Removal of Total Hardness and Alkalinity from RO – Reject Water

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***Abstract*—**Eskom has a zero-effluent discharge policy stipulation in their water use license. Thus, the wastewater generated from various processes should be treated and recycled for re-use. One of these types of water is RO-reject. This can be treated through the crystallization process along with coagulation and flocculation. The coagulant used in this study was Rheofloc 5023, the flocculent Rheofloc 5414; lime was used to increase the pH of the water samples. An optimal dosage for the lime was found to be 250 ppm, when the dosages of the coagulant and the flocculent were 2 ppm and 1 ppm, respectively. Under such conditions, turbidity, total hardness and alkalinity removal of 18, 26 and 67% were respectively achieved. The reduction of the levels of such parameters in water can contribute to minimize the probability of scale formation in the downstream process.

***Keywords*—**RO–Reject, Total hardness, Alkalinity, Crystallization

I. Introduction

South Africa is classified as a water scarce country with an annual rainfall of 492mm [1 - 5]. The average rainfall for the rest of the world is 985mm. It is said by the Department of Water and Sanitation that the water demand for South Africa will exceed the water supply by 2025. It was determined in 2001 that the mining and industrial use of water was about 10.5% of the total water use in South Africa [6]. Furthermore mine effluents contribute significantly to the pollution of surface water [7 – 16].

Eskom is South Africa's main supplier of electricity. The utility has numerous power stations of various types. The water for this study was collected from Grootvlei Power Station, which is one of the return-to-service power stations. The Grootvlei Power Station is situated close to Balfour, Mpumalanga in South Africa. It is operated by Eskom and has a total station capacity of 1200 MW[17]. To generate this power, clean water is needed. Water is obtained from the environment, treated and used. Eskom has a zero effluent discharge policy which stipulates that no water is discharged back into the environment.

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The water used in this study is RO-reject water. Reverse osmosis (RO) is used to remove dissolved solids from water. RO process is based on a semipermeable membrane with a negative pressure difference. Particles that are not small enough cannot pass through the membrane pores (Fig.1). These particles are called the RO reject. In most cases the reject is classified as saline water and in extreme cases it is classified as brine. Thus it consists of several scaling agents such as calcium carbonate and magnesium carbonate and sulphate. These scaling agents should be removed to prevent scaling in further downstream processes[18 - 21].

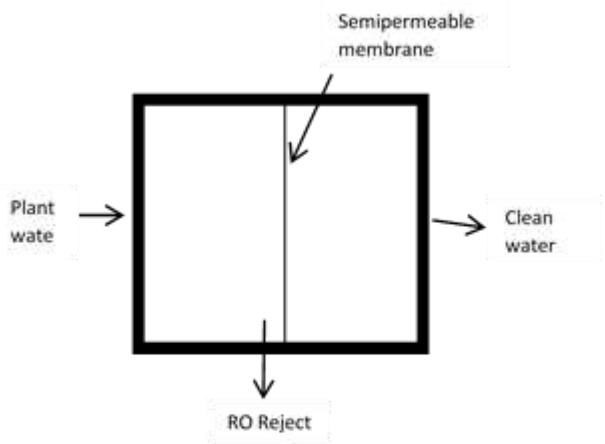

Fig 1: Representation of Reverse Osmosis

The reagents used for the treatment of the water were Polyamines (Rheofloc 5023 as coagulant and Rheofloc 5414 as flocculent) and hydrated lime ($CaCO_3$). The flocculation process results as the agglomeration of suspended coloids which settle as their density increases [22 - 26].

Polyamines are cationic molecules with a medium molecular weight, mostly linear. It is soluble, have a long shelf life, it has no odour and can be used over a wide pH range. Due to the length of the polyamine molecules, it wraps flocs together[27].

Lime softening can remove up to 78% of Strontium. This was achieved when the pH of the water was raised to 10.7 due to the addition of lime. At the same pH the reduction in calcium ion concentration was 86.4%. Magnesium removal was not noticed until the pH was above 10 to precipitate Mg $(OH)_2$. Thus strontium, calcium and magnesium removal are pH dependant and lime is relatively less expensive and highly effective substance to increase the pH [28].

However using the coagulants and flocculants in conjunction with the lime can improve the results significantly. However a coagulant aid may be necessary to neutralise the surface charge. Polymeric flocculants are being used to neutralise surface charge and aid in the agglomeration of particles through charge patch or bridging. This will ensure

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the particles to sink or float depending on the density of the particles[27]. The density of $CaCO_3$, the product that will probably form, is high relative to the density of water, thus it will sink. The removal of the agglomerated particles will then aid in the softening of the water.

