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Effect of Operating Parameters on Bromine Extraction from Desalination Plant Discharge

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Abstract

A comprehensive utilization of concentrated seawater is crucial in order to promote the development of the desalination industry as a key solution to global freshwater. Debromination of the desalination plant effluent as well as the bromine product extraction are two parallel goals, which have been the subject of many research studies as well as industrial operations. In this investigation, bromine extraction is investigated experimentally form the effluent of the Konarak desalination plant located in Chabahar bay, Iran. For this purpose, an air blow-out method is used, and the effects of the operating parameters including the temperature, pH, and chlorine gas flow rate are examined in a continuous reactor. The parameters are optimized, and the trend is discussed in details. The bromine concentration of the sample collected from the Pozm Tiyab area, close to the plant discharge point, has been determined to be 1.172 g/L using ion chromatography. A pre-concentration procedure is conducted in order to reach a concentration of 3.100 g/L by evaporation. A reactor with the dimensions of 60 mm × 800 mm is designed and assembled for the experimental studies. In order to investigate the operating parameters, a central composition design (CCD) method is used. Among the factors studied, only the chlorine gas flow rate has a substantial effect on the bromine recovery, and the effects of the other two factors are negligible in the pH range of 2-3 and the temperature range of 50-70 °C. At the three chlorine concentrations of 1, 1.5, and 2 L/min, the bromine production increases almost linearly with the increasing chlorination injection rate. The Br2 gas is recovered with a maximum rate of 93.8% and a bromine loss of 185 mg/L in the mother liquid. The optimum operating parameters to achieve this recovery are a pH of 2.5, a temperature of 60 ˚C, and a chlorine gas flow rate of 1.5 L/min.

1. Introduction

Due to the global water crisis, the desalination industry has flourished in order to obtain new water resources and increase the total supply of freshwater [1]. However, the discharge of concentrated wastewater has raised a lot of the environmental challenges as well as the loss of valuable chemical resources [2-4]. Thus for environmental protection and resource recovery, the utilization of desalination plant discharge has received a lot of attention in the past few decades [5].

Bromine is so reactive, and hence, does not occur in the free elemental state in nature, and only exists as bromide ion (Br^-) [6], mostly in seawater, salt lakes, inland seas, and brine wells [7]. There are two different goals when treating bromide-containing brines, production of bromine product as well as debromination of the brine, specially desalination of plant effluents [8, 9]. Bromine is widely used in the chemical and pharmaceutical industries, mainly used as flame retardants, pesticide, and reactive intermediates in organic synthesis and pharmaceutical ingredients [10, 11]. On the other hand, an additional benefit from brine mining is the significant reduction of the volume of effluent discharge and increase of the recovery of

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desalination plants [2]. The production of bromine from brines is economic when the bromide concentration is more than 2.5 g/L [12]. In the sources with a lower concentration, it will have to be pre-concentrated, mostly in solar evaporation ponds.

In most sources, concentration of the chloride ions is more than 100 times higher than the bromide ions. Therefore, selectivity is the most significant role playing factor in the extraction of bromine from these sources. Only a few industrial processes for bromine extraction from brine resources have been established. The most used techniques include the steaming-out and air blow-out methods [13]. Compared with steam distillation, air the blow-out method is used for brines with lower bromide concentration, so it is applied more widely [1]. These methods still have many weaknesses, mainly high energy consumption and capital costs [1, 14]. Other methods are solvent extraction, ion exchange, membrane-based methods as well as electrochemical oxidation techniques that are all in the early development stages [1, 15, 16].

