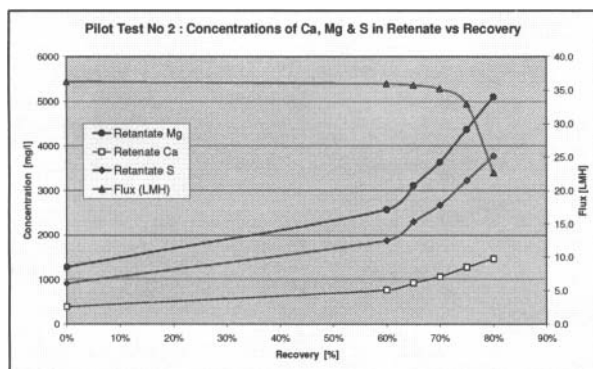

Figure 5: Pilot Test 2 Analytical Trends

Predicting the point at which calcium sulfate nucleation occurs is very important in managing a sea water NF process. Precipitation of calcium sulfate is suppressed by the total ionic strength of sea water (in excess of 45 g/L TDS). Residence time in a membrane element is also short (<0.2 sec). As a result, the conditions under which precipitation occurs cannot be readily determined from standard thermodynamic data. The point at which calcium sulfate precipitation occurs on the membrane surface was therefore determined empirically based on the observation of flux versus pressure relationships.

Figure 6 shows a linear relationship between flux and pressure at low flux rates. A breakpoint is observed at a flux rate of about 45 LMH, the critical flux. This is the point where the membrane was operated in pilot test 1, explaining why the recovery results were poor. Poor membrane performance in pilot test 2 also occurred around 75% recovery, evidenced by the sideways shift in the 80% and 85% flux / pressure curves as shown in Figure 6

For each recovery level up to 75% the relationship between applied pressure and flux was very similar and the applied pressure to achieve these recoveries was approximately 2200 kPa. Increasing the pressure beyond 2200 kPa caused a significant rate of flux decline suggesting that resistance at the membrane surface increased significantly with the increase in applied pressure causing a concentration polarization effect. The maximum achievable recovery, while maintaining a flux of 35 LMH, appears to be 75%. Beyond this point, the pressures required to achieve an equivalent flux increased significantly. The transmission of calcium at 75% recovery was 8.3%, however this

reduced to 7.8% at 80% recovery. This is another indication of calcium precipitation on the membrane surface and in the feed. At 85% recovery it was observed that precipitation was occurring in the feed vessel as well.

For these reasons 75% recovery was chosen as the practical concentration limit for a sea water NF plant.
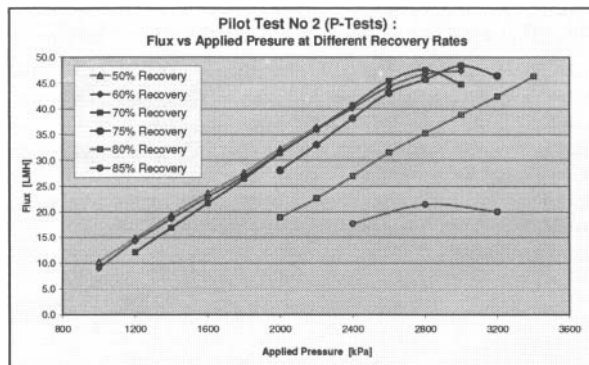

Figure 6: The Relationship between Pressure and Flux at a Range of Recoveries

Anti-scalents are commercially available to reduce the rate of nucleation and these products achieve an induction period during which scaling can be avoided. However, they do not indefinitely prevent the precipitation of minerals such as calcium sulfate under supersaturation conditions and the application of anti-scalents was therefore not considered in this investigation.

Another advantage of sea water NF is the fact that the NF permeate is largely free of the scaling species and it is therefore an ideal feed to a sea water RO (SWRO) plant producing potable water.

Simulations were performed using Dow's Reverse Osmosis System Analysis (ROSA) simulation software to compare untreated sea water feed with the NF permeate feed. The results are summarized below in Table 5.

