IMPLEMENTING SEAWATER NEUTRALISATION AT GOVE

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Abstract

The compaction of stored bauxite residue from the Bayer process generates an alkaline supernatant liquor (SNL), the composition of which is refinery specific and dependant on both process and climatic factors. The remediation and disposal of this liquor is often necessary to maintain refinery water balance. This project was initiated to determine the optimum conditions required for converting Rio Tinto Alcan (RTA) Gove SNL to an environmentally acceptable discharge using seawater neutralisation.

The use of seawater to "neutralise" Bayer process streams and render them environmentally suitable for discharge is a well-established practice. RTA sought to improve this from a coarsely controlled "end-of–pipe" process, reliant on large storage areas and high dilution rates, to a finely controlled, dedicated system fully integrated within the refinery flowsheet.

The study from initial concept to final engineering design involved a multidisciplinary team from both Australia and Canada. The project incorporated extensive laboratory studies on process neutralisation chemistry as well as specialised solid / liquid separation to achieve the appropriate engineering design.

This study extended the current knowledge of seawater neutralisation by incorporating a red mud stream. This imparted greater control over the neutralisation chemistry, and also ensured the residual precipitate was easily compressed into a paste suitable for dry stacking.

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1. Introduction

The use of seawater to neutralise the waste water from the Bayer industry has been known for many years (Goldfard; 1984). It has not been widely implemented, however, with waste water storage the preferred option. But, as more stringent environmental regulations have been introduced and available land for waste storage has decreased, a more cost effective and permanent solution to these waste problems is required.

Seawater neutralisation is a suitable option because the impurities within the liquor stream are chemically converted to an inert form and removed from solution, not just diluted as in some waste water treatments.

Knowledge of the chemistry behind seawater neutralisation is critical in understanding the applicability of this process. The term "neutralisation" relates to a series of chemical reactions which occur when seawater is combined with dilute Bayer liquor. The reactions begin with the precipitation of a range of species such as calcium carbonate ($CaCO_3$), calcium hydroxyaluminates ($Ca_2Al(OH)_7$) and other alkali-earth metal hydroxides. As the pH drops, the formation of hydrotalcite-like species becomes favoured (Hanahan; 2004). These are probably the most important precipitates in the reaction, and consist of Layered Double Hydroxides (LDH) of general formula ($[M_{(1-x)}Al_x(OH)_2]X.nH_2O)_n$. In the case of the divalent metal M being magnesium, the compounds are mostly termed hydrotalcites, or hydrocalumite if calcium is present (Smith; 2005). Often, both are present simultaneously. Hydrotalcite promotes agglomeration of other particles in solution, and removes various cations and anions. Under appropriate conditions, hydrotalcite precipitate can aid in the removal of toxic heavy metals and anions (vanadates, molybdates and arsenates) via a combination of adsorption, absorption and intercalation. The extent to which these anions are incorporated into the hydrotalcite matrix depends on the pH of the system. At higher pH levels (up to 11.5), an abundance of carbonate and its favoured intercalation precludes most

other anionic species from being incorporated. This reaction yields species with the typical formula $Mg_4Al_2(CO_3)(OH)_{12}.H_2O$. At lower pH levels (around 8.5), the concentration of available carbonate drops due to a shift in the carbonate/bicarbonate equilibrium, allowing incorporation of other anions. In this case, the predominant formula becomes $Mg_8Al_2(CO_3)_{0.5}X(OH)_{20}.H_2O$, where X represents anionic species other than carbonate. Larger organic impurities can also be removed under some circumstances mostly by adsorption.

However, for these reactions to proceed, the correct stoichiometry of ions must be present. This requires a high degree of control exerted over the process.