In the steaming-out and air blow-out techniques, bromine selective separation is conducted via the oxidation of bromide to bromine, mainly using chlorine or hydrogen peroxide [17]. The method is based on two principles; bromide has a lower standard redox potential than chloride (1.07 V *vs.* 1.36 V) [18] and bromine is extremely volatile (with a heat of vaporization of 30.91 kJ/mol and a boiling point of 59 $^{\circ}$C) [19]. There are not many oxidizing agents that can oxidize bromide ions. Chlorine acts as the most suitable and economical oxidizer, and produce bromine gas through an electro-chemical reaction, as follows (Eq. 1) [1]:

$$2Br^{-} + Cl_2 \rightarrow 2Cl^{-} + Br_2 \quad (1)$$

The reaction is so rapid with the specific rate and activation energy of 88.8 mol s^{-1} and 71.1 kJ mol^{-1} at 25 $^{\circ}$C, respectively [20]. The production of bromine is done in two steps. In the first step, the bromide ions are oxidized with the chlorine gas, and the bromine ions are stripped with the steam in the same column. The second step is the purification of the bromine ions from water and chlorine by rectification in a distillation column. These steps are carried out in the columns interlinked [15]. The purification is eased by the fact that the bromine ions have a low solubility in water (only 33.5 g/L in 25 $^{\circ}$C) [11] so pure bromine (99.9%) can be withdrawn at the bottom of the thermal separation column.

Chabahar is located in the Sistan and Baluchestan Province, Iran. It is a free port on the coast of the Gulf of Oman, and is the Iran's southernmost city. Pozm Tiyab is a small town in the Konarak, from which the sample for this research work was collected near the effluent discharge of the desalination plant. The desalination plant in Konarak has been established for 15 years, and produces 15-30 L/s desalinated water. The bromine ion concentration in the several samples collected from different parts of the Chabahar bay is compared with the Dead sea in Table 1.

Table 1. Comparison of bromine concentration in Chabahar and dead sea.

Chabahar Samples	Bromine concentration (g/L)	Dead sea Samples	Bromine concentration (g/L)
2	3.84	Initial solution	4.5
3	5.80	Carnalite extraction pond	9
4	8.10	Final brine	12
5	10.60	Potash extraction pond	5
6	11.28	Bischofite solution	6

The values of bromine ion concentration in Chabahar bay shows an excellent potential for bromine production, as the sole producer in Iran. On the other hand, some environmental research work results show that bromine-containing effluent discharged from the Konarak desalination plant has influenced the community structure in the Chabahar bay significantly (operating since 2007). The lack of bromine resources in the country as well as the need for debromination of the desalination plant effluent in the Chabahar bay highlights the need and importance of this research work. Also while the production of bromine ions from brine and sea waters using chlorine gas has been established and many plants all around the world are operating using this method, very limited research works on the role of the operating parameters and the optimized values have been reported [21]. Due to the declining trend of bromine ion concentration in brine resources (5-10% per year) [22], the quest for optimal recovery of bromine in these lower grade resources serves as another strong motivation. In the electro-chemical reactions involving gas phases, five major controlling parameters include the pH, redox potential, flow rate, temperature, and pressure [23]. Among the major factors, the three parameters of pH, temperature, and chlorine gas flow rate in constant redox potential at ambient pressure were examined using a central composite design (CCD) procedure. The trend observed in bromine recovery were investigated, and the optimum operating parameters were selected. This research work aims to provide more insight regarding the optimized process parameters in the bromine extraction from seawater using the air blow-out method. The results obtained can be effectively used for a better understanding of the role of the operating parameters in bromine extraction and how they affect the bromine recovery as well as debromination of the brine.

2. Materials and methods

For this work, 600 L of the brine sample from Pozm Tiyab area in Chabar bay was collected. The area is about 0.5-1 km away from the effluent discharge point of the Konarak desalination plant. In order to increase the concentration, the volume of the sample was reduced to 150 L by evaporation.

