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Cyanide compounds removal efficiency in a reverse osmosis system using a water supply from a co-precipitation chemical process

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ABSTRACT

This article sets forth the removal efficiency for cyanide compounds in a full-scale reverse osmosis system supplied with water coming from a co-precipitation chemical process. This test was carried out for a month and a half, with a 72% recovery rate using Dow Filmtec thin-film composite membranes, the MDS-41 8040 model in the first and second stages, and the MDS-42 8040 model in the third stage. The results showed removal efficiencies for ammonia at 92.71%, and thiocyanates at 91.76%. For WAD cyanide, the system did not achieve good removal efficiency (47.93%), probably due to its low molecular weight and low inlet concentration level (0.054 ppm); however, this value is lower than the Peruvian Environmental Quality Standard (<0.10 ppm). According to the results, the highest removal efficiency has been determined for cyanate compounds at an average value of 95.33%. The results have shown the best average removal efficiency for monovalent cyanide compounds of higher molecular weight (cyanates and thiocyanates) and by-products of the oxidation and hydrolysis process of cyanide compounds (ammonium ion), representing an overall average efficiency of 93.26%. This finally confirms that the rejection of components in reverse osmosis is related to molecular weight and ionic charge.

Keywords: Cyanates; Thiocyanates; Chemical co-precipitation; Monovalent cyanide compounds; WAD cyanide

1. Introduction

Mining is an important extractive industry for the development of many countries in South America and the world [1]. Gold mining operations generate a great variety of waste products, which have to be disposed of responsibly in compliance with the environmental regulations of each country [2]. The increasingly restrictive environmental regulations regarding the quality of treated water discharged into receiving bodies of water and the proportionate use of this natural resource has led to the use of increasingly cleaner technologies [3].

The liquid effluents produced through the cyanide leaching technique create an immediate problem that represents an environmental threat due to the presence of toxic compounds in the liquid phase, such as cyanide species, toxic metal ions, and processed chemicals [1].

It is well known that sodium cyanide and some of its derivatives are toxic and that cyanide compounds are classified as hazardous. Cyanide (CN^-) in solutions after the gold extraction process by leaching is present in industrial effluents as free cyanide, weakly complexed cyanide, and strongly complexed cyanide, which constitute total cyanide. Because these materials are hazardous not only to humans, but also to receiving bodies of water, their toxicity is mainly related to their form and appearance [4]. The toxicity of other compounds; related to cyanide, produced from the reactions of cyanide with sulfur ore or water treatment, can generate thiocyanate (SCN^-), and those that occur with oxygen and water, form cyanate (CNO^-) and

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ammonia (NH_3) [5]. These compounds are important, both from a toxicological perspective and from a water treatment perspective [6].

Different treatment methods currently apply to these types of effluents, such as cyanide destruction, chemical precipitation, neutralization, ion exchange, electrochemical processes, and membrane separation processes [7]. Up until now, the commonly used solution in industries to remove cyanide involve complex chemical processes, such as alkaline chlorination, hydrogen peroxide oxidation, and INCO SO_2/air oxidation. In addition to cyanide treatment, processes must be evaluated for their effectiveness in treating and removing residual metals present in cyanide solutions. Although the metallurgical processes used in a mining operation are similar, the wastewater characteristics vary widely, and there is not just one procedure for its purification.

The traditional processes as neutralization and chemical precipitation use large amounts of alkaline reagents. Their main disadvantage is the generation of large volumes of sludge containing heavy metal compounds that must be disposed of. These processes have a certain limitation for complying with increasingly stringent environmental laws regarding treated effluents [1,3].

Within these treatment alternatives, membrane separation processes present many attractive advantages, for example, generation of high quality permeate, metal concentrations, and low operating costs. Rejection capacities of 90%–99% for cyanide and higher than 92% for other important ions have been reported with the use of polyamide composite membranes, which ensures the quality of the treated effluent [8]. This process is presented as a technically, environmentally, and economically viable alternative for the treatment of liquid effluents in the mining sector [9].