Once the chemical considerations have been addressed, there is also a need to optimise solid-liquid separation. There are a number of ways to achieve this (see Section 2.1), however it appears the most efficient is addition of red mud to the process. This improves the settling of the colloidal, gelatinous clay-like precipitates, and improves adsorption of heavy metal species. The overall result is a decrease in effluent pH, a dramatic drop in the dissolved ion concentration and consumption of potentially polluting waste water. However, there are several complex interactions of process variables specific to each refinery which are critical to neutralisation efficiency, and these must be addressed before seawater neutralisation can be utilised effectively. This paper relates the optimisation of these variables for conditions specific to the future Gove flow sheet, and the local environment.

2. Background

2.1 Previous Investigations

The use of seawater neutralisation at Gove was first proposed as an effective means of reducing the inventory of SNL stored in the red mud dams. A 1996 study by CSIRO (Vaughan; 1996) investigated several parameters including:

- geochemical behaviours of heavy metals
- bench-scale tests of SNL neutralisation

- effluent toxicity testing on various trophic levels (algae, prawn larvae and barramundi)
- risk assessment of ecological impacts of the process.

Analysis of the neutralised effluent showed there was a sharp decrease in the concentration of several important species (B, Ca, Mg, Mn and Sr). Low levels of arsenic and selenium were detected that were higher than the background levels, but were below the ANZECC water quality guideline limits at the time. Mo and V were also detected, but these had no specific limits in the ANZECC guidelines at that time (NHRMC; 2000). Molybdenum (as molybdate) has a low toxicity and its concentration in the effluent was far below levels considered toxic. Vanadium, however, was of more concern. Its concentration was higher (8mg/L) and was considered it would be toxic to some marine species. As a result, a specific target of 100μg/L for vanadium in any future effluent was stipulated. This was a key conclusion, and made a requirement for all subsequent studies.

Analysis of residual solids provided some indication of what was occurring during the neutralisation reaction. The main components of the solid consisted of hydrotalcite ($[Mg_6Al_2CO_3(OH)_{16}].4H_2O$), calcium oxalate ($CaC_2O_4$) and calcium carbonate ($CaCO_3$), plus trace amounts of other salts, confirming the predicted chemistry.

The settling rates after neutralisation were also studied. More efficient settling occurred with the higher levels of seawater, but overall, these results were quite poor.

Ecotoxicological tests on the effluent were also conducted. This involved exposing tiger prawns, barramundi juveniles and marine algae (*Nitzschia closterium*) to varying dilutions of the effluent. Sensitivity between the different organisms varied. The algae and tiger prawn larvae showed some sensitivity to the effluent in high concentrations, but barramundi were largely unaffected. A statistical analysis of the data across all tests concluded that if the 5:1 effluent is diluted approximately 200 times after discharge, no adverse effects (chronic or acute) would be expected on the receiving environment.

This CSIRO study demonstrated that seawater neutralisation could be viable for SNL treatment, but optimised environmental and process control parameters would be required. This led to implementation of the current system to neutralise SNL at Gove (Figure 1).
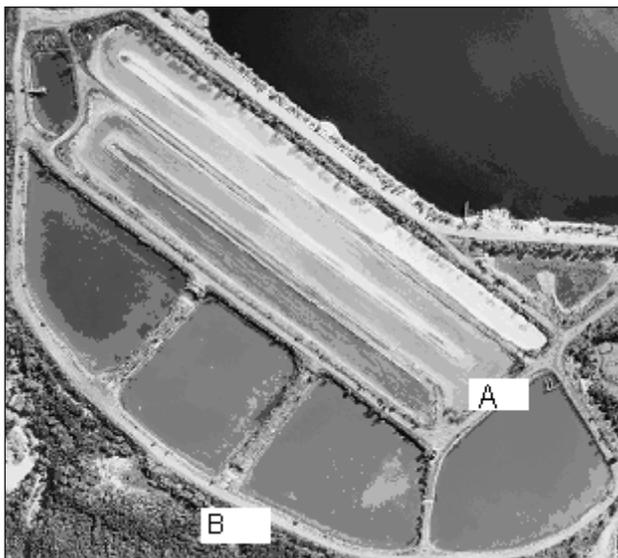

