



Ion Exchange Method for Sulphate Removal from Treated Wastewater of Al-Doura Refinery

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طريقة التبادل الأيوني لإزالة الكبريتات من المياه المعالجة لمصفاى الدورة

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Abstract

The treated wastewater by industrial Wastewater Treatment Plant (WWTP) which commonly named (INGECO) in Al- Doura refinery suffers from highly sulphate concentrations that exceeding the EPA specified limits of (250 mg/L). Therefore, laboratory water quality historical data have been reviewed and analyzed to determine annual rate, maximum and peak sulphate concentrations that found to be (360 mg/L), (425 mg/L) and (550 mg/L) respectively, and by field inspection which has been done to specify the reasons of highly sulphate concentrations appeared in wastewater of most refinery process units, found that the maximum required sulphate remaining concentration should be not more than (10 mg/L) to use in energy units.

In this study, about (78) samples have been taken from industrial wastewater and prepared for average, maximum and peak sulphate concentrations to use in ion exchange method by using strong base anion (Amberlite IRA402 OH) resin. Results obtained by using strong base anion resin refer to the optimum (dosage, mixing time and mixing speed) to use in sulphate removal for reuse purpose were (85 g/L, 30 min and 83 rpm), (70 g/L, 16 min and 75 rpm) and (100 g/L, 17 min and 80 rpm) for each of average, maximum and peak concentrations respectively. While for disposal purpose, were (0.56 g/L, 16 min and 100 rpm), (5.8 g/L, 10 min and 88 rpm), (8 g/L, 12 min and 70 rpm) respectively. The advantage of this method was achieving of highly sulphate removal, and inexpensive in treatment cost about (0.07 \$US/m³) and (0.0007 \$ US/m³) for reuse and disposal. The most important disadvantages are product saline solution so that it requires the disposal of additional costs.

Keywords: Wastewater Treatment Plant (WWTP) ; Industrial Wastewater ; Sulphate Conc. and Al-Doura refinery.

المستخلص

تعانى المياه المعالجة بواسطة محطة معالجة المياه الصرف الصناعية التي تسمى بالانجيكو في مصفى الدورة، من ظهور تراكيز عالية تتجاوز المعايير المحددة من قبل وزارة البيئية العراقية للطرح للانهار والتي يجب ان لا تتجاوز (250mg/L). لذلك، تم تحليل البيانات المخبرية ليجاد المعدل السنوي، الاقصى وذروة تراكيز الكبريتات في المياه المطروحة الى النهر، حيث وجد بانها (360mg/L)، (425mg/L)، (550mg/L) على التوالي. وقد اظهرت نتائج التحري الميداني التي اجريت لمعظم وحدات عمليات الانتاج للمصفاى بأن الحد الأقصى المسموح به لنوعية المياه المستخدمة يجب ان لا يتجاوز (10mg/L).

في هذه الدراسة، تم اخذ (78) عينة من مياه الصرف الصناعي واعداد معدل واقصى وذروة تراكيز الكبريتات ليتم استخدامها في عملية التبادل الأيوني بواسطة راتنج شديد القاعدية لازالة الايونات السالبة اسمه التجاري (Amberlite.IRA402 OH)، وقد اظهرت نتائج استخدام راتنج شديد القاعدية لازالة الايونات السالبة بأن أمثل (جرعة، وقت خلط وسرعة خلط) للأزالة الكبريتات من المياه المعالجة الصناعية لأجل إعادة استخدامها وفقاً لمعدل واقصى وذروة تركيز هي (90g/L ، 22.5min)، و(83rpm) و(70g/L)، (15min ، 70rpm) و(100g/L ، 17min)، و(80rpm) على التوالي. اما لغرض طرحها الى النهر فقد كانت (0.7g/L ، 15min)، و(100rpm)، (5.8g/L ، 11min)، و(90rpm)، و(8.0g/L ، 14min)، و(70rpm) عند نفس معدل، اقصى وذروة التراكيز للكبريتات على التوالي. حيث انه من اهم فوائد هذه الطريقة هي إنتاج مياه بتراكيز قليلة او خالية من الكبريتات، وبكلفة معالجة رخيصة تقدر بحوالي ($0.07\text{US}/\text{m}^3$) في حالة اعادة استخدام المياه وحوالي ($0.0007\text{US}/\text{m}^3$) في حالة طرح المياه للنهر. اما من اهم مساوئها هي تخليفها لمحلول ملحي يتطلب التخلص منه تكاليف إضافية.

الكلمات المفتاحية: محطات معالجة المياه، مياه الصرف الصناعي، تراكيز عالية

من الكبريتات ومصفاى الدورة



Introduction

Sulphate is a naturally occurring substance that contains sulphur and oxygen. It is present in various mineral salts that are found in soil and rocks sulphate forms salts with a variety of elements including barium, calcium, magnesium, potassium and sodium. Sulphate may be leached into water from the soil and is commonly found in most water supplies. Magnesium, potassium and sodium sulphate salts are all soluble in water, calcium and barium sulphate are not very easily dissolved in water. There are several other sources of sulphate in water, decaying plant and animal matter may release sulphate into water, numerous chemical products including ammonium sulphate fertilizers contain sulphate in a variety of forms, the treatment of water with aluminum sulphate (alum) or copper sulphate also introduces sulphate into a water supply. Human activities such as the combustion of fossil fuels and sour gas processing release sulphur oxides to the atmosphere, can give rise to sulfuric acid in rainwater (acid rain) which in turn results in the return of sulphate to surface waters in the environment, as well as the source of sulphate in the water resulting from the addition of sulfuric acid for the purpose ion exchange resin regeneration [7 and 8]. Although sulphate is non-toxic, except at very high concentrations, it exerts a purgative effect:

1. Precipitation of sulphate can cause damage to equipment through the formation of calcium sulphate scale [11].
2. At high concentrations of precipitation sulphate may affect the efficiency of many industrial processes. The corrosive effect of high sulphate waters, particularly towards concretes, is increasingly becoming a major water quality problem for mining operations [10].
3. Sulphate, especially precipitation of gypsum, may impair the quality of treated water. In many arid environments gypsum becomes the dominant contributor to salinity in the vicinity of the discharge [13].



4. People consuming drinking water containing sulphate in concentrations exceeding 500 mg/L commonly experience cathartic effects, resulting in purgation of the alimentary canal [14]. Dehydration has also been reported as a common side effect following the ingestion of large amounts of sulphate.
5. Saline water can lead to the salinization of irrigated soils, diminished crop yield and changes in biotic communities [12].

EPA [16] determined sulfate in drinking water currently has a secondary maximum contaminant level (SMCL) of (250 mg/L), based on aesthetic effects (i.e., taste and odor). This regulation was adopted by Iraqi Ministry of Environment (MOE) as enforceable standard for effluent disposal to class (A) streams which was taken as limitation in this study.

Sulphonate Removal Techniques

Literature studies were conducted to investigate the different generally available techniques to remove sulphate from industrial wastewater. These techniques can be divided into physical processes such as membrane filtration, chemical treatment such as precipitation technique and biological sulphate reduction [3, 2, 1, 5, 6 and 9].

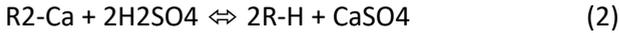
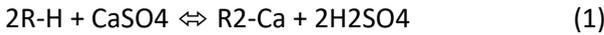
- Physical Sulphate Removal Techniques:
 - o Membrane filtration such as RO (reverse osmosis), SRO (Seeded Reverse Osmosis), SPARRO (Slurry Precipitation and Recycle Reverse Osmosis), ED (Electro Dialysis) and EDR (Electro Dialysis Reversal).
 - o Ion exchange
- Chemical Precipitation Sulphate Removal Techniques:
 - o Gypsum precipitation.
 - o Ettringite precipitation such as SAVMIN and CESR (Cost Effective Sulphate Removal).
 - o Barite (barium sulphate) formation.

- Biological Sulphate Removal Techniques:
 - o Bioreactors.
 - o Constructed wetlands.

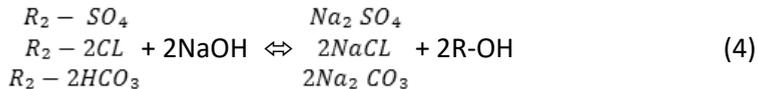
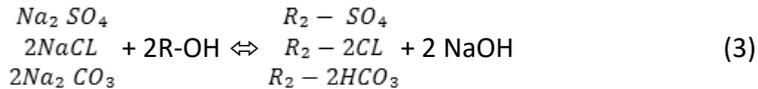
In this study, ion exchange method by using strong base anion (Amberlite IRA402 OH) resin has used to remove sulphate from treated wastewater.

Ion Exchange

The untreated wastewater is pumped into the cation loading section where it passes through fluidized contact stages. Calcium ions and other cations are removed from the feed water through cation-exchange with strong acid cation resin (R-H). This is demonstrated in reaction equation (1) and regeneration in equation (2).



After the wastewater has flown through the cation resin contactor, the water is pumped to a degassing tower to remove carbonate alkalinity. Next the water is pumped into the anion loading section where it passes through fluidized contact stages. Anions such as sulphate ions are then removed from the wastewater through anion-exchange with a strong base anion resin (R-OH). This is shown in reaction equation (3) and regeneration in equation (4). The treated water has a neutral pH, and is also low in dissolved calcium, sulphate and other dissolved substances including metals [15, 4 and 19].





Materials and Methods

After inspection of water quality used in various refinery processes which showed in Table (1), it is found that the main highly concentration of sulphate in wastewater reach about (2900 mg/L) coming from blow down water of energy units. But when it reach the WWTP, the concentration reduced to less than (600 mg/L) by using ion exchange process, then decreased to average level when mixed with other lesser concentration wastewater into WWTP influent collection basin. There wasn't any specific process to control or remove sulphate to reach the effluent disposal limit in WWTP. Laboratory test results for five years (2008 to 2013) have been analysis as shown in Fig (A) to determine annual rate and maximum sulphate concentration which found to be (360 mg/L) and (425 mg/L) respectively. Also, peak concentration which possible to reach WWTP for more than (15) days per year considered to be (550 mg/L).

Jar test device used to get optimum concentration, time and mixing speed used to remove the sulphate in the water. In the case sulphate removal and reuse of water, at first change dosage Anion resin strong added to water and has proven time (1hr) and mixing speed (120 rpm) and thus get the best dosage used to remove sulphate Secondly proved dosage added in the water and mixing speed and change a time (1/4, 1/2, 1) hr to get a better time to remove sulphate, after that proved dosage added in the water and time and change a mixing speed (80, 100, 120) rpm to get a better mixing speed to remove sulphate but in case sulphate reduce to reach the acceptable range to subtract (not more than 250 ppm), at first change dosage Anion resin strong added to water and has proven time (1/2 hr) and mixing speed (120 rpm) and thus get the best dosage used to remove sulphate Secondly proved dosage added in the water and mixing speed and change a time (1/4, 1/2) hr to get a better time to remove sulphate, after that proved dosage added in the water and time and change a mixing speed (80, 100, 120) rpm to get a better mixing speed to remove sulphate.