Reverse osmosis is the most widely used desalination process based on membrane separation in the world, capable of rejecting almost all of the colloidal and dissolved matter present in an aqueous solution, creating two streams: concentrate (brine) and permeate (almost water pure) [10]. This process is based on the membrane based demineralization technique, used to separate dissolved solids such as ions from a solution [8]. These membranes generally act as selective barriers, which allow some species to selectively cross this barrier (such as water), permeate through the membrane while retaining other dissolved species (such as ions) [11]. The reverse osmosis process is based on the technique of separation driven through differential pressure. The applied pressures in reverse osmosis vary between 10 to 15 bar (brackish water desalination) and 60 to 80 bar in seawater desalination [12].

The reverse osmosis process can be used in two main applications: to purify water or to concentrate and recover dissolved solids from feedwater [13]. The most common application is the production of drinking water, but it is also applied in the treatment of effluents and the separation of organic and inorganic compounds from aqueous solutions for industrial applications [14]. The objective of this article is to explain the application of this technology in the reuse and recovery of process water in the mining sector.

Due to the rise and expansion of this technology in recent decades for dealing water scarcity, not just in arid regions, pollution and exploitation of underground aquifers and surface waters have led to a decrease in this resource, and this process has become especially interesting due to the continued development of membrane technologies more resistant to extreme conditions, as well as the ability to generate excellent quality water for reuse [15].

With that in mind, this article presents the performance evaluation of a large-scale reverse osmosis treatment plant used as a post-treatment stage of mining effluents from two processes: cyanide destruction and chemical co-precipitation with ferric sulfate (Fig. 1). The main objective of this article is to show the removal efficiency of the main cyanide species and treatment byproducts: WAD cyanide, thiocyanates, cyanates, and ammonia in a reverse osmosis unit.

2. Materials and methods

2.1. Study material

Comprised of the pre-treated wastewater from the chemical co-precipitation stage with ferric sulfate, and post treated wastewater in a reverse osmosis process. The characterization of the pre-treated effluent is shown in Table 1.

2.2. Experimental equipment

The water treatment plant consists of an ultrafiltration (UF) system and a reverse osmosis (RO) system. These systems are made up of various pieces of equipment designed to treat feedwater. Pre-treatment equipment removes suspended and colloidal material from feedwater. This stage has disc filters, ultrafiltration, and a chemical injection system. The reverse osmosis system consists of low-pressure and high-pressure feed pumps and a set of membranes. This unit removes dissolved solids from feedwater generating a clean permeate stream and a concentrate stream that carries the removed salts.

Feedwater from the chemical co-precipitation stage is pumped into a feed tank, and from that through the disc filters that, remove suspended particles greater than 120 um. These filters automatically backwash based on time or pressure differential. After the disc filters, the effluent is fed directly to the ultrafiltration system, and this unit removes almost all of the remaining suspended solids from the feedwater. Due to the high degree of level of the pre-treatment stage, the ultrafiltration system operates at a recovery rate of 95%.

Cartridge filters are located before the reverse osmosis unit, and a 10–15 psi pressure differential across this unit indicates that filters are clogged and should be changed. They are used as a protective barrier before the reverse osmosis unit.

The reverse osmosis unit includes a chemical injection system to protect the membrane system: sodium bisulfite to control the presence of oxidizing agents, acid to control calcium carbonate scaling potential, and a scale inhibitor to control the formation of major scale by calcium carbonate and calcium sulfate.

The reverse osmosis system has a three-stage array, which allows for a 72.00% recovery rate. The reverse osmosis unit consists of 30 pressure vessels with an 18:8:4 array.

Each vessel contains 6 standard size spiral wound membrane elements: 8 inch in diameter and 40 inch length.

During the production stage, the effluent from cartridge filters enters the first stage made up of 18 pressure vessels. Permeate from these vessels is directed to a permeate collector, and concentrate from these vessels is then sent to the second stage comprised of 8 pressure vessels.