Figure 1. Gove labyrinth (A) and decant ponds (B)

Approximately 400m³/hr of seawater was combined with 30m³/hr of SNL in the open settlement channel (labyrinth), A in Figure

1. The neutralisation reaction occurred as the combined effluent flowed along the channel, precipitating hydrotalcite. Removal of contaminants from the SNL did occur, and effluent clarity was good, however operational and quality control problems were identified (ANC, dredging requirements). Also, the capacity of this facility would not be adequate for future SNL volumes once refinery expansions were completed. Therefore, a series of experiments specific to Gove were performed to improve this installation (Fawell; 2000).

At this point it was decided to evaluate the addition of Red Mud Slurry (RMS) to the process. It was anticipated this would aid co-precipitation, increase mud densities, improve effluent clarity and also increase the removal of particulate or dissolved heavy metal species via adsorption. The mud was added during neutralisation, and settling rates improved immediately, with settling times greatly reduced. The effectiveness of synthetic flocculants also increased. Numerous flocculant types were investigated over a range of dosages, to determine the best product. However the efficiency of the heavy metal ions removal from solution was not measured, so the effect of flocculation on this aspect could not be identified. It was evident from these tests that the labyrinth, under current operating conditions was not suitable for full seawater neutralisation if both SNL and red mud were to be used.

Therefore, the commissioning of a pilot trial was initiated to evaluate the process under operational conditions at Gove (ie using SNL and red mud). The primary tasks of this trial were to achieve full SNL neutralisation, minimise the quantities of red mud and seawater required, and to produce an effluent suitable for discharge. This focused on the removal of V, As, Mo, Al, Mg and Ca, and some process control optimisation (Haggard; 2002).

The reagent ratios were based on previous testwork. Primary neutralisation was performed in an agitated tank, with the contents then pumped to a deep cone washer, flocculated and clarified. The overflow was diverted to a containment pond, while the underflow reported to a trial stacking area.

An important operational aspect of this trial was the use of evaporation seawater (ESW) from the evaporation train at Gove, rather than fresh seawater. This would reduce total costs associated with pipe work and seawater pumping, utilise a current discharge stream while also neutralising the small amount of impurities associated with ESW. ESW has slightly elevated pH, alkalinity, and TSS relative to fresh seawater. The other significant difference is temperature. ESW exits evaporation at around 50°C, so this would increase kinetics of neutralisation reactions. It was determined that this stream would be suitable and all subsequent trials and test work used this stream as the seawater source.

A thorough investigation of the chemistry was also attempted by analysis of the effluent and the solid residue. These results mirrored those of previous reports, which strongly indicated that intercalation of vanadate and other oxyanions into hydrotalcite is responsible for the majority of vanadium removal. However the results also indicated that formation of other calcium species does contribute to vanadium removal.

The need for a reliable, rapid method to measure the neutralisation efficiency of the process was also identified. Measuring the molar ratio of Mg in feed to Al in liquor, in conjunction with measuring the ratio of seawater to soda in the feed, provided this. By maintaining the Mg/Al ratio between 3 and 5, and keeping the SW/Na_2O ratio between 285 and 315, adequate removal of vanadium and other impurities was ensured. The addition of red mud slurry was also necessary to ensure sufficient dissolved aluminium was present for hydrotalcite formation, and to improve solid/liquid separation.

Another important process identified was the phenomenon of "alkaline reversion". This occurred if effluent remained in agitated contact with red mud, to cause a steady increase in pH and dissolved metal concentration in the neutralised effluent. The exact cause and nature of this effect was not fully understood, but it would have a significant impact on the overall process if left unattended.