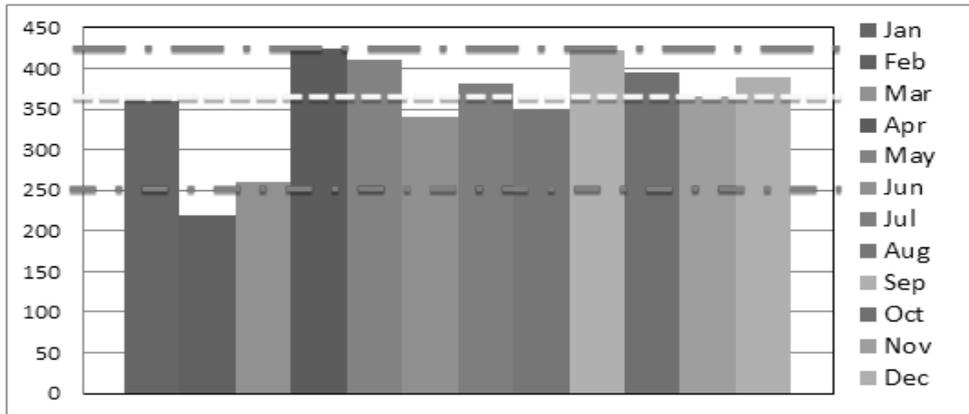


Figure (A): Mean monthly rate of treated water sulphate concentrations

Table (1): Sulphate concentration in various refinery processes

	SO ₄ (mg/L)	
	Influent	Effluent
sedimentation basins	(190 – 320)	283
After DMF and ACF		272
After storage tanks when adding chemical substances		300
RO unit	300	(4 – 6)
Water resulting from RO unit (reject)		980
Energy unit – 1	15	15
Energy unit – 1 (reject)		1000
Energy unit – 2 and energy unit – 3	15	0
Energy unit – 2 and energy unit – 3 (reject)		2900
Chiki units (one and two)		120
Hydrogenation units (one and two)		530
Grease unit – 1		155
Grease unit – 2		185
Grease unit – 3		270
	300	360



Results and Discussion

The ion exchange process is considered one of the more effective method to remove dissolved solids from water, because (in this study) using a Strong Base Anion (SBA) resin in the hydroxide form that remove all anionic contaminants present such as (SO_4^{2-}). For this reason, it was tried to use SBA for sulphate removing from refinery treated water for each purposes of reuse by considering (60 min) retention time and disposal by considering (30 min) retention time.

- (24) treated water samples of (500 ml) each contents (360 mg/L) SO_4 concentration have been prepared to use in jar test device to obtain optimum design parameters, (12) samples for reuse criteria and (12) samples for disposal criteria. Test results and analysis that obtained from each run are discussed below:

* By fixing Mixing Speed (N) to be (120 rpm), and with variable Mixing Time (tmix) of (15, 30 and 60 min) and (SBA) dosages of (300, 200 and 100 g/L), the results obtained were itemized and showed in Figs. (1, 2, 3, 7, 8, 9, 13, 14 and 15). The SO_4 curve of Figs. (1, 7 and 13) illustrates that at (15 min) retention time more than (85%) of SO_4 have been removed, and at (60 min) all sulphate entirely removed. While TDS and Cl curves had the same behavior exponential increasing until reached (15 min) then tends to linearly with relative increasing until reached the end point. This caused by the fact that this type of resin (Amberlite IRA402 – OH) used was removing all anion present in water in according to its highly valence and atomic number, in which the elements of high valence removed before the other elements of low valence. Therefore, sulphate (SO_4^{-2}) was removed first before the chlorides (Cl^-) in a typical series of removal as follows:



$F^- \approx OH^- < HCO_3^- < CH_3COO^- < Cl^- < HSO_3^- < Br^- < NO_3^- < SCN^- < I^- < ClO_4^-$
 $< CrO_4^{2-} < C_2O_4^{2-} < SO_4^{2-}$. While when it was equal in valence number the elements of higher atomic number has priority in removal than the others. As shown in equation (3) perversely, the increasing of NaOH in water led to rising of pH value. This substance (NaOH) used to regeneration of this type of SBA resin, so that this process was releasing the elements of low valence (chlorides) in the water which interact with existing sodium in water and led to increasing of TDS and chloride concentrations as clearly appeared in these and all other next ion exchange Figs. (1, 4, 7, 10, 13, 16, 19, 22, 31, 34, 37, 40, 43, 46, 49, 52, 55, 64, 67, 70, 73, 76, 79, 82, 85 and 88) of average, maximum and peak SO_4 concentrations with variable: SBA dosages, tmix and N for reuse or disposal purposes, so that it would not discussed more because all of them have the same reasons. Removal efficiencies to reach specified SO_4 reuse concentration of (10 mg/L) are illustrated in Figs. (3,9 and 15) by using variable mixing times and SBA dosage, the optimum mixing time to remove sulphate concentration form (360mg/L) to (10 mg/L) for reuse of treated water were (19.0, 22.5 and 30.0 min) respectively. While on the other hand, Figs. (2, 8 and 14) refer to increase of pH values with decreasing of temperature values to reach maximum value of (9, 9 and 9.1) in (22, 22.2 and 22.2 °C) at end point of (60 min), the increasing in SBA concentrations led to increase pH due to chemical reaction of (Amberlite IRA402 OH) with Na_2SO_4 which was shown in equations (3) before, which led to an increase NaOH in water that increase pH, the lesser selected dosage of (100 g/L) was sufficient to remove sulphate in (30 min) as mentioned



before, it was preliminary specified to be recommended dosage for reuse purpose.

- * While in the same N of (120 rpm) and Mixing Time (t_{mix}) of (15, 30 and 60 min), by using (SBA) dosages of (0.2, 0.5 and 1 g/L), the obtained results were shown in Figs. (4, 5, 6, 10, 11, 12, 16, 17 and 18). Figs. (4, 10 and 16) illustrates that when SBA dosage increased SO_4 concentrations proceeding in decreased with times to reach (295, 260 and 215 mg/L) at specified (30 min) for disposal. While at the same time with these small dosage it can be observed that Cl and TDS concentrations tended to decrease due to the initial reaction of resin with all presence anions in solutions in according to equations (3) that shown before. This led to slightly decreasing on pH values and slightly rising in temperature as shown in Figs.(5, 11 and 17). Figs. (6, 12 and 18) illustrates the SO_4 removal efficiency curve to the specified concentration for disposal. The optimum mixing time to reduce sulphate concentration form (360 mg/L) to (250 mg/L) was found to be (48.0, 49.0 and 16.0 min) respectively. Preliminary, the dosage of (1.0 g/L) was specified to be recommended dosage for disposal purpose.
- * Another test was done by fixing SBA dosage and t_{mix} to be (100 g/L) and (60 min) respectively, and with variable N to be (80, 100 and 120 rpm). The results obtained were shown in Figs. (19, 20 and 21). From SO_4 curve in Fig (19) it was found that when N reach (100 rpm) chloride exponential increased from (520 mg/L) to reach (800 mg/L) at (120 rpm) with increasing in TDS, and SO_4 dropped to (20 mg/L) in (80 rpm). pH increased to (9.0) with slightly drop in temperature as



shown in Fig (20). Figure (21) illustrates SO_4 removal efficiency curve, the optimum mixing speed for (100%) sulphate removal efficiency for reuse purpose was found to be (83 rpm).

- * Fixing SBA dosage and t_{mix} to be (1.0 g/L) and (30 min) respectively, with variable N of (80, 100 and 120), the results obtained showed in Figs. (22, 23 and 24). From SO_4 curve in Fig (22), it was found that the SO_4 concentrations led to decrease until reached (250 mg/L) at (100 rpm) with slightly acidic solution as shown in Fig (23). (100 %) removal efficiency for disposal purpose could be obtained by using (100 rpm) as appeared from Fig (24).
- * All data obtained from ion exchange testes that mentioned above of specified retention time (60 and 30 min) for reuse and disposal respectively, and mixing speed of (120 rpm), have been collected and analyzed with Figs. (25, 26, 27, 28, 29 and 30) for decision making about optimum dosages used for the overall viewing to be a laboratory reference of process operation. As shown in Fig (25a and b), the sulphate concentrations tend to drop to (220 mg/L) at (0.2 g/L) dosage then return to rise to reach (245 mg/L) at (0.5g/L) then linear decreasing with more dosage to reach (0.0 mg/L) at (100g/L) dosage. This indicated that with the same conditions, it couldn't consider that there was a positive relationship between SBA resin dosages and removal of sulphate, particularly when using smaller dosages, and recommending for further studies for knowing sulphate behavior in the lag dosage intervals located between (1.0 to 100 g/L) by using same procedure directed in this study. As in this case, it can considered (85 g/L) is an optimum dosage for reuse and recommended for



(0.19g/L) dosage for disposal because it located in small dosage interval as shown in Fig. 25b. On the other hand, Figs. (28a and 28b) sulphate remaining curve was descent linearly when retention time was (30 min) until reaching (215 mg/L) when dosage = (1.0 g/L) then continue in reducing until reach (10 mg/L) at (100g/L) dosage and still remaining in same concentration when dosage increased to (200g/L) the slightly reduced to (5.0 g/L) when at the end point, so that it can be considered that remained constant with any more dosages because of lag in time or mixing speed. As in this case, the optimum dosage found to be (100 g/L) for reuse and (0.58g/L) for disposal. Fig(26a and 26b) indicates that TDS and chloride tends to decrease until reach (1135 mg/L) of TDS at (0.5 g/L) and (244 mg/L) of chloride at (0.2 g/L) dosage, then continues increasing. Fig. 29A and 29B shown the same behavior of previous Figures of (60 min) retention time, this indicates that time effecting just on reaction rate not on behavior. It can he concluded from other Figs. (27A, 27B, 30A and 30B) that pH tends to be acidic in smaller dosages and alkaline with higher dosages of SBA resin.