Permeate produced from this second stage also enters the permeate collector. Concentrate from this stage enters the third and final stage comprised of 4 pressure vessels. Permeate produced in the last stage enters the permeate stream, where the total permeate flow is then determined.

The concentrate stream from the third stage passes through a concentrate control valve for pressure and concentrate flow control.

The design specifications of the reverse osmosis unit, the subject of evaluation in this article are shown in Table 2.

2.3. Method

A plant test was established to evaluate the performance of the reverse osmosis treatment plant. This test was carried out by taking samples of the influent and effluent water of the reverse osmosis system for one and a half months.

Table 1
Characteristics of pre-treated wastewater supplied to the reverse osmosis system

Parameter	Quantity
Cyanates, mg/L	206.700
Thiocyanates, mg/L	327.642
WAD cyanide, mg/L	0.054
Ammonia, mg/L	65.69
Calcium, mg/L	529.219
Cadmium, mg/L	<0.003
Cobalt, mg/L	0.356
Chromium, mg/L	0.029
Cesium, mg/L	<0.002
Copper, mg/L	1.194
Iron, mg/L	0.202
Potassium, mg/L	44.264
Lithium, mg/L	0.016
Magnesium, mg/L	1.171
Manganese, mg/L	0.009
Molybdenum, mg/L	0.264
Sodium, mg/L	1175.156
Nickel, mg/L	0.149
Lead, mg/L	0.002
Antimony, mg/L	0.029
Selenium, mg/L	0.317
Strontium, mg/L	1.460
Zinc, mg/L	0.088
Nitrates, mg/L	60.497
Nitrites, mg/L	6.160
Sulfates, mg/L	4084.416
pH	7.25
Specific conductivity, us/cm	7010.00

Source: Plant process laboratory – 2020.

Approximately 1 L samples of the feed, permeate, and concentrate streams were collected, which were sent to the processing plant laboratory for their respective analysis.

The reverse osmosis unit worked at a treatment capacity of 400 m^3/h and a 72% recovery rate during the test.

2.4. Control and analysis

2.4.1. Parameters measured during the operation

Ammonia (Standard methods: Ammonia-Selective Electrode Method 4500-NH_3 D), cyanates (Standard methods 4500-CN^- L), thiocyanates (Standard methods 4500-CN^-M), WAD cyanide (Standard methods: Weak Acid Dissociable Cyanide 4500 CN^-– I), pH (Standard methods: Electrometric Method 4500-H^+ B), conductivity (Standards methods: Laboratory Method 2510 B), nitrates (Determination of inorganic anions by ion chromatography EPA 300.1), nitrites (Determination of inorganic anions by ion chromatography EPA 300.1), sulfates (Determination of inorganic anions by ion chromatography Method EPA 300.1). Elements: calcium, cadmium, cerium, cobalt, chromium, cesium, copper, iron, potassium, lithium, magnesium, manganese, molybdenum, sodium, nickel, lead, antimony, selenium, strontium, and

Table 2
Design specifications of the reverse osmosis treatment plant

Specification	Description
Design flow, m^3/h	400
Recovery rate, %	72.00
Array	18:8:4
Elements in pressure vessels	6.0
Membrane specification: first and second stages	
Type: Dow Filmtec thin-film composite membrane	
Model: MDS-41 8040	
Active area, m^2	40
Feed spacer, mil	28
Permeate flow, m^3/d	34.2
Average operation flux, LMH	13
Minimum salt rejection, %	99.25
Maximum pressure, bar	68
Membrane specification: third stage	
Type: Dow Filmtec thin-film composite membrane	
Model: MDS-42 8040	
Active area, m^2	34
Feed spacer, mil	31
Permeate flow, m^3/d	30.6
Average operation flux, LMH	11
Minimum salt rejection, %	99.60
Maximum pressure, bar	68

Source: Descriptive report of water treatment plant – membrane specifications zinc. (Determination of trace elements by inductively coupled plasma – Mass Spectrometry Method EPA 200.8) [16,17].