Thus scale forming agents including Ca and Mg ions along with carbonates and sulphates can be removed from the water.

Several tests will be conducted to determine the optimal dosage for all the above mentioned reagents. These tests include jar tests, pH, conductivity, turbidity, total hardness and alkalinity.

The pH can be measured using hydrogen ion concentration in an aqueous solution using equation:

Equation1: pH equation

$$pH = \log[H^+]$$

The pH provides data about the acidity of the solution and is usually measured using a probe that is connected to a pH meter[29]

The conductivity is the potential for a substance to conduct or transmit heat, electricity and sound. Metal ions increase the conductivity of a solution. Thus a lower conductivity will prove that metal ions are removed from water. Conductivity is normally measured with a probe connected to a conductivity meter and the units for conductivity are Siemens/meter (S/m) [30].

The turbidity of the water can be described as the haziness or cloudiness of the water. Dissolved solids can increase the turbidity of water. Turbidity is measured in Nephelometric Turbidity Units (NTU) and it is normally measured with a spectrometer as shown in the illustration below[31]:

The total hardness is the concentration of the hardening agents in the water, the scaling agents that needs to be removed. These substances are the $CaCO_3$ and $MgCO_3$. In this method, 25ml of sample is added to 25ml demineralised water. A total hardness buffer (2ml) is added to this sample as well as Calmagite. The Calmagite is the indicator. After the addition of the indicator, the sample will be purple. EDTA, the titrant, is added until the end point is reached. The endpoint is blue in colour[32]. The volume of EDTA added is measured and used in the following equation to determine the total hardness:

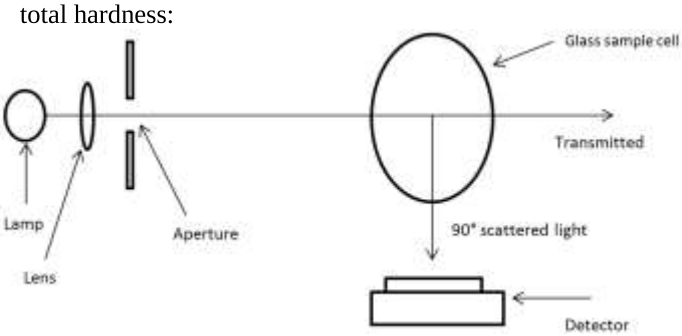

Fig 2: Spectrometer

Equation 2: Total hardness (mgCaCO$_3$/L)

$$\text{Hardness}\left(mg\,\frac{CaCO_3}{L}\right) = \frac{A \times B \times 1000}{mL\ sample}$$

With:
A = mL titrant for sample
B = mg $CaCO_3$ equivalent to 1mL EDTA titrant.

The Magnesium hardness as Magnesium concentration in the sample can then be calculated by subtracting the Calcium hardness from the Total hardness.

The alkalinity can provide information about the Carbonate, bicarbonate and hydroxide content of a sample. A chosen amount of sample can be used for this test and 0.1N sulphuric acid is used as titrant[32].

There are two methods that can be used to determine the alkalinity, one with a pH meter and one with indicators.
In the first method (pH meter) the starting pH is measured. If above 8.3, the titrant is added until pH of 8.3 and a measurement is taken followed by the addition of titrant until a pH of 4.5.

In the second method, Phenolphthalein is used as an indicator for a pH of 8.3, when the colour of the sample changes from pink to transparrent5. Thereafter Bromosol Green is added for the 4.3 pH, where the sample colour changes from blue to green[32].

The alkalinity can then be calculated using the following equations:

Equation 3: Alkalinity (mgCaCO$_3$/L)

$$\text{Alkalinity}\left(\frac{mgCaCO_3}{L}\right) = \frac{A \times N \times 50000}{mL\ sample}$$

Where:
A = mL standard acid used
N = Normality of standard acid

Equation 3: Total Alkalinity (mgCaCO$_3$/L)

$$\text{Total alkalinity}\left(\frac{mgCaCO_3}{L}\right) = \frac{(2B - C) \times N \times 50000}{mL\ sample}$$

With:
B = mL titrant to first recorded pH
C = total mL titrant
N = Normality of acid
$CO_3^{2-} = 2P - 2[OH^-]$
$HCO_3^- = T - 2P + [OH^-]$
Where:
P = phenolphthalein alkalinity
T = Total alkalinity

The relationship between the different alkalinities is illustrated in the following Table 1.