- (27) treated water samples of (500 ml) each contents (425 mg/L) SO_4 concentration have been prepared to use in jar test device to obtain optimum design parameters, (15) samples for reuse criteria and (12) samples for disposal criteria. Test results and analysis that obtained from the each run are discussed below:
 - * By fixing Mixing Speed (N) to be (120 rpm), variable Mixing Time (tmix) of (15, 30 and 60 min) and (SBA) dosages of (20, 30, 40 and 100g/L) have been run in the device. Test results obtained were



itemized and showed in Figures (31, 32, 33, 37, 38, 39, 43, 44, 45, 49, 50 and 51) with times (15, 30 and 60 min). Figures (31, 37, 43 and 49) illustrates the increasing in SBA concentrations led to decrease SO_4 concentrations to reached (6 mg/L) which is led to remove (98.6 %) of sulphate. So that, the optimum dosage of SBA to remove sulphate (6mg/L) for reuse of treated water in refinery process was (100 g/L). Figures (32, 38, 44 and 50) illustrates that pH tended to increase with increasing dosages to be an alkaline solution with slightly decreasing in temperature. Figures (33, 39, 45) illustrated that the removal efficiency insufficient for sulphate removal for reuse while as appeared in figure (51) the optimum mixing time for maximum sulphate concentration is (16 min).

- * Fixing Mixing Speed (N) to be (120 rpm) and variable Mixing Time (tmix) and SBA dosages as shown in Figures (34, 35, 36), (40, 41, 42) and (46, 47, 48) with time (15, 30 and 60 min). Figures (34,40 and 46) illustrates the increasing in SBA concentrations led to decrease SO_4 concentrations, the optimum dosage of SBA was (6 g/L) to reduce sulphate concentration form (425 mg/L) to less than (250 mg/L), while on the other hand increased chlorides concentration and TDS. Figures (35, 41 and 47) shows a pH and temperature behavior curve, temperature slightly was rising with slightly decrease in pH values. Figures (36, 42 and 48) illustrates SO_4 removal efficiency curve by using variable mixing times for disposal of treated water, it was found from figure (36) the sulphate removal efficiency was insufficient, while form figures (42 and 48) the optimum mixing time to remove sulphate for disposal purpose was (10 and 7.5 min) respectively.

- * Reuse purpose test with variable mixing speed to be (80, 100 and 120rpm) and by fixing SBA dosage and t_{mix} to be (100 g/L) and (1 hr) respectively. From Figures (52, 53 and 54) it was found that SO_4 reached (5 mg/L) in (80 rpm), then slightly increase to (6 mg/L) when $N = (100 \text{ and } 120 \text{ rpm})$. it was found from SO_4 curve that the maximum removal have been occurred at $N = (80 \text{ rpm})$ of (98.8 %) removal efficiency, while increasing in chlorides concentration and TDS. Figure (53) illustrates the pH and temperature behavior curves, it was appeared that temperature tended to reduce with increasing of mixing speed in alkaline solution. Figure (54) illustrates SO_4 removal efficiency curve by using variable mixing speeds for reuse of treated water, the optimum mixing speed to remove sulphate concentration form (425mg/L) to (10 mg/L) for reuse of treated water was (75 rpm).
- * While by fixing SBA dosage and t_{mix} to be (6 g/L) and (30 min) respectively, and with variable N of (80, 100 and 120 rpm) as shown in Figures (55, 56 and 57). Figure (55) revealed from SO_4 curve that the concentrations SO_4 lead to decrease until reached (< 250 mg/L) at (100 rpm) of mixing speed, while increasing in chlorides concentration and TDS. Figure (56) illustrates pH curve which indicated that solution tend to be slightly acidic with slightly increasing in temperature. Figure (57) illustrates SO_4 removal efficiency curve by using variable mixing speeds for disposal of treated water the optimum mixing speed to reduce sulphate concentration form (425 mg/L) to (250mg/L) for disposal of treated water was (88 rpm).
- * All data obtained from ion exchange testes that mentioned above of specified retention time (60 and 30 min) for reuse and disposal



respectively, and mixing speed of (120 rpm), have been collected and analyzed. Figures (58, 59, 60, 61, 62 and 63) for decision making of about optimum dosages used for the overall viewing to be a laboratory reference of process operation. As showed in Figures (58 and 61), when retention time was (60 min) the sulphate concentrations tended to drop until reaching (175 mg/L) at (6 g/L) dosage then continue in reducing until reach (6 mg/L) at (100 g/L) dosage. As in this case, it can be considered that (70 g/L) was an optimum dosage for reuse and recommended for (4.5 g/L) dosage for disposal. On the other hand, when retention time was (30 min) the sulphate concentrations tended to drop to (50 mg/L) at (10 g/L) dosage then return to rise to reach (86 mg/L) at (20 g/L) then linear decreasing with more dosage to reach (7 mg/L) at (100 g/L) dosage. As in this case, the optimum dosage found to be (90 g/L) for reuse and (5.8 g/L) for disposal. Figures (59) indicate that TDS and chloride tended to increase until reaching (2076 mg/L) of TDS at (2g/L) and (920 mg/L) of chloride at (10 g/L) , then decreased to reach (1993mg/L) of TDS at (6 g/L) and (700 mg/L) of chloride at (20 g/L) then chloride continue increasing but TDS increased until reach (2459 mg/L) of TDS at (40g/L), then continue decreased. Figures (62) shows the same behavior of previous figures of (60 min) retention time in chloride but TDS tend to increase until reach (2412 mg/L) of TDS at (10 g/L) , then decreased reach (2023 mg/L) at (20 g/L) of TDS then continues increasing. It can be concluded from other figures of (60 and 63) that pH tended to be acidic in smaller dosages and alkaline with higher dosages of SBA resin.

- (27) treated water samples of (500 ml) each contents (550 mg/L) SO_4 concentration have been prepared to use in jar test device to obtain optimum design parameters, (15) samples for reuse criteria and (12) samples for disposal criteria. Test results and analysis that obtained from each run are discussed below:
 - * By fixing Mixing Speed (N) to be (120 rpm), variable Mixing Time (tmix) of (15, 30 and 60 min) and (SBA) dosages of (20, 30, 40 and 100g/L) have been run in the device. Test results obtained were itemized and showed in Figures (64, 65, 66, 70, 71, 72, 76, 77, 78, 82, 83 and 84) with times (15, 30 and 60 min). Figures (64, 70, 76 and 82) illustrates the increasing in SBA concentrations led to decrease SO_4 concentrations to reached (11 mg/L) which is led to remove (99.0 %) of sulphate. So that, the optimum dosage of SBA to remove sulphate for ruse of treated water in refinery process was (100 g/L). While on the other hand increasing chlorides concentration and TDS. A figure (65, 71, 77 and 83) illustrates that pH tended to increase with increasing dosages to be an alkaline solution with slightly decreasing in temperature. Figures (66, 72, 78 and 84) as illustrates SO_4 removal efficiency curve by using variable mixing times for reuse of treated water, it was found that from Figures (66, 72 and 78) the removal efficiencies were insufficient, while from figure (84) the optimum mixing time for peak removal is (17 min).
 - * Fixing Mixing Speed (N) to be (120 rpm) and variable Mixing Time (tmix) and SBA dosages as shown in Figures (67, 68, 69), (73, 74, 75) and (79, 80, 81) with time (15, 30 and 60 min). Figures (67, 73 and 79) illustrates the increasing in SBA concentrations led to decrease SO_4



concentrations, the optimum dosage of SBA was (10 g/L) to reduce sulphate concentration form (550 mg/L) to less than (250 mg/L), while on the other hand increased chlorides concentration and TDS. Figures (68, 74 and 80) illustrates pH and temperature behavior curves, it was appeared that temperature tend to increase with increasing of SBA dosage in acidic solution. Figures (69, 75 and 81) illustrates SO_4 removal efficiency curve by using variable mixing times for disposal of treated water, it was found that the figures (69 and 75) not sufficient to remove sulphate and disposal water while figure (81) is sufficient the optimum mixing time to remove sulphate concentration form (550 mg/L) to (250 mg/L) for disposal of treated water was (12 min).

- * Fixing SBA dosage and t_{mix} to be (100 g/L) and (1 hr) respectively, and variable N (80, 100 and 120) as shown in figures (85, 86 and 87). Figure (85) shows from SO_4 curve that the maximum removal have been occurred at N = (120 rpm) of (98 %) removal efficiency, while increasing in chlorides concentration and TDS. Figure (86) illustrates pH and temperature behavior curves, it was appeared that temperature tend to reduce with increasing of mixing speed in alkaline solution. It couldn't obtained (100%) SO_4 removal efficiency by using variable mixing speeds for reuse of treated water as appeared from Figure (87) total removal efficiency within this criteria could be reached is (98.18%) by using (80 rpm).
- * Fixing SBA dosage and t_{mix} to be (10 g/L) and (30 min) respectively, and variable N (80, 100 and 120) as shown in figures (88, 89 and 90). Figure (88) revealed from SO_4 curve that the concentrations SO_4 leads to decrease until reaching (< 250 mg/L) at (80 rpm) of mixing speed,



while increasing in chlorides concentration and TDS. Figure (89) illustrates pH curve which indicated that solution tended to be slightly acidic with slightly increasing in temperature. Figure (90) illustrates SO_4 removal efficiency curve by using variable mixing speeds for disposal of treated water the optimum mixing speed to reduce sulphate concentration from (550 mg/L) to (250 mg/L) for disposal of treated water was (70 rpm).

- * All data obtained from ion exchange testes that mentioned above of specified retention time (60 and 30 min) for reuse and disposal respectively, and mixing speed of (120 rpm), have been collected and analyzed Figures (91, 92, 93, 94, 95 and 96) for decision making about optimum dosages used for by overall viewing to be a laboratory reference of process operation. As showed in figures (91 and 94), when retention time was (60 min) the sulphate concentrations tended to drop to (230mg/L) at (10 g/L) dosage then continue in reducing until reached (11 mg/L) at (100 g/L) dosage. As in this case, it can be considered (100g/L) was an optimum dosage for reuse and recommended for (9.5g/L) dosage for disposal. On the other hand, when retention time was (30min) the sulphate remaining curve was descent linearly when retention time was (30 min) until reaching (190mg/L) when dosage = (10g/L) then continue in reducing until reach (14 mg/L) at (100 g/L) dosage it can considered (8.0 g/L) is an optimum dosage for disposal. Figure (92) indicate that TDS chloride tend to increase until reach (2990mg/L) of TDS at (20 g/L) and (1040mg/L) of chloride at (10 g/L) dosage, then decreasing reached (2955 mg/L) of TDS at (30 g/L) and (932 mg/L) of chloride at (20 g/L) then continues increasing. Figure (95) showed the

same behavior of previous figures of (60 min) retention time, this indicated that time effecting just on reaction rate not on behavior. It can conclude from other figures of (93 and 96) that pH tended to be acidic in smaller dosages and alkaline with higher dosages of SBA resin.

Table (2) and Table (3) illustrate the maximum and optimum design parameters recommended to use for remove and reduce refinery treated water sulphate of average, maximum and peak concentrations for reuse and disposal purposes respectively by using (mberlite IRA402 OH) resin.