The analysis of results focused on evaluating the removal efficiency of the reverse osmosis system for cyanide compounds and treatment by-products with the highest risk, as well as environmental impact due to their toxicity and concentration.

3. Results and discussion

Table 1 presents the physical–chemical analysis of the pre-treated wastewater supplied to the reverse osmosis system. This characterization showed a high-level concentration in regards to the compounds under study; cyanates: 206.700 ppm, thiocyanates: 327.642 ppm, and ammonia: 65.69 ppm; except that in regards to the WAD cyanide concentration: 0.054 ppm, an important part of this evaluation from a toxicological perspective, its presence in higher concentration levels cause adverse effects on the aquatic environment and fish deaths in receiving bodies of water.

Table 3 (Fig. 2), presents WAD cyanide concentration levels in the process streams of the reverse osmosis unit, showing a low average WAD cyanide concentration level in the feed: 0.060 ppm, a value lower than Peruvian environmental laws (<0.10 ppm WAD CN^-), resulting from the cyanide destruction process of the pre-treatment stage. The average removal efficiency for WAD cyanide at 47.93% was reached from the feed stream, for this inlet concentration level.

In the reverse osmosis process, the pH of the solution affects the ionization of dissoved constituents and subsequently their rejection: the greater the charge retained by a constituent, the higher the level of rejection. In the case of CN^- (free cyanide), the degree of rejection is strongly dependent on the pH of the feed stream, at a pH of 7.25, it mainly occurs in molecular form ($CN^- + H_2O \Leftrightarrow HCN + OH^-$). Other studies in reverse osmosis systems for cyanide removal have shown good results at a pH > 11.5 [1,18].

Results from Table 4, shows us higher removal efficiencies for cyanide in feed streams with pH > 11.5, and in a system working with a double pass reverse osmosis, reaching a cyanide removal of 98.98%. However the permeate from the second pass did not get values below the international environmental quality standards for free cyanide (<0.20 mg/L). For WAD cyanide the most important parameter in the determination of the stability or dissociation degree of these cyanide-metal complexes is the pH of the dissolution. It is also important to mention here that

Table 3
Determination of WAD cyanide in feed, permeate, and concentrate tank; and removal efficiency

Date	Cyanide (WAD CN^-)			
	Feed (mg/L)	Permeate (mg/L)	Concentrate (mg/L)	% Removal
02-06-2020	0.062	0.034	0.142	45.16
02-12-2020	0.059	0.035	0.136	40.68
02-18-2020	0.055	0.028	0.129	49.09
02-24-2020	0.052	0.025	0.131	51.92
03-02-2020	0.074	0.034	0.148	54.05
03-08-2020	0.068	0.039	0.149	42.65
03-14-2020	0.052	0.025	0.200	51.92
Average	0.060	0.031	0.148	47.93

Source: Plant process laboratory – 2020.