The treatment of only RMS with seawater in the absence of additional SNL liquor was therefore examined (Smith; 2002). This assessed the feasibility of residue treatment directly from the refinery prior to disposal to a storage area. The optimum ratios of red mud to seawater needed to be established to give the most efficient neutralisation. In this case, the main variable monitored was pH, and how this variable could be used to control neutralisation efficiency. The pH decreased to around 9 after neutralisation, however rose again almost immediately upon settling. After analysing the solids and liquor, it was found neutralisation efficiency was poor. Evidence suggested there was insufficient magnesium or carbonate present to efficiently neutralise all the impurities to hydrotalcite and remove the reactive alkaline solids simultaneously. As a result, these reactive alkaline solids (mainly TCA and hydrocalumites) dissolved back into solution under the diluted conditions:

$$Ca_3Al_2(OH)_{12} \longleftrightarrow 3Ca^{2+} + 2Al(OH)_4^- + 4OH^-$$
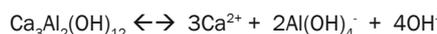
This was observed regardless if excess seawater was used. The rate of neutralisation was also slow, and an increase in carbonate and aluminium during neutralisation would be required to increase the formation of hydrotalcite and improve the neutralisation process. This confirmed that both red mud and SNL are required in any seawater neutralisation flow sheet to ensure optimum neutralisation occurred.

2.2 Current Considerations

The current study addresses the need for improved SNL treatment at Gove. This will ensure that red mud storage capacity will be sufficient for the increased output after the Gove expansion is complete. The main topic nominated for improvement was increased SWN capacity beyond the present labyrinth operation. This additional capacity is to be fed by outfall from the Evaporation Seawater (ESW) process stream, SNL and Red Mud Slurry (RMS) from Deep Washer underflow. The proposed flow sheet combines these streams in a dedicated reactor, followed by solid/liquid separation in a gravity thickener aided by synthetic flocculant. The overflow of this thickener will be discharged to the environment, while the underflow reports to the RDA for storage. This study aimed to optimise this process further to previous pilot trials, and develop adequate process control parameters.

The use of the correct ratio of SNL, red mud and seawater to achieve the optimum neutralisation conditions was the most important aspect. The main difference between this study and those completed previously was that the mud used was residue from a post expansion Gove flowsheet, with a higher % solids and lower soluble Al and Na concentrations. This results from installation of RTA Deep Cone Washers to replace the previous flat bottom washers. Therefore, the ratios of red mud, SNL and seawater were significantly different to previous studies. This may affect the settling and stacking properties of the neutralised mud, so this was also investigated.

Treatment of the residual liquor from the neutralised mud was also investigated. This saline SNL (SSNL) will collect after settling of neutralised red mud, and will be elevated in dissolved metal ions and other impurities.

Environmental considerations were another important aspect to the project. In order to acquire discharge licenses from

government regulators, it must be established that all aspects of environmental responsibility have been followed by Gove (Parry; 2006). This was addressed in the study.

3. Experimental

3.1 Establishing Primary Neutralisation Parameters

Red Mud Slurry (RMS), SNL and Evaporation Seawater (ESW) were collected within 48 hours of tests being performed. Na and Al values for all liquors were obtained by AAS, while ICPMS studies gave a Ca concentration of below 5ppm. Mg and Ca concentrations of seawater were published world averages (Table 1) (Handbook of Chemistry and Physics; CRC Press). The red mud was lab digested to predicted Gove Double Digest specifications. For batch neutralisations, the relative RMS volume was fixed at 0.6 while SNL was set to 0, 0.5, 0.8, 1 or 1.5. The required ESW volume for each SNL volume was then iterated to achieve a corresponding Mg/Al ratio of 2.0, 3.0, 3.6, 4.5 and 5.5.