Due to the corrosive nature of bromine gas as well as the toxicity of both bromine and chlorine, a careful design of the extraction reactor was required. For this reason, several features and characteristics were considered in the design and construction of the continuous pilot-scale reactor to prevent gas leakage. Therefore, a Pyrex container was used to build the reactor with Teflon valves. In order to increase the gas bubble contact time, the reactor was designed in the form of a column with a 60 mm diameter and a 800 mm height. A bubble spurger with 100 µm to 160 µm bubble size was equipped at the bottom of the reactor. The columns were equipped with the instrumentation equipment such as corrosion-resistant stainless steel regulators and rotameter with glass columns and Teflon fittings. A schematic outline of the reactor is shown in Figure 1. Two identical columns were required for a continuous operation, one for brine oxidation and another for production of distilled gas.

The volume of brine in each test was 1.750 L. The solution was heated and stirred during the test using a magnetic stirrer equipped with a heater to keep the temperature constant and uniform inside the column. The pH of the brine was adjusted using sulfuric acid (Merck, 98%). Simultaneously, the brine temperature was increased to the temperature specified in the designed test. At this stage, the brine was prepared for bromine extraction and transferred to the reactor.

In the next step, the chlorine gas was injected into the reactor controlled by a rotameter. The duration of the chlorine gas injection into the system was 60 s, and then the bromine distillation process was performed. For this purpose, the temperature of the solution reached 60 °C using steam and at this constant temperature; compressed air bubbles were injected into the reactor. The liberated bromine collection was undertaken for 40 min. The remaining brine solution was analyzed in order to calculate the bromine extraction recovery. Figure 2 illustrates the assembled reactor including the oxidation column, bubbler, and condenser.

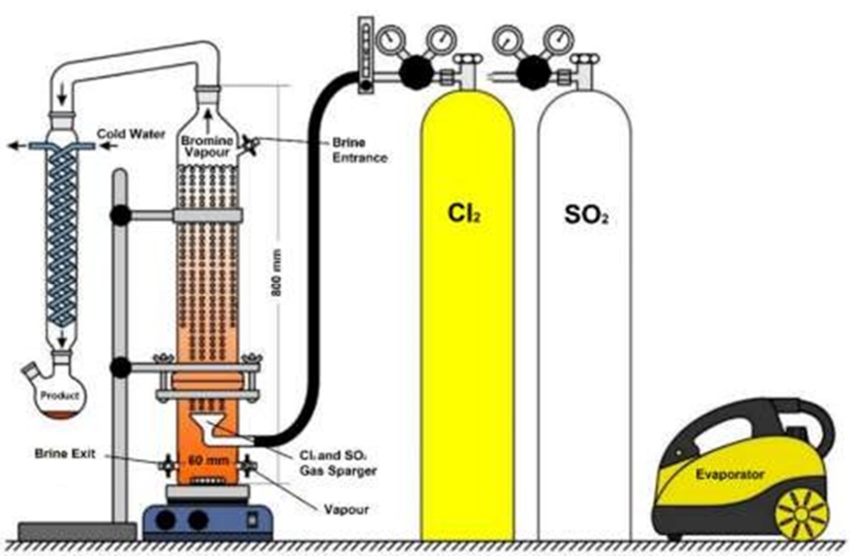

Figure 1. A schematic design of bromine extraction reactor.