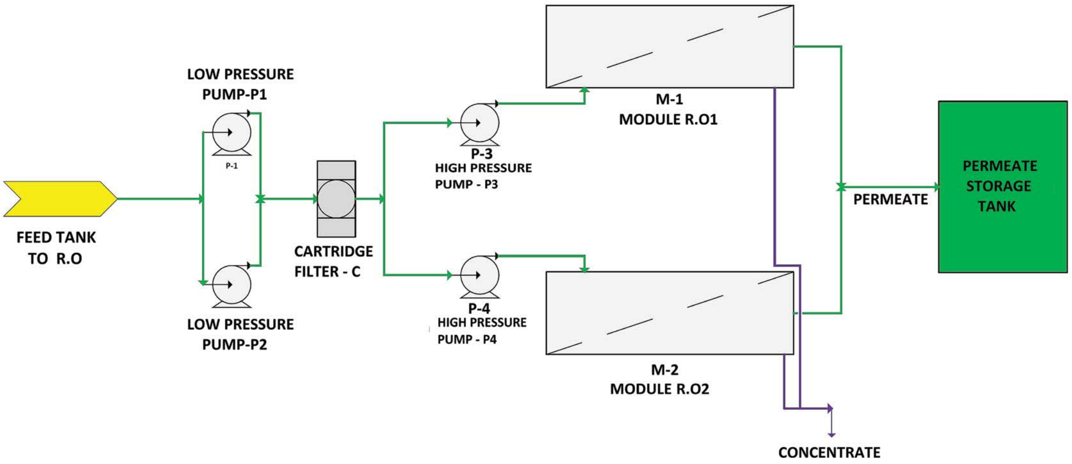

Fig. 1. Reverse osmosis plant flow diagram.

there are other parameters that affect the stability of WAD cyanide such as metal cation and redox potential.

As recommendation to improve cyanide removal efficiency, it is important to continue the tests in a double pass reverse osmosis system with a pH in the feed stream of the first pass lower than 6.0, in order to obtain the WAD cyanide dissociation into free cyanide; and with a pH in the feed stream of the second pass higher than 11.5, in order to obtain a better cyanide removal efficiency.

Table 5 (Fig. 3), presents the concentration levels of thiocyanates in the process streams of the reverse osmosis unit, showing an average concentration level of thiocyanates in the feed of 232.93 ppm, resulting from the chemical reaction between cyanide with unstable sulfur atoms during

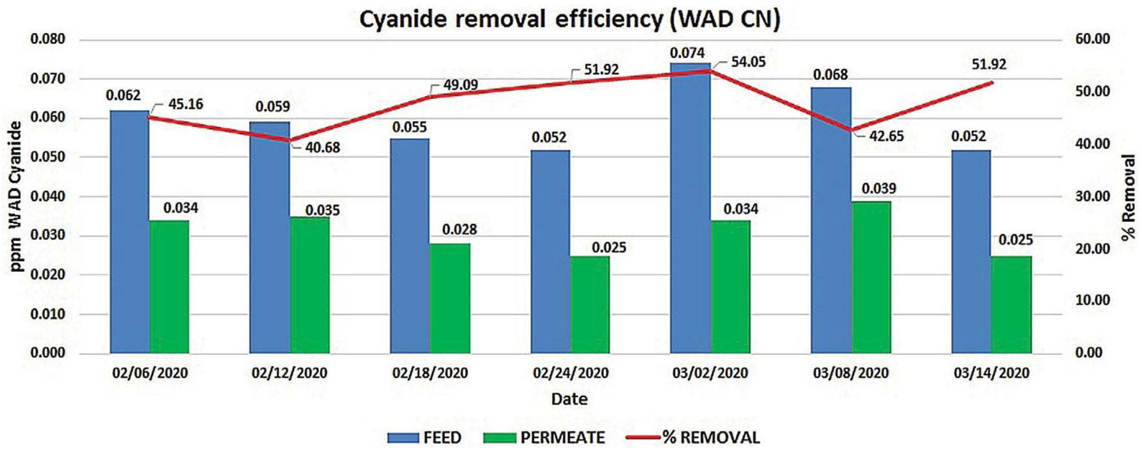

Fig. 2. Graph of WAD cyanide concentration in the feed, permeate; and removal efficiency.

Table 4
Analytical data of samples in the feed, concentrate, and permeate in a reverse osmosis system

Test	Reverse osmosis	Sample	pH	CN⁻ (mg/L)	% Removal
		Feed	10.20	92.90	
1	First pass	Concentrate	10.30	122.00	11.95
		Permeate	10.60	81.80	
	First pass	Feed	11.80	78.70	
		Concentrate	11.80	324.00	91.11
2		Permeate	11.00	7.00	
	Second pass	Concentrate	11.40	21.00	98.98
		Permeate	10.70	0.80	

Source: Adapted from Institute of Chemical Technology Prague, Faculty of Environmental Technology, Technická 5.