TABLE 1: ALKALINITY EXPLANATIONS

Result of titration	Hydroxide alkalinity as $CaCO_3$	Carbonate alkalinity as $CaCO_3$	Bicarbonate alkalinity as $CaCO_3$
P=0	0	0	T
P<1/2T	0	2P	T-2P
P=1/2T	0	2P	0
P>1/2T	2P-T	2(T-P)	0
P=T	T	0	0

II. EXPERIMENTAL SETUP

The jar test equipment used at the North-West University School for Chemical a d Minerals Engineering consist of six paddle stirrers connected to a variable speed motor. There is rapid and slow mixing periods during the jar tests. The rapid mixing ensures that the reaction takes place and slow mixing ensures that flocs or crystals agglomerate to form a precipitate [28].

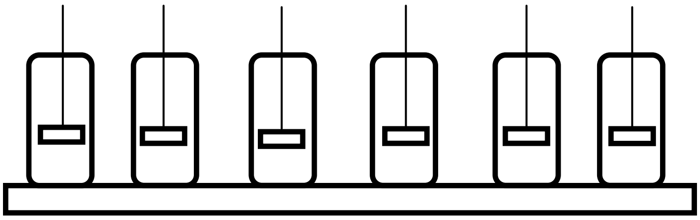

Fig 3: Jar test

The pH is calculated by measuring the hydrogen ions the pH

III. Experimental Procedure

Firstly, a 1000ppm Rheofloc 5023 (Coagulant) solution was made. Thus, 1ml of Rheofloc 5023 was added to 1L demineralized water. Adding 1ml of this solution then to the sample will add up to a 2ppm dosage. Exactly the same dosage was made up for the Rheofloc 5414 (Flocculent).

A 10 000-ppm lime solution was made up by the addition 10g CaOH powder to 1L water. Thus, 1ml of lime solution will provide a 20ppm dosage in a 500ml sample.

A water bath is used to heat the water to 40°C where it is pumped into a bracket where the 6 500ml samples are placed. This aids as a heat exchanger to increase the temperature of the samples to 40°C. After the desired temperature is reached, the different coagulant and flocculent dosages are done and rapid mixing occurs at 240rpm for 5 minutes. Thereafter slow mixing occurs at 80rpm for 90 minutes where lime is dosed until a pH of roughly 10.1 occurs. After the 90 minutes, the samples are left to settle for 30 minutes. After the settling time, the conductivity, turbidity, total hardness and alkalinity is tested.

Firstly, the coagulant dosage was varied between 0.2ppm and 7ppm. The flocculent dosage was 0.2ppm and the lime dosage
250ppm. These results of the tests are illustrated in Table 2.

Thereafter the same coagulant dosages were used with a flocculent dosage of 0.5ppm. The data obtained during this experiment is provided in Table3.

Next, the same coagulant dosages were used in conjunction with a 1ppm flocculent dosage and a 250ppm lime dosage.

The results obtained from this experiment are provided in Table 4.

The exact coagulant dosage was then used with a flocculent dosage of 7ppm and a lime dosage of 250ppm. The results obtained can be observed in Table 5.

Lime alone was also used in a study to predict the efficiency of the coagulants and flocculants. A lime dosage of 250ppm was used and the results obtained from the experiments supplied in Table 6.

The coagulant and flocculent was also used without the lime to observe the effect of lime on the process. The results are illustrated in Table 7.

IV. Results and Discussion

Table 2: Results for 0.2ppm flocculent dosage

Constant flocculent dosing of 0.2ppm (Rheofloc 5414)				
Coagulant dosage (ppm)	Conductivity removal (%)	Turbidity removal (%)	Total Hardness removal (%)	Alkalinity removal (%)
0.2	11	-115	20	43
0.5	9	-113	13	47
0.7	10	-140	20	50
2	10	-89	20	50
5	10	-148	20	83
7	11	-51	20	57

As can be seen from Table 2, with a 0.2ppm flocculent dosage, the maximum total hardness removal is 20%. The optimal dosage from this experiment is the 7ppm coagulant dosage, as it has the combined best conductivity removal (11%), Turbidity removal (-51%) and total hardness removal (20%). The 7-ppm coagulant dosage also has a 57% reduction in the alkalinity.