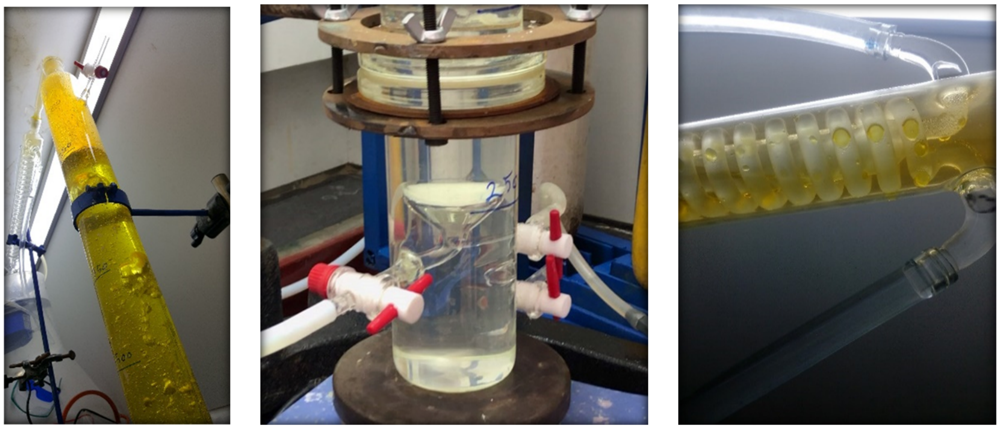

Figure 2. Assembled reactor including oxidation column, bubbler, and condenser showing the collected bromine gas.

In order to determine the effects of the operating parameters, the experiments were designated using the CCD method, by the Design Expert 11 software. The effects of three parameters including the chlorine gas injection rate, initial temperature, and pH of the feed brine were investigated in the following ranges (Table2). It has to be noted that the maximum and minimum of the parameters have been selected based on the initial experiments to define the appropriate ranges.

The difference between the bromine concentration in the feed and the remaining liquid after the bromine extraction was used in order to calculate the bromine recovery and the debromination efficiency. Bromine was quantified using ion chromatography (940 Professional IC Vario, Metrohm), as one of the of the best methods available for measuring bromide [24].

Table 2. CCD parameter ranges.

Parameter	Minimum	Maximum
Brine temperature (°C)	50	70
Brine pH	2	3
Chlorine gas injection rate (L/min)	1	2

3. Results and Discussion

The chemical analysis result of the seawater sample collected before and after pre-concentration by evaporation is shown in Table 3.

Table 3. Chemical analysis results of initial seawater sample.

Ion	Analysis method	Concentration (g/L)		Precipitation (%)
		Initial sample	After pre-concentration	
Ca^{2+}	Titration using EDTA[1]	0.19	0.16	78.9
Mg^{2+}	Titration using EDTA	25.90	64.73	37.5
Na^{+}	Flame photometer	74.69	37.50	87.4
K^{+}	Flame photometer	9.30	17.5	53.0
Cl^{-}	Titration using silver	177	213	69.9
SO_4^{2-}	Gravimetery	30.34	64.50	46.9
Br^{-}	Ion chromatography	1.172	3.1	33.9

After evaporation, the concentration of Br^- in the sample was increased to 3.1 g/L and used as the feed sample for the bromine extraction process. As the volume of the initial brine sample was quartered, the ion concentration before and after evaporation shows almost all the cations and anions precipitated to some degrees. Approximately 34% of bromide ions was also precipitated, which is required to be prevented in the further studies. The results of 20 experiments designated using the CCD method is presented in Table 4.

[1] Ethylene diamine tetra acetic acid

Table 4. Bromine extraction test results.

No.	pH	Temperature (°C)	Chlorine gas flow rate (L/min)	Br^- loss in the remaining brine (g/L)	Recovery (%)
1	2.0	50	2.0	0.337	88.4
2	3.0	50	1.0	1.056	63.4
3	3.0	70	1.0	0.965	66.4
4	2.5	60	1.5	0.176	93.8
5	2.5	60	2.5	0.185	93.5
6	2.5	80	1.5	0.432	85.6
7	2.5	40	1.5	0.378	86.3
8	2.5	60	1.5	0.420	85.6
9	3.0	70	2.0	0.277	90.5
10	2.5	60	0.5	1.760	39.7
11	2.5	60	1.5	0.794	81.7
12	2.5	60	1.5	0.754	74.0
13	2.5	60	1.5	0.313	89.3
14	3.0	50	2.0	0.351	87.9
15	3.5	60	1.5	0.655	77.9
16	2.0	70	2.0	0.630	78.7
17	2.5	60	1.5	0.397	86.6
18	2.0	50	1.0	0.877	69.8
19	1.5	60	1.5	0.770	73.8
20	2.0	70	1.0	0.884	70.5