Table 1. Component Concentrations

Samp	AAS Analysis (ppm)			World Average (ppm)		
	Na	Na₂O	Al	Al₂O₃	Mg	Ca
SNL	13335	17980	320	604		
RML	6910	9320	2689	5080		
ESW					1200	400

The neutralisation tests combined together RMS, SNL and ESW in a 1L bottle with agitation, while being maintained at 55°C in a water bath. After 3 minutes, the sample was centrifuged and then filtered through 0.45μm syringe filters. All samples were sent to Charles Darwin University (CDU) or the National Measurement Institute (NMI) for dissolved metals and inorganic analysis. A portion of unfiltered decant was analysed for suspended solids. All sample bottles were prepared in accordance with the Australian standards for relevant analyses.

3.2 Flocculation Testing

Three techniques were used to characterize the settling and compaction of the mud (Method 1234):

1. Jar tests - preliminary evaluation of the settling properties and screening of flocculants.
2. Dynamic settling analysis (C-FLOC™) which determines volumetric throughput (settling rate), the mass throughput and the flocculant dosage under different conditions.
3. Measurement of the visco-elastic properties of compacted mud samples by rotational viscometry.

Jar tests involved immersing a one-litre graduated cylinder in a controlled temperature bath set to 50°C, which simulated thickener conditions. Once cylinder contents were homogenized, the flocculant was added and dispersed with a plunger. A rake was used to improve the mud compaction. The settling rate, overflow clarity and compacted solids concentration were determined according to the standard procedure. The dynamic simulation of the mud decantation was accomplished using the RTA Settling Analyzer, C-FLOC™. Chemical analysis was also performed on the residual effluent.

The yield stress of the thickened slurry was determined using a Haake VT550 rotational viscometer equipped with the vane Sensor Rotor FL100 (star shape sensor).

3.3 Ecotox and Continuous-flow Optimisation

Effluent for ecotoxicology testing was produced from batch tests, but the reaction was dosed with flocculant to facilitate solid/liquid separation rather than centrifugation. Samples were sent to Charles Darwin University for metals analysis (ICPMS). The CSIRO Centre for Environmental Contaminants Research, Lucas Heights, performed the ecotoxicity work. (Adams; 2006).

The continuous-flow test work examined the impact of residence time and agitator speed on the neutralisation performance under continuous operation conditions. All experiments were conducted at RTA's QRDC laboratory using a bench scale continuous flow reactor system. Figure 2 shows a schematic of the test rig.
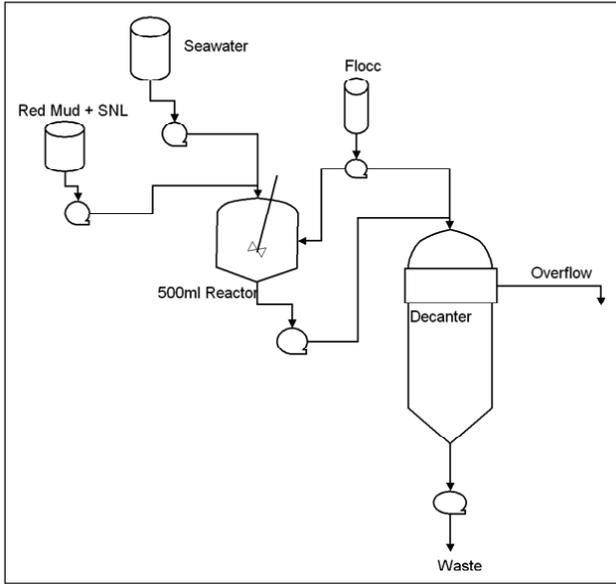

Figure 2. Continuous SNL neutralisation schematic

A series of batch tests were also performed to further explore the influence of red mud on the neutralisation chemistry. These were conducted by comparing the reaction of only SNL and seawater with analogous tests containing SNL, seawater and red mud.

4. Results and Discussion

Primary Neutralisation

For the neutralisation process to be economically and environmentally viable, it was critical that the pH of the effluent be reduced below 8.9, the total alkalinity kept below 50mg/L,

and dissolved metal concentrations of Al and V be kept below 700ppb and 100ppb respectively. Results from neutralisation tests encompassing the whole spectrum of variables can be seen in Figure 3. Samples with a final Mg/Al ratio greater than 3.6 had a noticeably lighter colour. The mud in these samples also displayed better immediate settling rates.