Table 5
Determination of thiocyanates in feed, permeate, and concentrate tank; and removal efficiency

Date	Thiocyanates (SCN^-)			
	Feed (mg/L)	Permeate (mg/L)	Concentrate (mg/L)	% Removal
02-06-2020	188.73	21.91	567.32	88.39
02-12-2020	258.00	14.40	745.00	94.42
02-18-2020	290.44	19.21	681.00	93.39
02-24-2020	298.76	23.07	875.69	92.28
03-02-2020	230.93	22.47	817.73	90.27
03-08-2020	184.44	14.95	682.59	91.89
03-14-2020	179.20	14.94	702.29	91.66
Average	232.93	18.71	724.52	91.76

Source: Plant process laboratory – 2020.

the aeration and ore leaching processes. The environmental effects of these compounds are mainly their toxicity and the generation of decomposition products such as ammonia. The average removal efficiency for thiocyanates of 91.76% was achieved.

Table 6 (Fig. 4), presents the cyanate concentration levels in the process streams of the reverse osmosis unit, showing an average concentration level of cyanates in the feed of 122.01 ppm, generated from the oxidation of thiocyanates, and another part from the direct oxidation of cyanide.

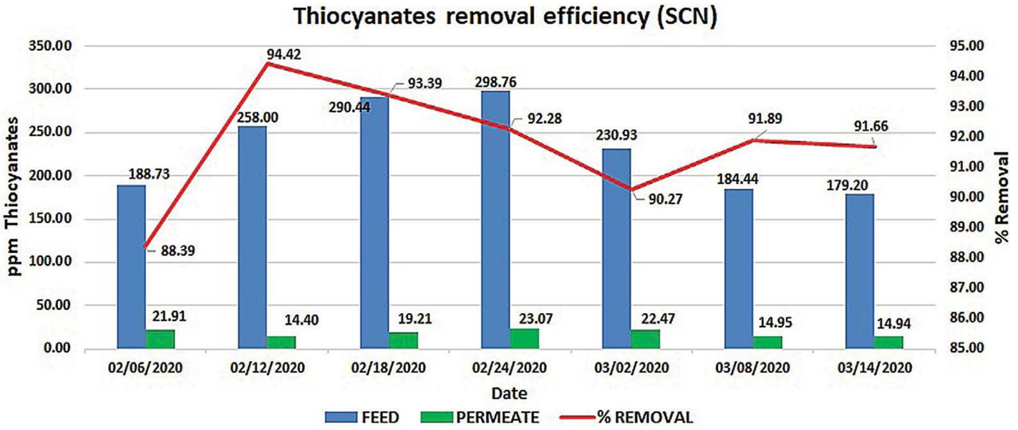

Fig. 3. Graph of thiocyanate concentration in the feed, permeate; and removal efficiency.

Table 6
Determination of cyanates in feed, permeate, and concentrate tank; and removal efficiency

Date	Cyanates (CNO^-)			
	Feed (mg/L)	Permeate (mg/L)	Concentrate (mg/L)	% Removal
02-06-2020	191.10	8.76	555.00	95.42
02-12-2020	65.10	3.09	624.00	95.25
02-18-2020	117.60	3.87	246.00	96.71
02-24-2020	99.60	3.39	378.00	96.60
03-02-2020	103.20	6.00	312.00	94.19
03-08-2020	126.00	7.74	489.00	93.86
03-14-2020	151.50	7.08	396.00	95.33
Average	122.01	5.70	428.57	95.33

Source: Plant process laboratory – 2020.