The recovery was calculated based on the remaining Br^- ions in the brine, as follows (Equation 2):

$$R = \left(1 - \frac{Tt}{Ff}\right) \times 100 \tag{2}$$

where:

T: Amount of remaining brine (L)

t: Br^- concentration in the remaining brine (g/L)

F: 1.750 L

f: 3.1 g/L

In order to evaluate the correlation between the operating parameters and bromine recovery, the experimental results were statistically analyzed. The results of ANOVA (Table 5) indicate that in the fitted model, only the effect of the chlorine gas flow rate is known as significant, while the effects of two other parameters including pH and temperature are not significant in the investigated range. The fitted power model with Lambda of 1.24 was selected using the Box-Cox diagram. The model lack of fit for bromine recovery is insignificant so the recovery follows the model with a 95.61% probability. According to the ANOVA table, the model is as follows:

Table 5. ANOVA table of tests designed for recovery model.

Factor	Sum of squares	Degrees of freedom	Mean squares	F-value	p-value	
Model	32378.50	9	3597.61	6.21	0.0043	Significant
A: Temperature	20.70	1	20.70	0.0357	0.8539	
B: Chlorine gas flow rate	24709.21	1	24709.21	42.64	< 0.0001	
C: pH	67.67	1	67.67	0.1168	0.7396	
AB	175.91	1	175.91	0.3036	0.5938	
AC	335.25	1	335.25	0.5785	0.4644	
BC	720.00	1	720.00	1.24	0.2911	
A^2	74.83	1	74.83	0.1291	0.7268	
B^2	5041.50	1	5041.50	8.70	0.0145	
C^2	1342.87	1	1342.87	2.32	0.1589	
Residuals	5794.68	10	579.47			
Lack of fit	905.37	5	181.07	0.1852	0.9561	Not significant
Pure error	4889.31	5	977.86			
Corrected Total	38173.18	19				

$$R = \sqrt[1/24]{X} \tag{3}$$

$$X = 123.733 + 4.013\,A + 209.92\,B + 15.674\,C + 0.937\,A.B + 1.294\,A.C + 37.947\,B.C + 0.0172\,A^2 + 56.641\,B^2 + 29.232\,C^2 \tag{4}$$

As shown in Figure 3, there are some differences between the actual and estimated values by the model. Thus in order to ensure the correctness of the experimental tests and the absence of systematic errors, the distribution of these residual was depicted *vs.* probability %. An excellent correlation between the residual amounts and the normal distribution confirms the insignificant lack of fit and probability % (Figure 4).

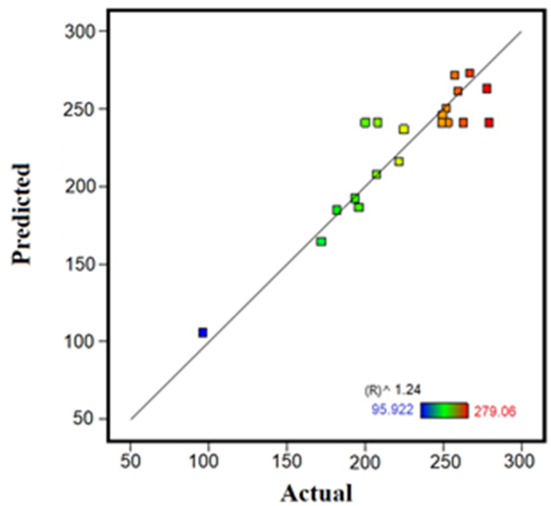

Figure 3. Experimental results *vs.* model estimated recovery.