From this data, the optimum neutralisation mixture was designated as the 845 ratio. With this mixture, the reaction contained:

- Volume ratios of SNL = 0.8, ESW = 5.89, RMS = 0.6;
- Molar ratios of Mg/Al = 4.0, (Mg + Ca)/Na = 0.52;
- pH = 8.9, [Al] = 686ppb, [V] = 97ppb.

These results complied with stipulated requirements. However these volume ratios required that the SNL and mud feeds produce a (Mg/Al) molar ratio of 4, and the ratio of (Mg+Ca)/Na be 0.52. Any process variations to soda or alumina concentrations would require relative volume ratios to be recalculated.

Kinetic testing was also performed to ascertain the rate of reversion. A steady increase in pH demonstrated that reversion had begun after 30 minutes agitation time. This was also accompanied by a rise in dissolved metal concentrations. Therefore, efforts must be made to separate the mud and effluent as quickly as possible after neutralisation. Interestingly, the total alkalinity of the samples decreased over the same time period. If the increase in dissolved metal concentration was in fact due to re-dissolution of the precipitated LDH materials, then alkalinity would be predicted to rise upon reversion, not fall. This suggests that the release of dissolved metals back into the water column can probably be attributed to more than one mechanism.

The presence of red mud in the neutralisation process has been shown to have a significant impact on the process. It is currently added primarily as a settling aid to assist in the removal of poor settling neutralisation products such as hydrotalcite. However, its full influence on chemical processes has yet to be established, so a series of batch tests were undertaken to explore this. Table
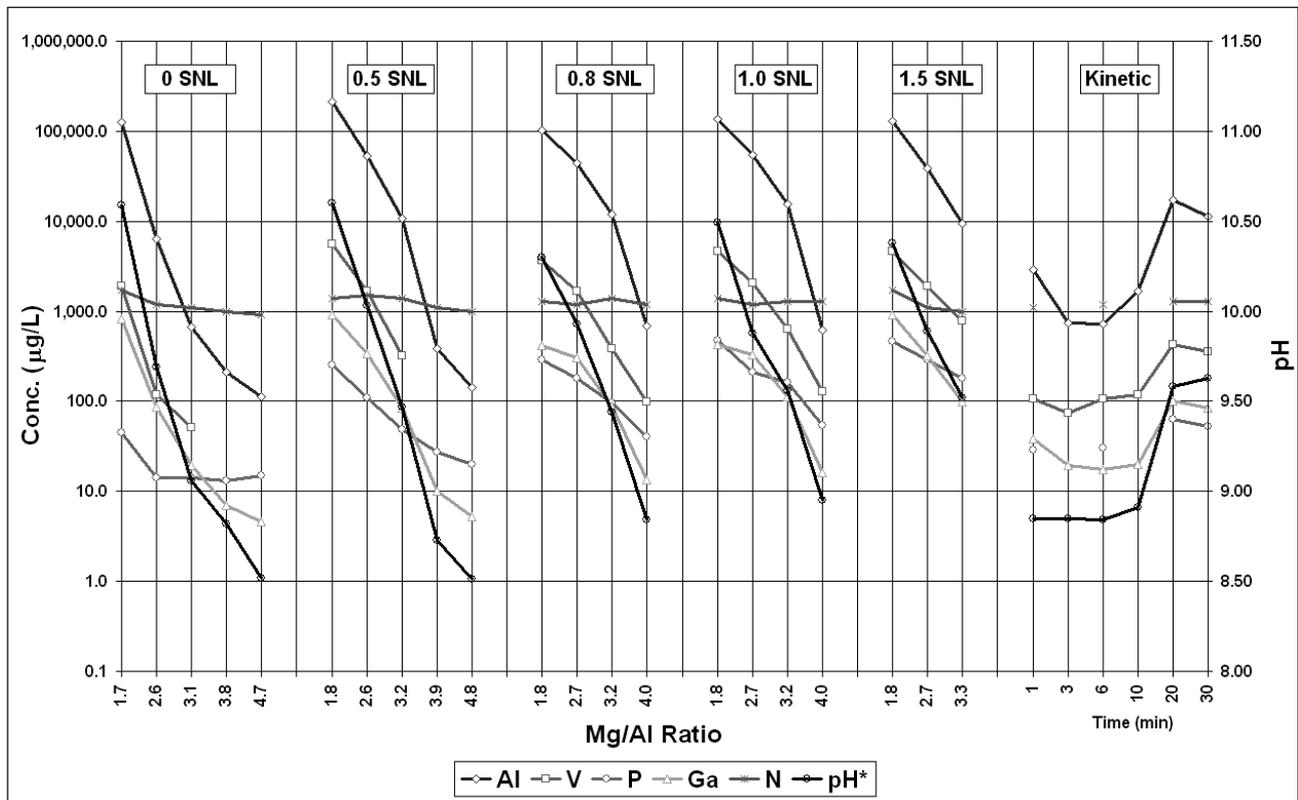