Table 5: Comparison of SWRO and Sea Water NF and RO

	Sea Water RO	Sea Water NF & RO
Feed Flow Basis	1000 m³/hr	1000 m³/hr
NF Permeate Recovery	N/A	75%
RO Plant Feed	1000 m³/hr	750 m³/hr
RO Permeate Recovery	48%	64%
Potable Water Production	480 m³/hr	480 m³/hr

The precipitation of calcium sulphate is performance limiting in both traditional SWRO and multi-effect distillation (MED) plants. When using sea water NF permeate as a feed to the RO unit, the limiting factor is instead osmotic pressure and the results of this comparison indicate that permeate recovery will increase from 48% to 64%. Hence, a significant reduction in the RO unit capacity can be achieved for equivalent potable water production,

reducing capital and operating costs and significantly reducing waste retentate discharge from the process.

Meeting fresh and potable water requirements will continue to be a global challenge for alumina refineries. For those refineries in proximity to the sea and with environmental approval to access sea water, the opportunity to combine sea water NF and RO for potable water and neutralization of refinery red mud solid residue using NF concentrate (retentate) offers great benefits.

The capital and operating costs for a sea water NF & RO plant will be higher than for an SWRO plant due to the extra membrane installations and pumping costs respectively. However the extra costs for the sea water NF plant need to be compared to the significant cost reductions from the smaller neutralization equipment required, where the volumes of NF concentrate added are about three times lower than for traditional sea water neutralization (refer Table 4). Pumps, piping, neutralization tanks and settler size are significantly reduced for an equivalent amount of neutralized red mud and these changes can be seen in the process schematic below in Figure 7
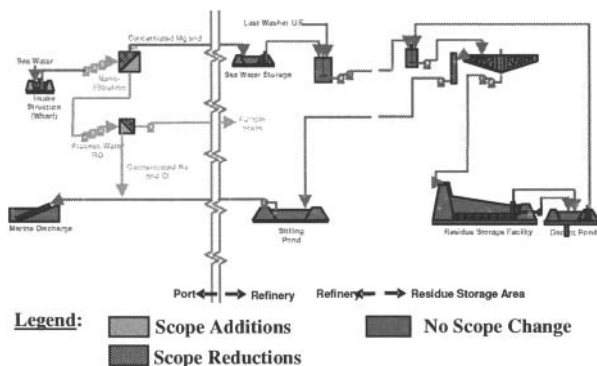

Legend: ▢ Scope Additions   ▪ No Scope Change
         ▪ Scope Reductions

Figure 7: Schematic Showing Sea Water NF and RO Process Impact on Bayer Residue Treatment

The cost comparisons are dependent on the particular situation but order of magnitude costing estimates have shown that using a sea water NF concentrate to neutralize alumina refinery residue has significant economic potential in situations where:

- An existing refinery using sea water neutralization is looking to expand without spending capital on neutralization equipment and especially if there is limited land area available for decantation and drying ponds.

- The refinery is not adjacent to the sea but still wants to neutralize its residue with a sea water based reagent.

- The refinery wants to reduce salinity of neutralized residue to improve options for re-use, and

- There is a local sea water desalination plant (e.g. SWRO, MED), where the combined benefits of nanofiltration to reduce scaling of desalination equipment and provide a sea water NF concentrate for refinery residue neutralization can be achieved

Conclusions

Nanofiltration of sea water has been shown to be a viable method to significantly concentrate calcium and magnesium while sodium

and chloride concentrations are maintained close to the original concentrations of sea water.

This NF concentrate improves the reactivity and kinetics of sea water neutralization of alumina refinery residue and the required volumes of NF concentrate are about three times lower than for neutralization with untreated sea water. The size of neutralization equipment like pumps, piping, neutralization tanks, settlers and decantation and drying ponds are significantly reduced, leading to reduced capital costs and footprint for new operations or enabling expansion of capacity for existing alumina refineries using sea water neutralization.

The low salinity of neutralized residue improves options for re-use in agriculture and soil beneficiation

The nanofiltration permeate is also a valuable and improved feed to sea water reverse osmosis (SWRO) and multi-effect distillation (MED) plants. With most of the scaling species removed by the NF membrane, RO process efficiency is much improved, leading to a significant reduction in the RO unit capacity for equivalent potable water production, reducing capital and operating costs and significantly reducing waste retentate discharge from the process.

Alumina refinery residues have different properties and reaction kinetics need to be specifically evaluated with on-site pilot tests which will determine actual reaction rates and equipment sizing.

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