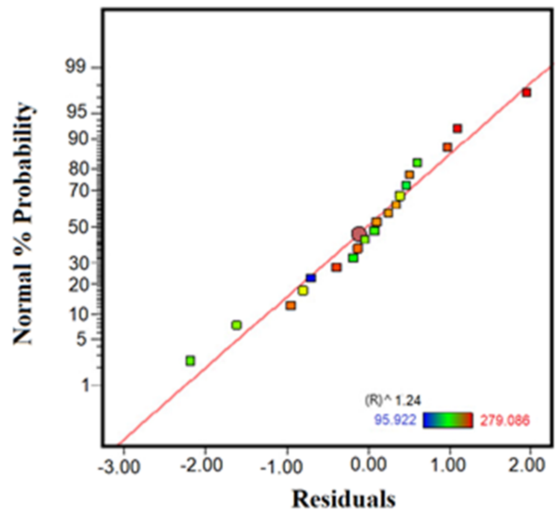

Figure 4. Residual s *vs.* normal distribution.

In order to investigate the role of the operating factors on the bromine extraction rate, the studied parameters are depicted in Figures 5, 7, and 8. Figure 5 shows that the bromine recovery is slightly reduced by increasing pH from 2 to 3, especially in the lower chlorine gas flow rates. A maximum bromine recovery around 2.4-2.6 can be distinguished, which is slightly lower than the reported values [21]. In the high chlorine gas flow rate, pH seems to be ineffective.

In order to better understand the effect observed in the pH graph, the pourbiax diagram of chlorine and bromine was generated using the concentration in the collected sample of this work, as shown in Figure 6.

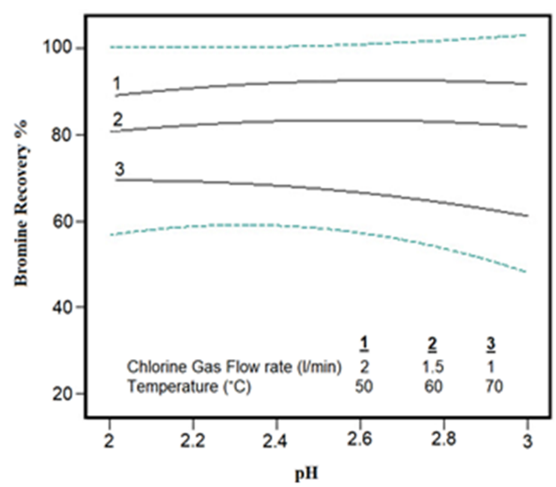

Figure 5. Bromine recovery % *vs.* pH in different amounts of chlorine gas flow rates and temperature.

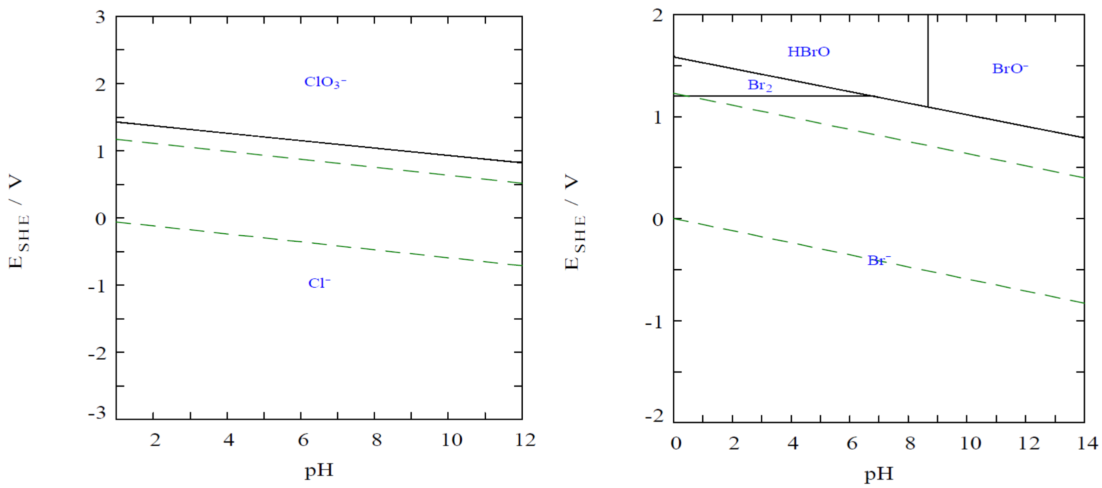

Figure 6. Pourbiax diagram of a) chlorine in 6.17 M and b) bromine in 16.20 mM at 25 ˚C.