Figure 3. pH and dissolved metal concentrations of neutralisation data with various Mg/Al ratios

2 displays the comparison of the standard sequence test (SNL, seawater and RMS combined at time zero) with and without mud. Direct effects on the effluent included:

- Increased pH;
- Increased alkalinity;
- Significant reduction in soluble Mg, Ca and V concentrations;
- An increase in some soluble metal concentrations, eg Al, Mo & U.

The neutralisation process can not achieve the intended target vanadium concentration of 100ppb using the 845 mixture without red mud. However the increase in pH due to the presence of mud may limit the quantity of SNL that can be processed. A compromise between these two variables is the simplest scenario.

Table 2. Effect of Mud Presence on Neutralisation

	2 min		3min		4min		Ave % change
Mud present	X	✓	X	✓	X	✓	
pH	8.22	8.42	8.36	8.72	8.42	8.47	2.4%
Alkalinity	560	801	581	496	581	781	20.6%
Cond (mS)	57	58	57	46	57	58	-5.3%

Analysis of the saline SNL (SSNL) produced from settling of the neutralised mud showed an increase in dissolved metals concentration and pH (maximum pH 10.9). Reprocessing of this stream through the primary neutralisation process was therefore the only way to ensure discharge compliance.

Flocculation Testing and Rheology

Settling rate was the parameter used to determine the ability of a flocculant to settle the neutralized mud. The target 845 mix was chosen to screen the flocculant with 3 minutes of reaction time at normal agitation intensity. Clarities were better than 20mg/L a reduction in the feed solids concentration slightly reduced the flocculant dosage required. It was determined that settling rate was dependent on the anionicity of the flocculant.

Double dosing of the flocculant to the reactor and the clarifier improved the overflow clarity. An increase in the SNL proportion in the mixture significantly reduced the settling rate and compaction of the mud, while overflow clarity was not affected. Therefore, optimising mud addition is essential for effluent settling.

A significant reduction in the concentration of chemical impurities was also seen after neutralisation. Nominal V concentrations were in the range 55 to 351µg/L for a 845 neutralization condition.

The rheology of the neutralised mud showed a marked increase in yield stress above a threshold concentration of solids (41% solids) which is typical for suspension of fine material like red mud. This paste-like mud would be suitable for stacking in the Gove "mud farming" system.