Table (2): Maximum and optimum design parameters recommended to use for sulphate removal from refinery treated water for reuse by using SBA

Influent SO ₄ (mg/L)	SBA Dosage (g/L)		Retention time (min)		Mixing speed (rpm)		Effluent SO ₄ (mg/L)		Removal efficiency (%)	
	Max.	Opt.	Max.	Opt.	Max.	Opt.	Max.	Opt.	Max.	Opt.
360	100	85	60	30	120	83	0	10	100	100
425	100	70	60	16	80	75	5	10	98.8	100
550	100	100	60	17	120	80	11	15	98	99.09

Table (3): Maximum and optimum design parameters recommended to use for sulphate removal from refinery treated water for disposal by using SBA

Influent SO ₄ (mg/L)	SBA Dosage (g/L)		Retention time (min)		Mixing speed (rpm)		Effluent SO ₄ (mg/L)		Removal efficiency (%)	
	Max.	Opt.	Max.	Opt.	Max.	Opt.	Max.	Opt.	Max.	Opt.
360	1	0.56	30	16	120	100	215	250	40.28	100
425	6	5.8	15	10	120	88	220	250	48.24	100
550	10	8	15	12	120	70	230	250	58.18	100
			30		80					



Conclusions

1. The inspection of refinery water quality used in various processes that showed in table (1) before indicated that the main highly concentration of sulphate in wastewater that reach about (2900 mg/L) was coming from blow down water of energy units, because of using sulfuric acid for resin regeneration in ion exchange process.
2. The analysis of WWTP effluent water quality historical data indicated that the annual rate, maximum and peak sulphate concentrations are found to be (360 mg/L, 425 mg/L and 550 mg/L), and pH ranged from (7.0 to 7.6).
3. The maximum and the optimum (dosage, mixing time and mixing speed) to use for sulphate removal for reuse and disposal purpose at annual rate, max and peak sulphate concentrations have been shown in tables (2) and (3).
4. The considered design parameters of (1.0 hr and 120 rpm) for reuse purpose, and (30 min and 120 rpm) for disposal, the results obtained as shown in Table 2 and 3 indicated that (30 min) and (83 rpm) are sufficient for reuse, and (16 min) and (100 rpm) are the optimum for disposal.
5. The increasing of (Amberlite IRA402 OH) resin dosages led to decrease SO_4 concentrations while on the other hand increasing in chlorides concentration.
6. The increasing in SBA concentrations led to increase pH due to chemical reaction of (Amberlite IRA402 OH) with Na_2SO_4 .
7. The increasing of (Amberlite IRA402 OH) resin dosages leads to decrease temperature in the beginning of reaction and (TDS) increased, because the rate of chemical reactions generally slowed in the beginning so that temperature decreased but TDS increase because increasing chlorides.



8. Strong base anion resin which was worked in pH= (2.0) and worked in pH ranged between (7.0 and 8.0), but weak base anion resin which was worked only in low pH.
9. When was used (Amberlite IRA402 OH) resin with samples have pH =(7.0), it achieved about (98.93%) removal but GYP-CIX process to remove sulphate, when pH = 2.7 it achieved about to more than (95%) sulphate removal.
10. The major advantage of this method was to produce a high quality water and cheap in treatment cost of about (0.07 \$ US/m³) in case of reusing and of about (0.0007 \$ US/m³) in case of disposal. A major disadvantage was producing of brine that required disposal with incurs additional costs.

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Ion Exchange by using (IRA402 - OH)

For Reuse of Treated Water

For Disposal of Treated Water

SO₄ = 360 mg/L

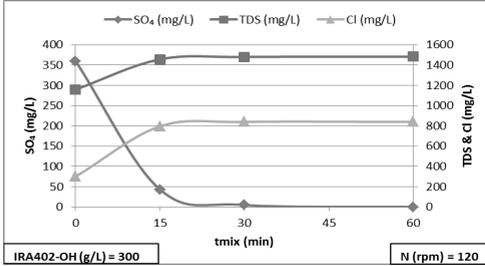


Figure (1): SO₄, TDS and Cl behavior curves by using variable mixing times for reuse of treated water.

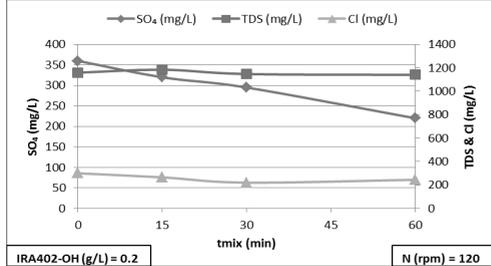


Figure (4): SO₄, TDS and Cl behavior curves by using variable mixing times for disposal of treated water.

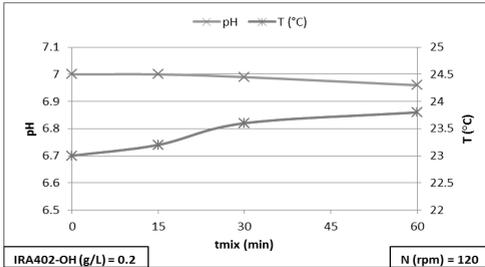


Figure (2): pH and temperature behavior curves by using variable mixing times for reuse of treated water.

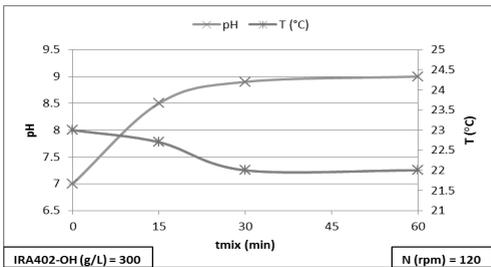


Figure (5): pH and temperature behavior curves by using variable mixing times for disposal of treated water.

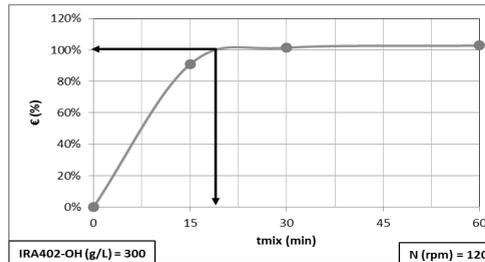


Figure (3): SO₄ removal efficiency curve by using variable mixing times for reuse of treated water.

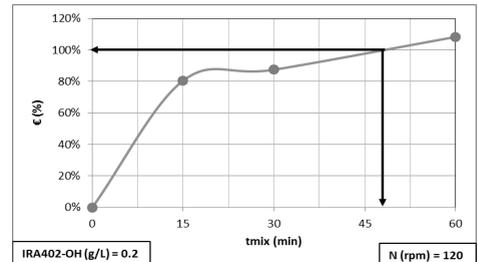


Figure (6): SO₄ removal efficiency curve by using variable mixing times for disposal of treated water.

Ion Exchange by using (IRA402 - OH)

For Reuse of Treated Water

For Disposal of Treated Water

SO₄ = 360 mg/L

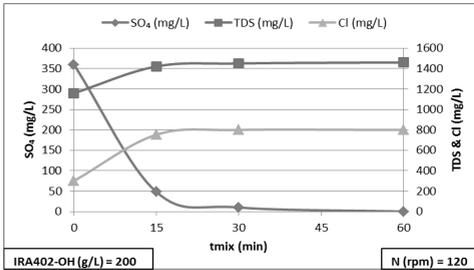


Figure (7): SO₄, TDS and Cl behavior curves by using variable mixing times for reuse of treated water.

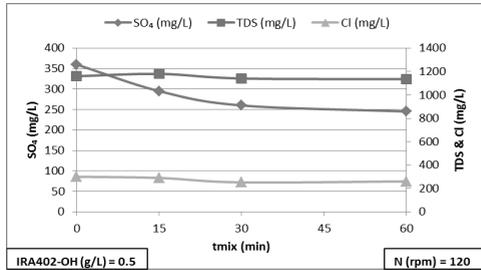


Figure (10): SO₄, TDS and Cl behavior curves by using variable mixing times for disposal of treated water.

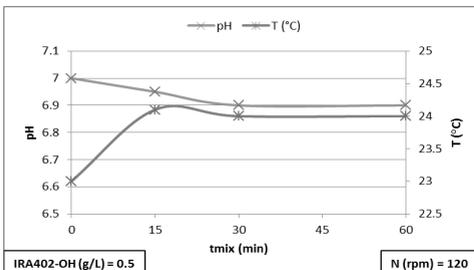


Figure (8): pH and temperature behavior curves by using variable mixing times for reuse of treated water.

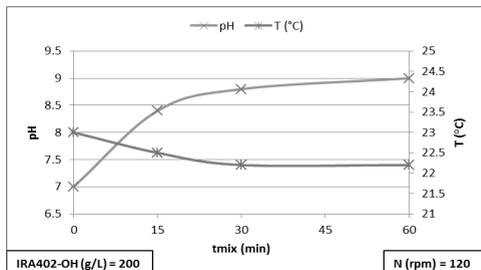


Figure (11): pH and temperature behavior curves by using variable mixing times for disposal of treated water.

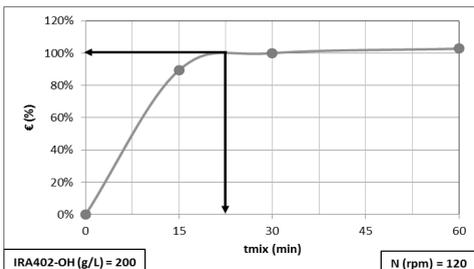


Figure (9): SO₄ removal efficiency curve by using variable mixing times for reuse of treated water.

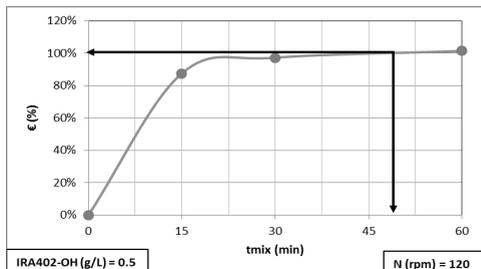


Figure (12): SO₄ removal efficiency curve by using variable mixing times for disposal of treated water.

Ion Exchange by using (IRA402 - OH)

For Reuse of Treated Water

For Disposal of Treated Water

SO₄ = 360 mg/L

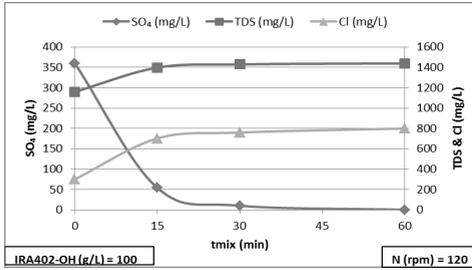


Figure (13): SO₄, TDS and Cl behavior curves by using variable mixing times for reuse of treated water.