The brine is neutral to alkaline (pH = 7-8), and if the pH is not decreased before oxidation of bromine, both the injected chlorine gas and the produced bromine can be easily hydrolyzed as depicted in Figure 5 [21]:

$$Cl_2 + H_2O = H^+ + Cl^- + HClO \quad (5)$$

$$Br_2 + H_2O = H^+ + Br^- + HBrO \quad (6)$$

$$3Br_2 + 3H_2O = 6H^+ + 5Br^- + BrO_3 \quad (7)$$

If the concentration of H^+ is high enough, then hydrolysis of bromine can be inhibited, which is also the purpose of acidification. The higher the bromine recovery by decreasing pH is the result of prevention of chlorine and bromine gas hydrolysis, which has been also been reported before [21]. When the pH of the brine is further reduced, the oxidation rate does not seem to be improved greatly, and even a slight drop can be observed. This effect may be attributed to the high concentration of H^+ that screen the bromide ions and limit the access of chlorine gas. Thus it is essential to control the pH value of the oxidation liquid precisely. However, this effect can be overcome by a higher chlorine gas flow rate. A higher temperature can also prevent reduction of recovery in a higher pH (Figure 7).

As mentioned earlier, the main role playing factor in the bromine recovery is the amount of chlorine gas flow rate used for oxidation of bromide to bromine. It can be depicted in Figure 8 that the recovery increases significantly with the amplification of chlorine flow rate. However, the noteworthy point in this diagram is when the pH of the diagram decreases from 3 to 2. The improving effect of the chlorine gas flow rate can be significantly limited at a lower pH.

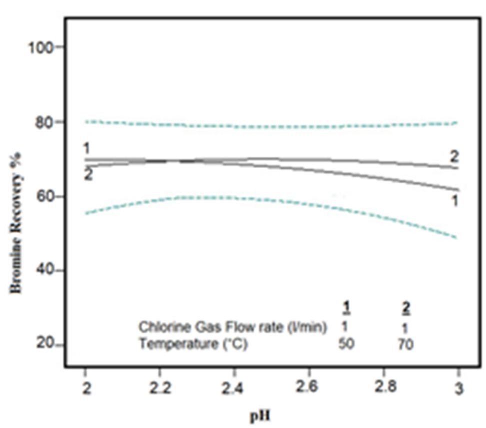

Figure 7. Bromine recovery % *vs.* pH in different temperatures.

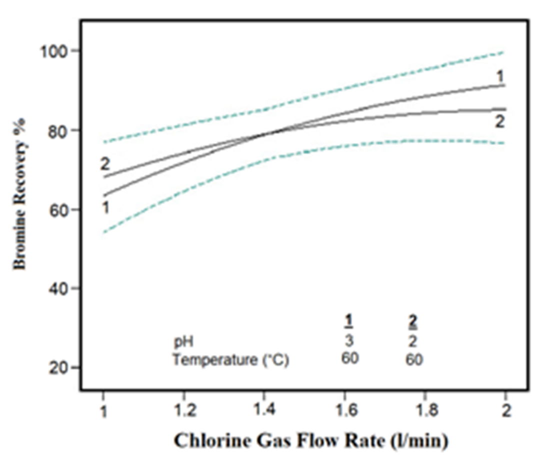

Figure 7. Effects of chlorine gas measures on recovering bromine extraction.

If the amount of chlorine flow rate is not sufficient for the oxidation of bromine, the Br^- ions cannot be oxidized to bromine, and the recovery drops. As observed earlier, when the concentration of H^+ is too high, the chlorine gas flow rate cannot be as effective as in a higher pH. Thus the control of both pH and chlorine gas flow rate plays a crucial role in the bromine extraction optimization.