Ecotox Analysis

Toxicity testing was conducted by CSIRO on the filtered neutralised wastewater samples using the marine diatom, *Nitzschia closterium*. This species is found in northern Australia coastal environments and has been isolated from Darwin Harbour. A review of the science of ecotoxicology for marine environments performed under the RTA Gove Marine Health Monitoring Program (MHMP) concluded that "only one toxicity test could be considered to have relevance and be of the appropriate type of test to be of use to Rio Tinto Alcan Gove". This test is the 72 hour growth inhibition test using the marine diatom, "*Nitzschia closterium*".

The results showed all three samples reduced growth rates of the diatom when undiluted. Mixture 845 showed the greatest effect, followed by mixture 555, then mixture 036 (see Figure 3, "0.5 SNL" and "0 SNL" respectively). Nevertheless, the toxicity tests showed the specified neutralisation mixtures would cause no negative effects to microalgae outside current mixing zones and there should be no measurable toxicity within 100m of the discharge point. RTA Gove has made a commitment that the size of this mixing zone will decrease over time.

Optimum neutralisation efficiency and maximum SNL consumption was achieved with the 845 mixture, but there was little difference between the 555 and 845 mixtures in terms of dissolved metals concentration or ecotoxicity.

The neutralised effluent also showed that while total loads of dissolved and particulate metal loads may increase slightly (Cu, Mo and Ga in particular), metal concentrations beyond 100m of the outfall would be well below levels expected to have any adverse biological impact, based on bioavailability studies.

Continuous-flow Optimisation

The residence time and agitation of a continuous-flow reactor will have a strong impact on neutralisation efficiency. The agitation must be sufficient to fully mix the reagents, but not so aggressive that shear causes flocculated particles to re-suspend. These two variables will also dictate the extent to which reversion can occur during the initial process. These effects were monitored by measuring pH and metal ion concentration while varying residence time and agitation speed. The results suggested there was no effect on the reaction, with all four residence times and agitation speeds approximately equal (Table 4). There was also minimal variation for alkalinity, total phosphorus or dissolved metals (Al, V, Cu and Ga) concentrations, which all remained below specified limits.

Table 3. Results for pH and alkalinity with variation in residence time and agitation

Res. time	Spd	pH	Alkalinity (mg CaCO$_3$/L)
B	L	8.7	470.4
D	L	8.6	437.4
A	M	8.5	425.4
B	M	8.6	420.4
C	M	8.7	525.5
D	M	8.5	430.9
B	H	8.7	415.9
D	H	8.6	405.4

5. Conclusions

Primary Neutralisation

The neutralisation mixture, designated 845, which would enable the refinery to operate the SWN process at optimum conditions, was determined as

- Volume ratios: SNL = 0.8, ESW = 5.89, RMS = 0.6
- Molar Ratios: Mg/Al = 4.0, (Mg + Ca)/Na = 0.52
- pH = 8.9, [Al] = 686ppb, [V] = 97ppb.

Saline SNL residue from neutralised mud showed an increase in dissolved metals concentration and pH. Reprocessing of this stream through the primary neutralisation process would be required before it could be discharged safely to the environment.

Flocculation Testing

For nominal operating conditions, settling rates up to 20m/h were obtained with clarities of 20mg/L. Dual dosing to the reactor and clarifier improved the overflow clarity, but an excess of SNL in the mixture significantly reduced the settling rate and compaction of the mud. The neutralized mud behaved as regular red mud, while compaction and a transition to the paste regime was seen at around 41% solids.

Ecotox Analysis and Continuous-flow Optimisation

Analysis of the neutralised effluent for dissolved metal concentrations found that levels of Al, Cr, Ni, Ga, Mo and V were all below specified limits and ANZECC 2000 guidelines. The metal concentrations beyond 100m of the outfall from the refinery would be well below levels expected to have any biological impact. The particulate load will be unchanged if the TSS stays in the range of 20 – 50mg/L as expected. Variations in the residence time or agitation speed of the continuous reaction had no detectable impact on the neutralisation process. Excluding red mud from the reaction negatively impacted the pH reduction and metals removal efficiency of the process.

Acknowledgements

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