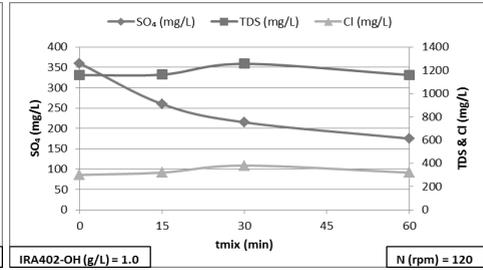


Figure (16): SO₄, TDS and Cl behavior curves by using variable mixing times for disposal of treated water.

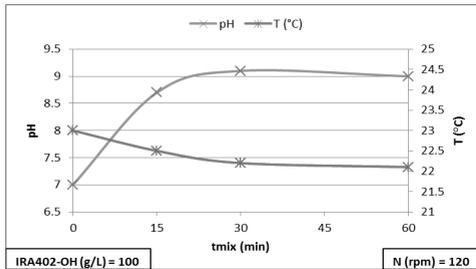


Figure (14): pH and temperature behavior curves by using variable mixing times for reuse of treated water.

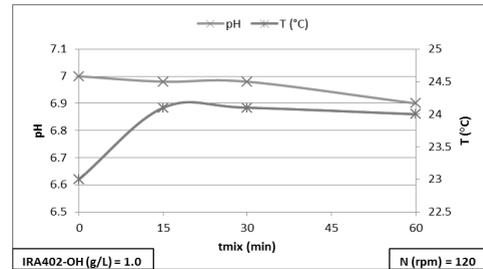


Figure (17): pH and temperature behavior curves by using variable mixing times for disposal of treated water.

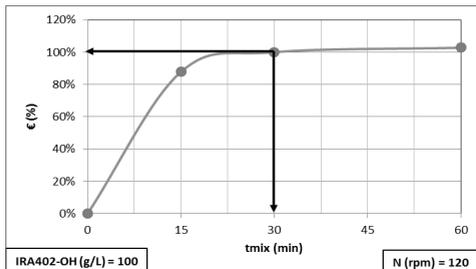


Figure (15): SO₄ removal efficiency curve by using variable mixing times for reuse of treated water.

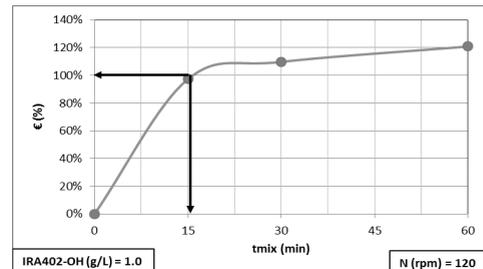


Figure (18): SO₄ removal efficiency curve by using variable mixing times for disposal of treated water.

Ion Exchange by using (IRA402 - OH)

For Reuse of Treated Water

For Disposal of Treated Water

SO₄ = 360 mg/L

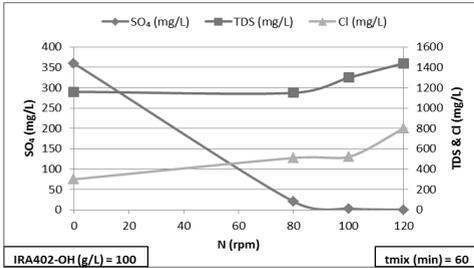


Figure (19): SO₄, TDS and Cl behavior curves by using variable mixing speeds for reuse of treated water.

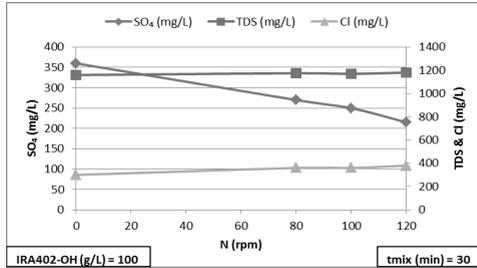


Figure (22): SO₄, TDS and Cl behavior curves by using variable mixing speeds for disposal of treated water.

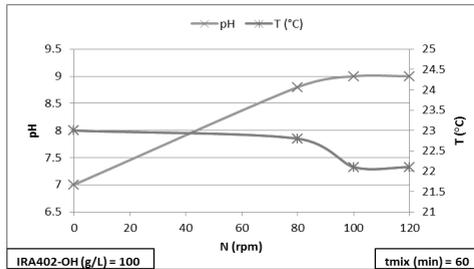


Figure (20): pH and temperature behavior curves by using variable mixing speeds for reuse of treated water

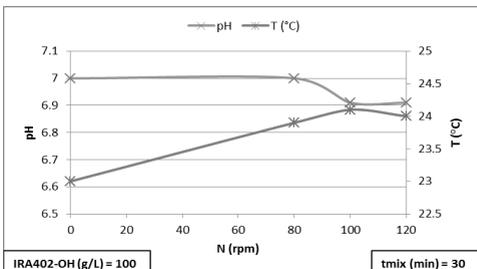


Figure (23): pH and temperature behavior curves by using variable mixing speeds for disposal of treated water

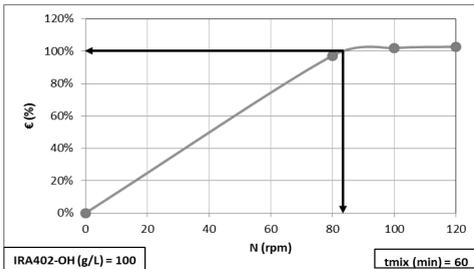


Figure (21): SO₄ removal efficiency curve by using variable mixing speeds for reuse of treated water.

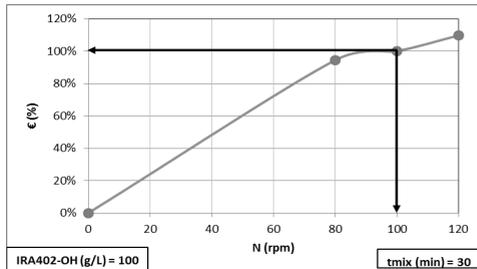


Figure (24): SO₄ removal efficiency curve by using variable mixing speeds for disposal of treated water.

Ion Exchange by using (IRA402 - OH)

For Decision Making

For Decision Making

SO₄ = 360 mg/L

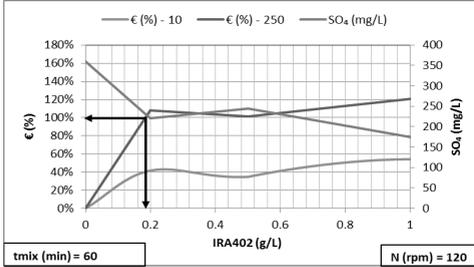


Figure (25A): Overall SO₄, reuse & disposal removal efficiency behavior curves by using variable IRA402 dosages in (60 min) retention time.

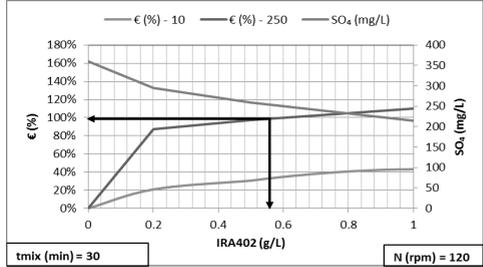


Figure (28A): Overall SO₄, reuse & disposal removal efficiency behavior curves by using variable IRA402 dosages in (30 min) retention time.

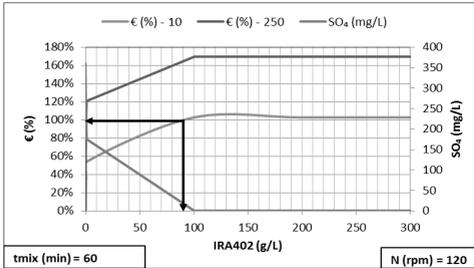


Figure (25B): Large scale for small interval between (0.0 and 1.0 g/L) IRA402 dosages of figures (25 A).

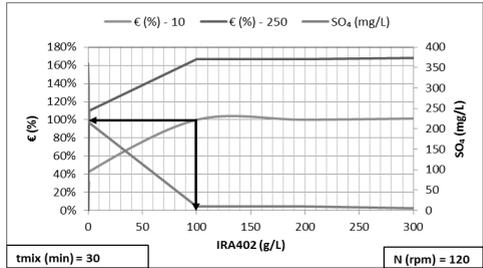


Figure (28B): Large scale for small interval between (0.0 and 1.0 g/L) IRA402 dosages of figures (28 A).

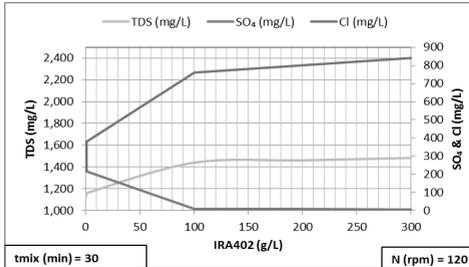


Figure (26A): Overall SO₄, TDS and Cl behavior curves by using variable IRA402 dosages for reuse or disposal in (60 min) retention time.

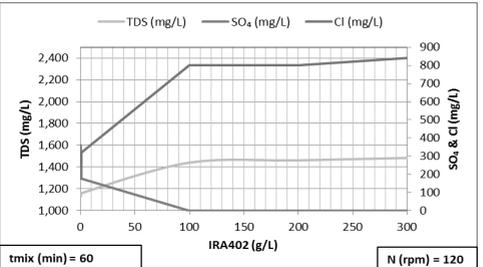


Figure (29A): Overall SO₄, TDS and Cl behavior curves by using variable IRA402 dosages for reuse or disposal in (30 min) retention time.

Ion Exchange by using (IRA402 - OH)

For Decision Making

For Decision Making

SO₄ = 360 mg/L

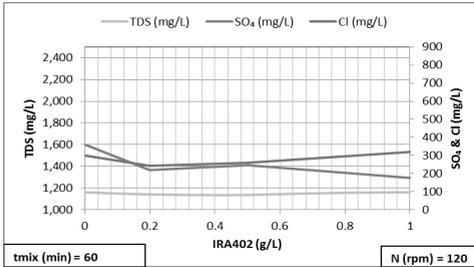


Figure (26B): Large scale for small interval between (0.0 and 1.0 g/L) IRA402 dosages of figures (26 A).

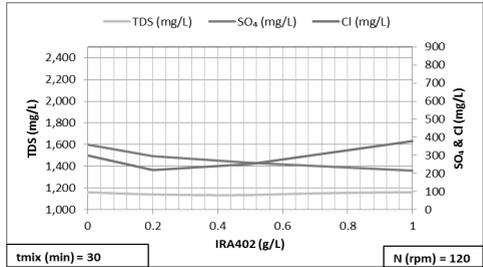


Figure (29B): Large scale for small interval between (0.0 and 1.0 g/L) IRA402 dosages of figures (29 A).