4. Conclusions

Analysis of the bromide content of the Chabahr bay near the Konarak desalination plant discharge with a comparable bromine concentration in the Dead sea provides the urge for utilization of the concentrated seawater in order to protect the environment as well as the production of valuable products such as bromine. The air-blow out technique seems to be an appropriate method for bromine extraction from this resource.

The pre-concentration using the low-cost evaporation method can effectively increase the bromine concentration, However, it has to be carefully controlled in order to prevent the precipitation of bromide with other salts. The CCD experimental results show that among the three investigated operating factors, the chlorine gas flow rate, pH, and temperature are the most effective parameters, respectively. If the amount of chlorine is not sufficient for the oxidation of bromine, the Br^- ions cannot be oxidized, and the recovery is linearly reduced. The positive effect of the high chlorine gas flow rate is limited at a lower pH. Thus the control of both the pH and the chlorine gas flow rate plays a crucial role in the bromine extraction optimization. The results obtained shed light on the oxidation of bromine by chlorine gas, and could be effectively used in the optimization of air blow out techniques via operating at optimum operating conditions.

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اثر پارامترهای عملیاتی در استحصال برم از پساب کارخانه‌های آب شیرین کن

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چکیده:

توسعه صنعت آب‌شیرین‌کنی به عنوان راه‌حل کلیدی تأمین آب شیرین در دنیا، مستلزم بهره‌برداری کامل از پساب کارخانه‌های آب شیرین‌کنی است. برم‌زدایی از پساب کارخانه‌های آب شیرین‌کن علاوه بر استحصال محصول برم اهدافی هستند که در بسیاری از مطالعات تحقیقی و عملیات‌های صنعتی دنبال شده‌اند. در این تحقیق، استحصال برم از پساب کارخانه آب‌شیرین‌کن کنارک، در خلیج چابهار ایران به روش آزمایشگاهی مورد مطالعه قرار گرفته است. برای این منظور، با استفاده از روش دمش هوا اثر پارامترهای عملیاتی شامل دما، pH و شدت جریان گاز کلر بررسی شده است. پارامترهای عملیاتی بهینه شده و روندهای مشاهده شده مورد بحث و بررسی قرار گرفته است. عیار برم نمونه جمع‌آوری شده از منطقه پزم تیاب نزدیک به محل تخلیه پساب کارخانه، به روش کروماتوگرافی یونی، ۱/۱۷۲ گرم بر لیتر تعیین شده است. نمونه با استفاده از روش تبخیری برای افزایش غلظت تا ۳/۱۰۰ گرم بر لیتر پرعیار شده است. راکتوری با ابعاد ۶۰ میلی‌متر در ۸۰۰ میلی‌متر جهت انجام مطالعات طراحی و ساخته شده است. به منظور بررسی پارامترهای عملیاتی، روش طراحی آزمایشات ترکیب مرکزی (CCD) مورد استفاده قرار گرفته است. در میان پارامترهای مورد مطالعه تنها شدت جریان گاز کلر اثر قابل‌توجهی داشته و اثر دو پارامتر دیگر در محدوده pH برابر ۱/۵ تا ۲ و دمای ۵۰ تا ۷۰ درجه سانتی‌گراد قابل‌چشم‌پوشی بوده است. در سه شدت جریان ۱، ۱/۵ و ۲ لیتر بر دقیقه گاز کلر میزان تولید برم تقریباً به صورت خطی با افزایش می‌یابد. گاز برم با حداکثر میزان بازیابی ۹۳/۸ درصد و افت برم ۱۸۵ میلی گرم در لیتر استحصال شده است. در این شرایط پارامترهای عملیاتی بهینه pH برابر ۲/۵، دمای ۶۰ درجه سانتی‌گراد و نرخ گاز کلر ۱/۵ لیتر بر دقیقه بوده است.

کلمات کلیدی: برم، دمش هوا، پساب کارخانهآب شیرین کنی.