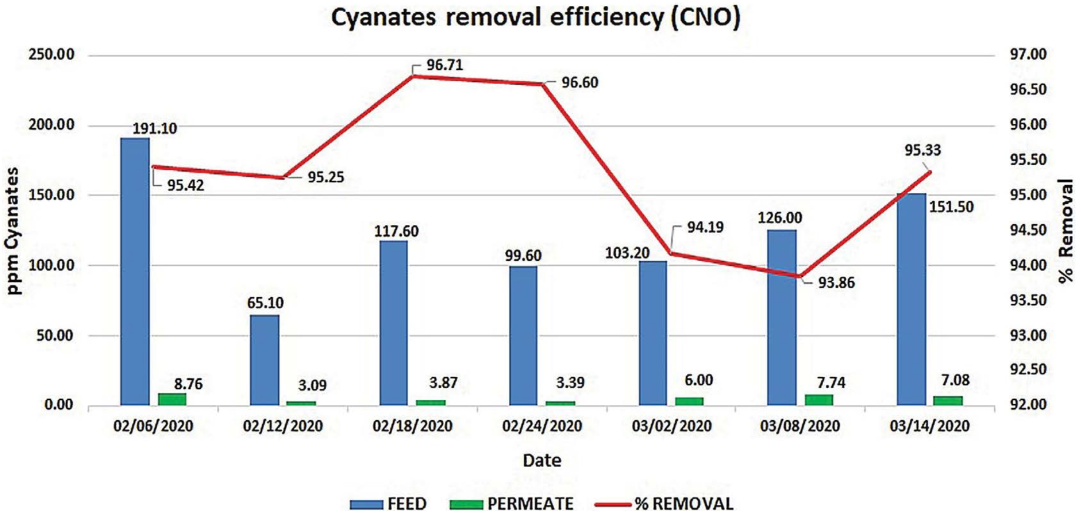

Fig. 4. Graph of cyanate concentration in the feed, permeate; and removal efficiency.

Table 7
Determination of ammonia in feed, permeate, and concentrate tank; and removal efficiency

Date	Ammonia (NH_3)			
	Feed (mg/L)	Permeate (mg/L)	Concentrate (mg/L)	% Removal
02-06-2020	57.44	4.44	165.57	92.27
02-12-2020	79.78	5.90	172.43	92.60
02-18-2020	53.79	2.32	139.64	95.69
02-24-2020	57.92	5.15	206.43	91.11
03-02-2020	54.52	4.22	184.57	92.26
03-08-2020	45.66	3.29	170.00	92.79
03-14-2020	50.87	3.93	160.28	92.27
Average	57.14	4.18	171.56	92.71

Source: Plant process laboratory – 2020.

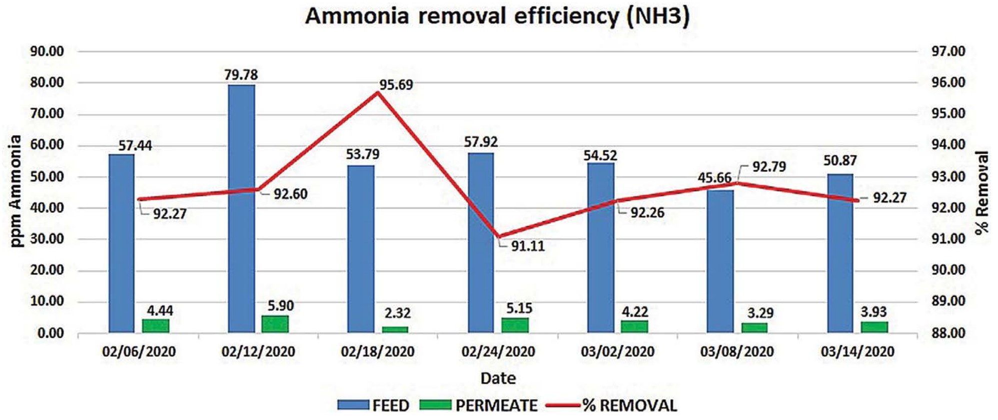

Fig. 5. Graph of ammonia concentration in the feed, permeate; and removal efficiency.