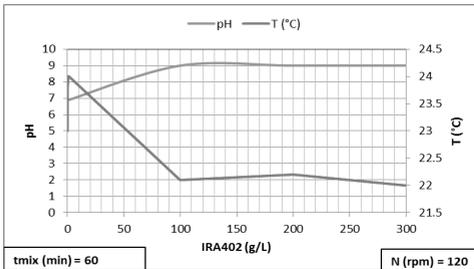


Figure (27A): Overall pH and temperature behavior curves by using variable IRA402 dosages for reuse or disposal in (60 min) retention time.

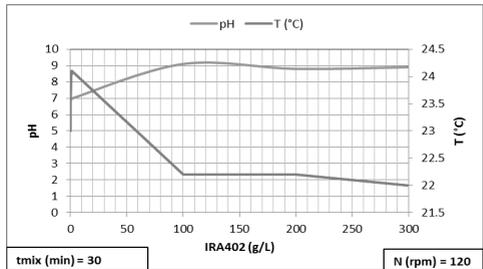


Figure (30A): Overall pH and temperature behavior curves by using variable IRA402 dosages for reuse or disposal in (30 min) retention time.

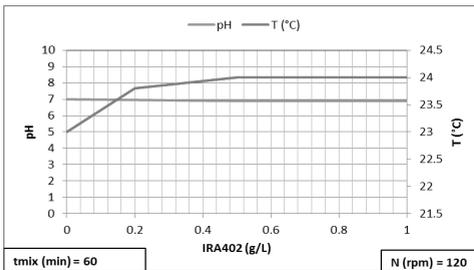


Figure (27B): Large scale for small interval between (0.0 and 1.0 g/L) IRA402 dosages of figures (27A).

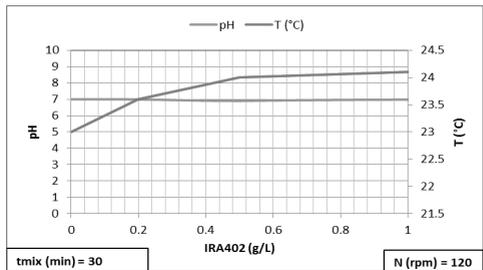


Figure (30B): Large scale for small interval between (0.0 and 1.0 g/L) IRA402 dosages of figures (30A).

Ion Exchange by using (IRA402 - OH)

For Reuse of Treated Water

For Disposal of Treated Water

SO₄ = 425 mg/L

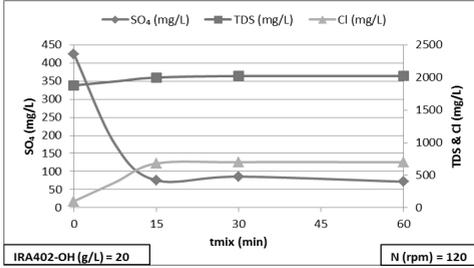


Figure (31): SO₄, TDS and Cl behavior curves by using variable mixing times for reuse of treated water.

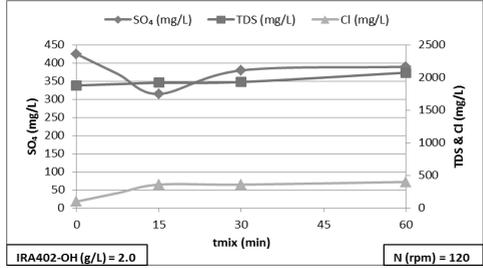


Figure (34): SO₄, TDS and Cl behavior curves by using variable mixing times for disposal of treated water.

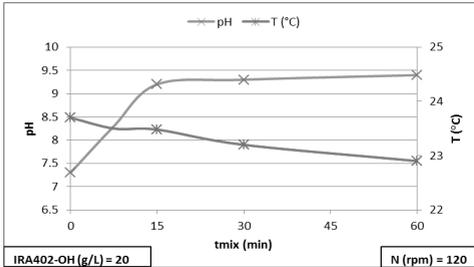


Figure (32): pH and temperature behavior curves by using variable mixing times for reuse of treated water.

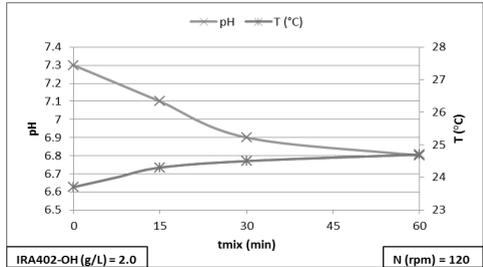


Figure (35): pH and temperature behavior curves by using variable mixing times for disposal of treated water.

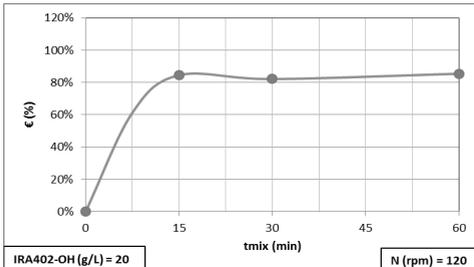


Figure (33): SO₄ removal efficiency curve by using variable mixing times for reuse of treated water.

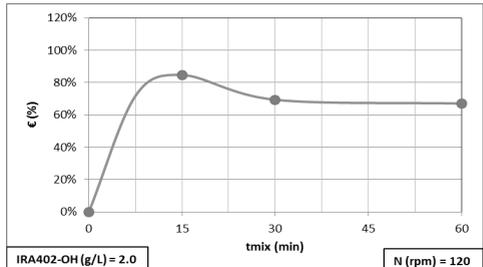


Figure (36): SO₄ removal efficiency curve by using variable mixing times for disposal of treated water.

Ion Exchange by using (IRA402 - OH)

For Reuse of Treated Water

For Disposal of Treated Water

SO₄ = 425 mg/L

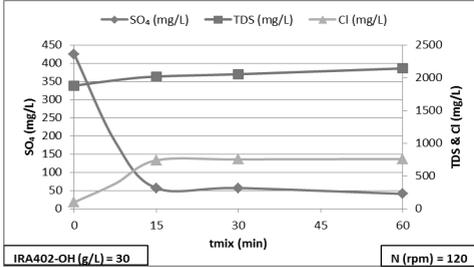


Figure (37): SO₄, TDS and Cl behavior curves by using variable mixing times for reuse of treated water.

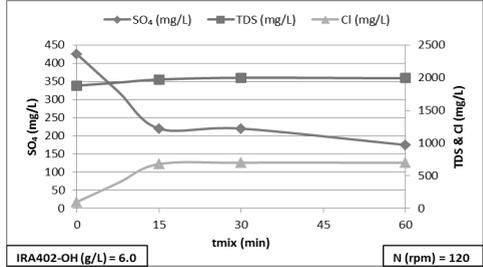


Figure (40): SO₄, TDS and Cl behavior curves by using variable mixing times for disposal of treated water.

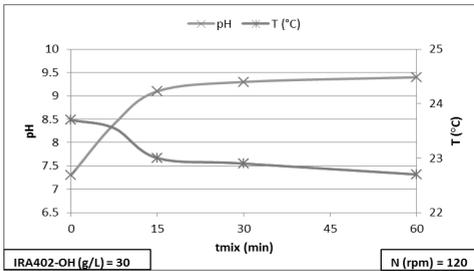


Figure (38): pH and temperature behavior curves by using variable mixing times for reuse of treated water.

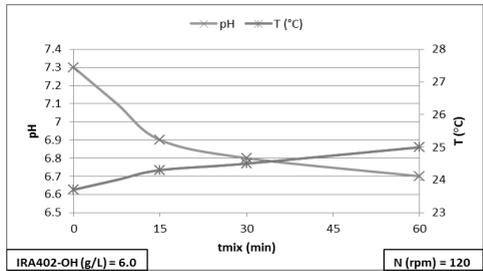


Figure (41): pH and temperature behavior curves by using variable mixing times for disposal of treated water.

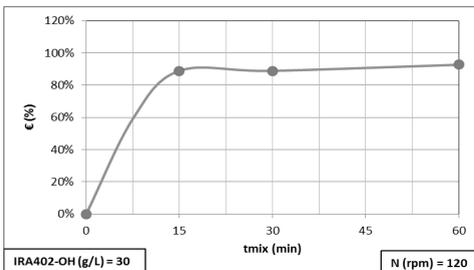


Figure (39): SO₄ removal efficiency curve by using variable mixing times for reuse of treated water.

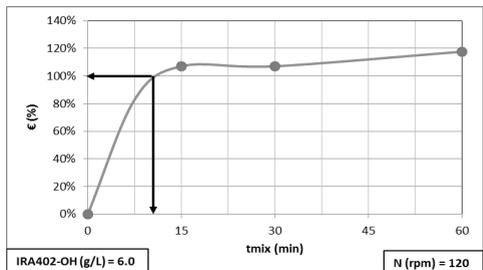


Figure (42): SO₄ removal efficiency curve by using variable mixing times for disposal of treated water.

Ion Exchange by using (IRA402 - OH)

For Reuse of Treated Water

For Disposal of Treated Water

SO₄ = 425 mg/L

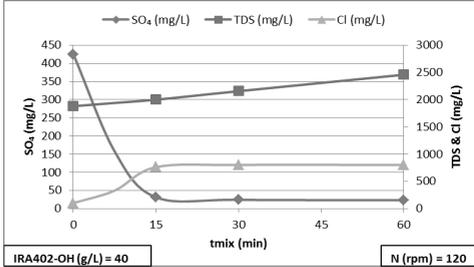


Figure (43): SO₄, TDS and Cl behavior curves by using variable mixing times for reuse of treated water.

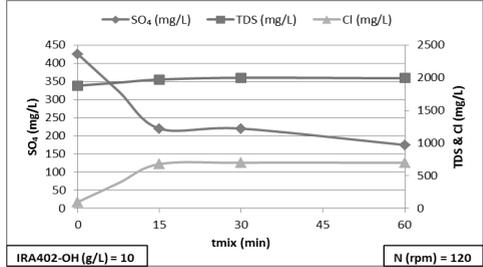


Figure (46): SO₄, TDS and Cl behavior curves by using variable mixing times for disposal of treated water.

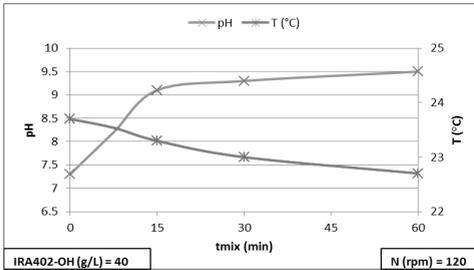


Figure (44): pH and temperature behavior curves by using variable mixing times for reuse of treated water.