Table 3: Results obtained with 0.5ppm flocculent dosage

Constant flocculent dosing of 0.5ppm (Rheofloc 5414)				
Coagulant dosage (ppm)	Conductivity removal (%)	Turbidity removal (%)	Total Hardness removal (%)	Alkalinity removal (%)
0.2	11	-165	21	57
0.5	12	-686	24	63
0.7	12	-174	25	57
2	13	-119	24	60
5	12	-186	24	60
7	12	-196	24	57

The 0.7ppm dosing had the highest total hardness removal and thus was considered to be the optimum dosage of coagulant. The conductivity removal was 12%, the turbidity increased. The starting turbidity was low, has a turbidity increase of 1.74 times will not be a problem as the turbidity was already low before treatment occurred. With this dosage, there is also a 57% reduction in thee alkalinity.

Table 4: Results obtained with 1ppm flocculent dosage

Constant flocculent dosing of 1ppm (Rheofloc 5414)				
Coagulant dosage (ppm)	Conductivity removal (%)	Turbidity removal (%)	Total Hardness removal (%)	Alkalinity removal (%)
0.2	13	-21	23	50
0.5	13	-592	27	60
0.7	13	-73	26	57
2	12	18	26	67
5	13	26	24	57
7	12	-102	26	60

The 0.5ppm dosage yielded the highest total hardness removal (27%), a conductivity removal of 13% and an increase in the turbidity of 5.92 times. The alkalinity reduction at this dosage is 60%. This is not the highest, but in conjunction with the total hardness removal, it is the best dosage for this experiment.

Table 5: Results obtained with 7ppm flocculent dosage

Constant flocculent dosing of 7ppm (Rheofloc 5414)				
Coagulant dosage (ppm)	Conductivity removal (%)	Turbidity removal (%)	Total Hardness removal(%)	Alkalinity removal(%)
0.2	14	-89	25	43
0.5	13	32	22	50
0.7	12	-76	27	57
2	13	-62	26	57
5	13	-112	28	57
7	13	-29	25	53

A coagulant dosage of 5ppm yielded the highest total hardness removal (28%) as well as 13% conductivity removal and an increase of 1.12 times of the turbidity. The alkalinity removal is 57%, which is also the best removal percentage of this experiment.

Thus a coagulant dosage of 5ppm, flocculent dosage of 7ppm and a lime dosage of 250ppm will be the optimal dosage for the removal of scaling agents in the RO-reject water. This is proven with the 28% removal of total hardness, which represents the calcium and magnesium ions in the water. These ions along with carbonates, sulphate and hydroxides are the main cause of scaling in power plants heat exchangers. This is also the overall best total hardness removal, thus the optimal dosages for the treatment of RO-reject water.

TABLE 6: RESULTS OBTAINED WHEN ONLY LIME WAS USED

Conductivity removal (%)	Turbidity removal (%)	Total Hardness removal (%)	Alkalinity removal (%)
13	-89	23	48

Thus the total hardness removal when only lime was used is 23% with a conductivity removal of 13% and an increase in the turbidity with 89%. The alkalinity removal is lower (48%) than most of the experiments where coagulants and flocculants were used. Thus the coagulants and flocculants are necessary in the reduction of scaling agents in the RO-reject water.

TABLE 7: RESULTS OBTAINED WITHOUT LIME ADDITION

Conductivity removal (%)	Turbidity removal (%)	Total Hardness removal (%)	Alkalinity removal (%)
3	-59	0	0

As can be seen in the Table 7, the lime plays an integral part in the removal of scaling agents. Without lime the conductivity was reduced by 3%. There was a slight increase in the turbidity and the total hardness removal was 0%. There was absolutely no alkalinity removal as well. Thus without an increase in the pH due to the lime, the scaling agents will not be removed from RO-reject water.

V. CONCLUSION AND RECOMMENDATIONS

After numerous tests it can be confirmed that coagulation and flocculation occurred to remove scaling agents from RO-reject water. Lime alone only removes 23% of the total hardness from the water whereas with the optimal dosage with coagulants, flocculants and lime ensures a total hardness reduction of 28%. The optimal dosage for the lime was found to be 250ppm, while the optimal dosage for the coagulant (Rheofloc 5023) was found to be 2ppm and 1 ppm for the flocculent (Rheofloc 5414) when considering the reduction of turbidity, total hardness and alkalinity. It could be therefore concluded that a coagulant, a flocculent and a substance to increase the pH are all necessary for optimal removal of scale forming agents.

Further suggestions include the consideration of alternative coagulants and flocculants for a comparative study.

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