Similar to thiocyanates, its importance from the toxicological perspective is the generation of decomposition products and ammonia by hydrolysis of this compound under acidic conditions. The average removal efficiency for cyanates of 95.33% was achieved.

Table 7 (Fig. 5), presents the ammonia concentration levels in the process streams of the reverse osmosis unit as showing an average ammonia concentration level in the feed of 57.14 ppm, reaching at this stream concentration level, an average removal efficiency for ammonia of 92.71%, which has been relatively limited due to lack of greater displacement of the equilibrium in the formation of ammonium ions, an element removed by reverse osmosis; keeping the feed pH lower than 6.0, would have allowed for better removal efficiencies for this compound because the presence of ammonia and ammonium ions is a function of pH, $NH_3+H_2O \Leftrightarrow NH_4^+ + OH^-$ (influent pH=7.25) [8].

Table 8 (Fig. 6), presents a summary table of removal efficiencies for the cyanide compounds and treatment by-products under evaluation: WAD cyanide, thiocyanates, cyanates, and ammonia.

Table 8
Summary table of cyanide compound removal efficiency

Date	% Removal efficiency			
	Cyanide	Thiocyanates	Cyanates	Ammonia
02-06-2020	45.16	88.39	95.42	92.27
02-12-2020	40.68	94.42	95.25	92.60
02-18-2020	49.09	93.39	96.71	95.69
02-24-2020	51.92	92.28	96.60	91.11
03-02-2020	54.05	90.27	94.19	92.26
02-08-2020	42.65	91.89	93.86	92.79
03-14-2020	51.92	91.66	95.33	92.27
Average	47.93	91.76	95.33	92.71

Source: Tables 3, 4, 5, and 6.

4. Conclusion

- It is concluded that the highest removal efficiency in the reverse osmosis unit with the use of mixed membranes of the type: Dow Filmtec thin-film composite membranes, the MDS-41 8040 model in the first and second stages, and the MDS-42 8040 model in the third stage, was achieved for the cyanate compound, at an average value of 95.33%.
- Average removal efficiencies of 91.76% and 92.71% have been achieved for thiocyanate and ammonia respectively. The first one with characteristics

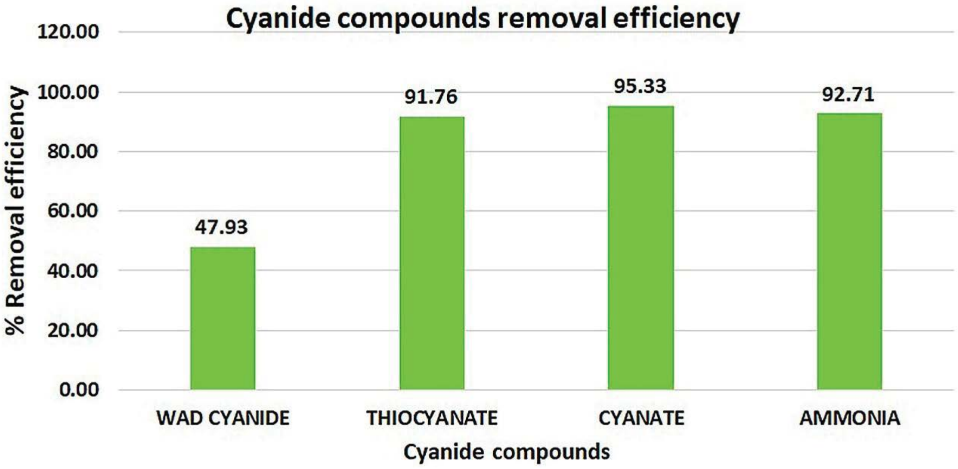

Fig. 6. Cyanide compounds removal efficiency.

similar to cyanate and the last one mainly due to influent pH = 7.25, allowing a greater presence of ammonium ions for removal.

- Cyanide has presented the lowest removal efficiency at 47.93%, probably due to the lower inlet concentration and mainly to its molecular form as monovalent species, as well as due to the lower pH in the feed stream.

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