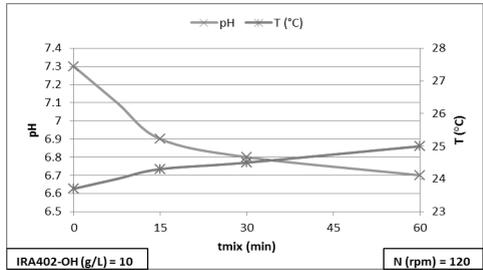


Figure (47): pH and temperature behavior curves by using variable mixing times for disposal of treated water.

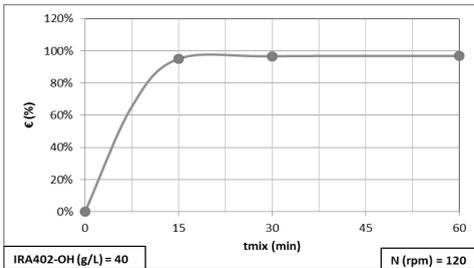


Figure (45): SO₄ removal efficiency curve by using variable mixing times for reuse of treated water.

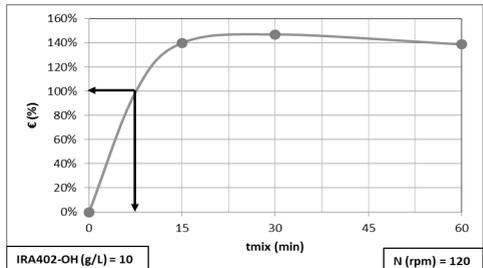


Figure (48): SO₄ removal efficiency curve by using variable mixing times for disposal of treated water.

Ion Exchange by using (IRA402 - OH)

For Reuse of Treated Water

For Disposal of Treated Water

SO₄ = 425 mg/L

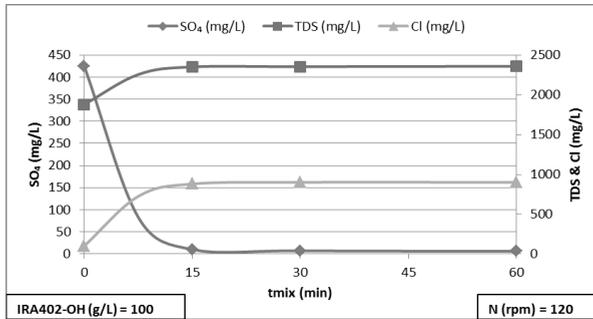


Figure (49): SO₄, TDS and Cl behavior curves by using variable mixing times for reuse of treated water.

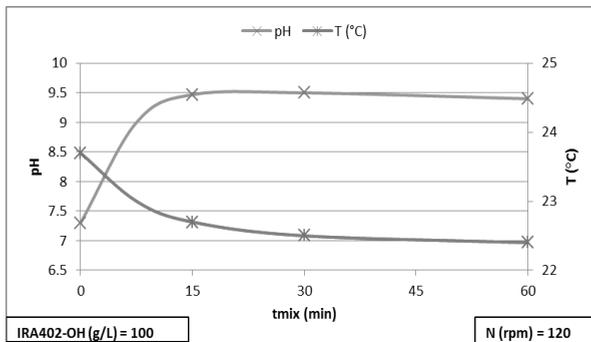


Figure (50): pH and temperature behavior curves by using variable mixing times for reuse of treated water.

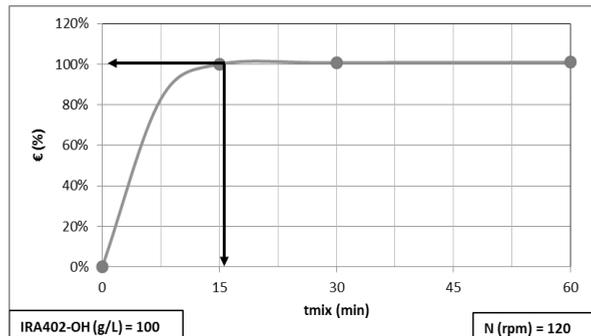


Figure (51): SO₄ removal efficiency curve by using variable mixing times for reuse of treated water.

Ion Exchange by using (IRA402 - OH)

For Reuse of Treated Water

For Disposal of Treated Water

SO₄ = 425 mg/L

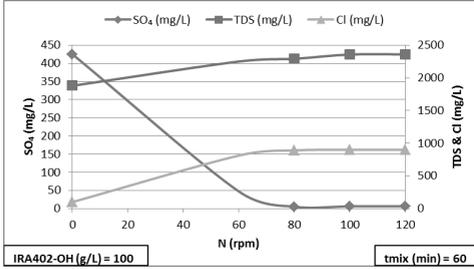


Figure (52): SO₄, TDS and Cl behavior curves by using variable mixing speeds for reuse of treated water.

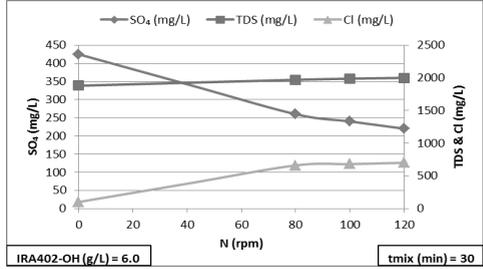


Figure (55): SO₄, TDS and Cl behavior curves by using variable mixing speeds for disposal of treated water.

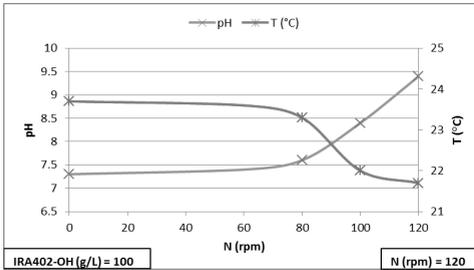


Figure (53): pH and temperature behavior curves by using variable mixing speeds for reuse of treated water.

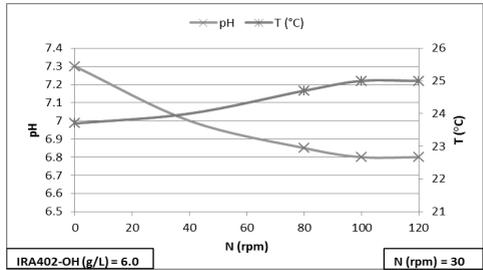


Figure (56): pH and temperature behavior curves by using variable mixing speeds for disposal of treated water.

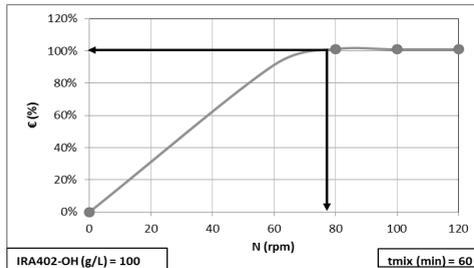


Figure (54): SO₄ removal efficiency curve by using variable mixing speeds for reuse of treated water.

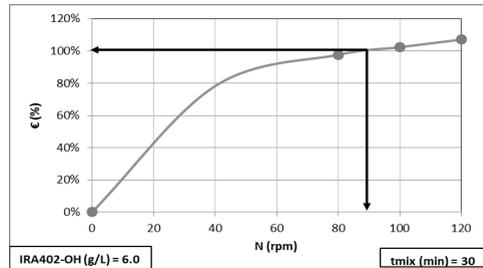


Figure (57): SO₄ removal efficiency curve by using variable mixing speeds for disposal of treated water.

Ion Exchange by using (IRA402 - OH)

For Decision Making

For Decision Making

SO₄ = 425 mg/L

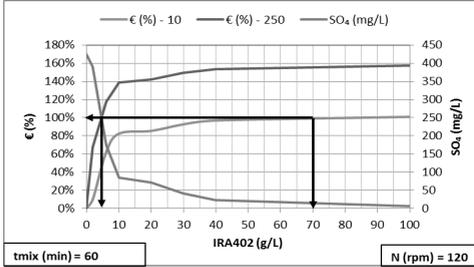


Figure (58): Overall SO₄, reuse & disposal removal efficiency behavior curves by using variable IRA402 dosages in (60 min) retention time.

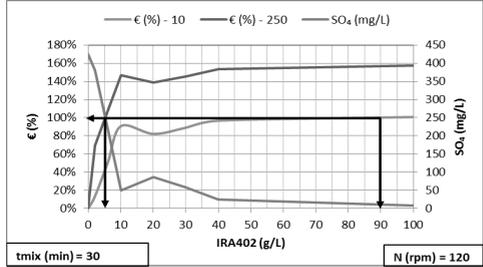


Figure (61): Overall SO₄, reuse & disposal removal efficiency behavior curves by using variable IRA402 dosages in (30 min) retention time.

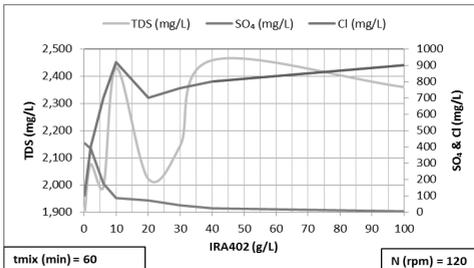


Figure (59): Overall SO₄, TDS and Cl behavior curves by using variable IRA402 dosages for reuse or disposal in (60 min) retention time.

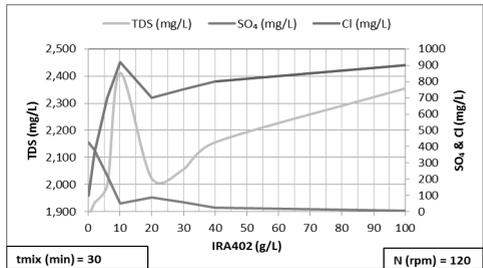


Figure (62): Overall SO₄, TDS and Cl behavior curves by using variable IRA402 dosages for reuse or disposal in (30 min) retention time.

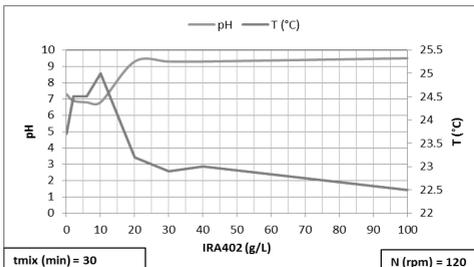


Figure (60): Overall pH and temperature behavior curves by using variable IRA402 dosages for reuse or disposal in (60 min) retention time.

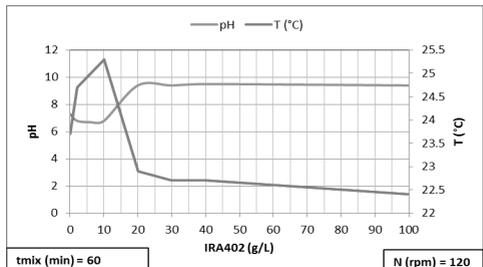


Figure (63): Overall pH and temperature behavior curves by using variable IRA402 dosages for reuse or disposal in (30 min) retention time.

Ion Exchange by using (IRA402 - OH)

For Reuse of Treated Water

For Disposal of Treated Water

SO₄ = 550 mg/L

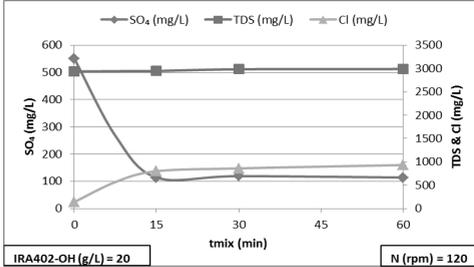


Figure (64): SO₄, TDS and Cl behavior curves by using variable mixing times for reuse of treated water.

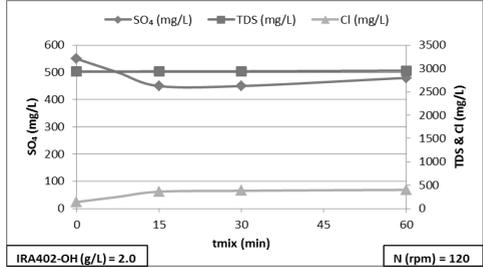


Figure (67): SO₄, TDS and Cl behavior curves by using variable mixing times for disposal of treated water.

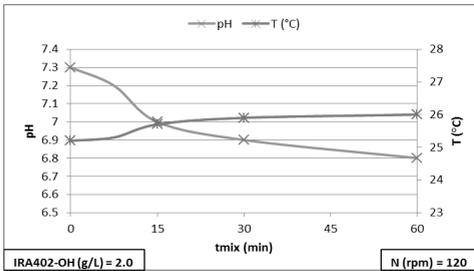


Figure (65): pH and temperature behavior curves by using variable mixing times for reuse of treated water.

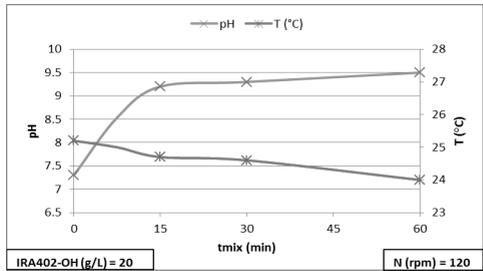


Figure (68): pH and temperature behavior curves by using variable mixing times for disposal of treated water.

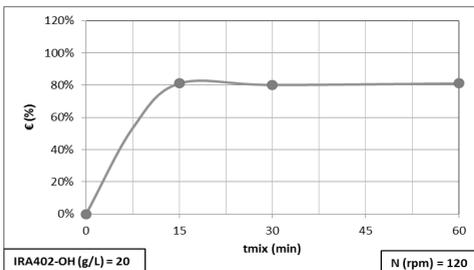


Figure (66): SO₄ removal efficiency curve by using variable mixing times for reuse of treated water.

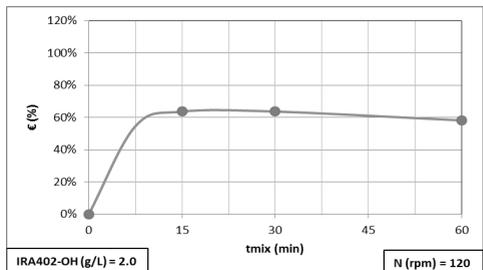


Figure (69): SO₄ removal efficiency curve by using variable mixing times for disposal of treated water.

Ion Exchange by using (IRA402 - OH)

For Reuse of Treated Water

For Disposal of Treated Water

SO₄ = 550 mg/L

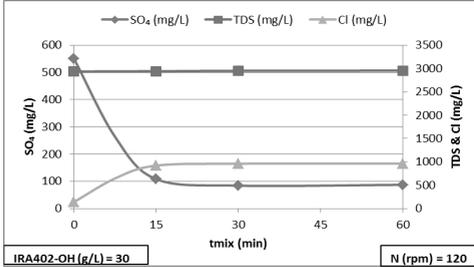


Figure (70): SO₄, TDS and Cl behavior curves by using variable mixing times for reuse of treated water.

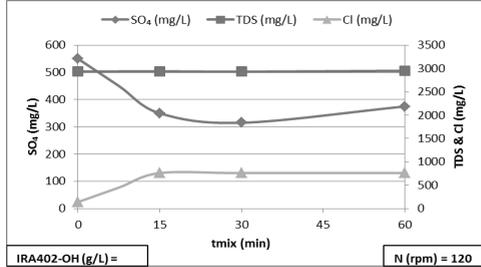


Figure (73): SO₄, TDS and Cl behavior curves by using variable mixing times for disposal of treated water.

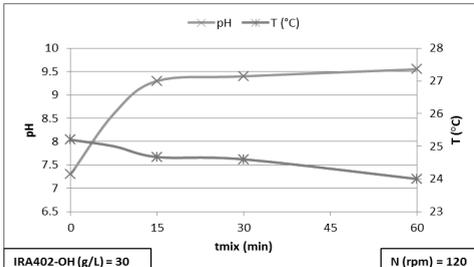


Figure (71): pH and temperature behavior curves by using variable mixing times for reuse of treated water.

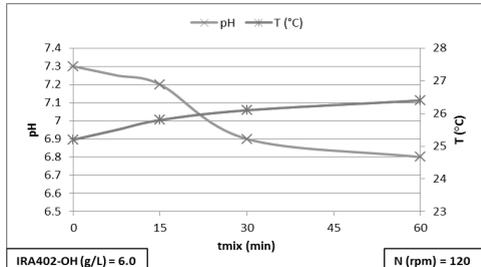


Figure (74): pH and temperature behavior curves by using variable mixing times for disposal of treated water.

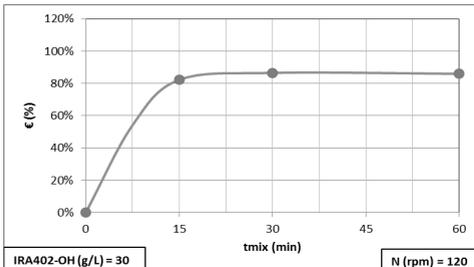


Figure (72): SO₄ removal efficiency curve by using variable mixing times for reuse of treated water.

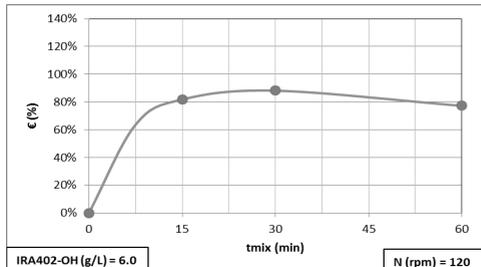


Figure (75): SO₄ removal efficiency curve by using variable mixing times for disposal of treated water.

Ion Exchange by using (IRA402 - OH)

For Reuse of Treated Water

For Disposal of Treated Water

SO₄ = 550 mg/L

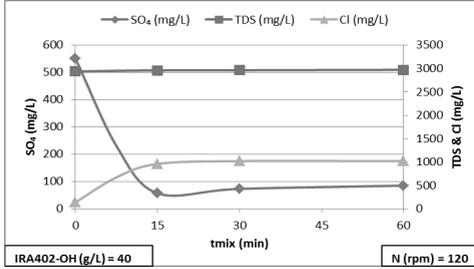


Figure (76): SO₄, TDS and Cl behavior curves by using variable mixing times for reuse of treated water.

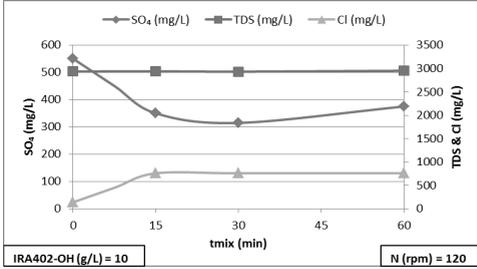


Figure (79): SO₄, TDS and Cl behavior curves by using variable mixing times for disposal of treated water.

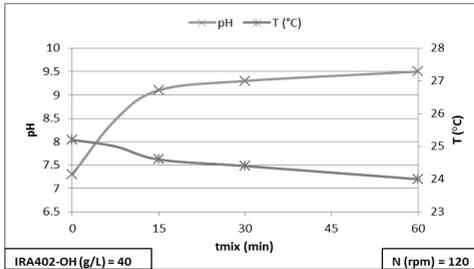


Figure (77): pH and temperature behavior curves by using variable mixing times for reuse of treated water.

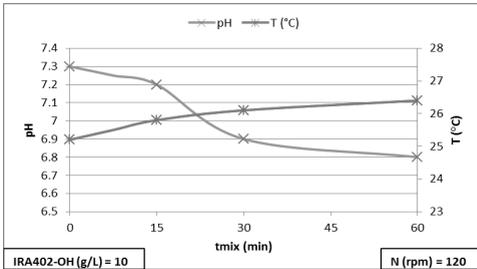


Figure (80): pH and temperature behavior curves by using variable mixing times for disposal of treated water.

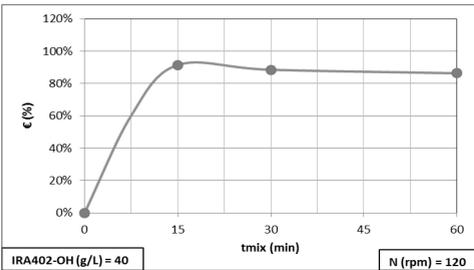


Figure (78): SO₄ removal efficiency curve by using variable mixing times for reuse of treated water.

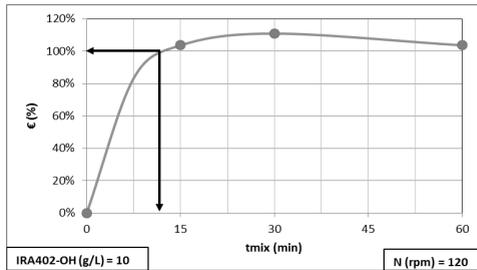


Figure (81): SO₄ removal efficiency curve by using variable mixing times for disposal of treated water.

Ion Exchange by using (IRA402 - OH)

For Reuse of Treated Water

SO₄ = 550 mg/L

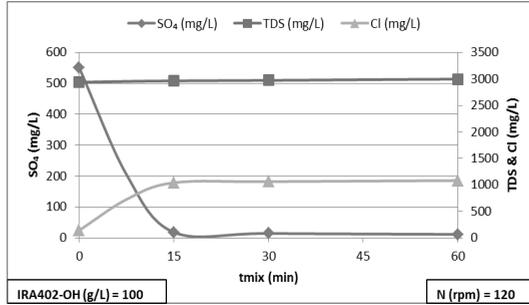


Figure (82): SO₄, TDS and Cl behavior curves by using variable mixing times for reuse of treated water.

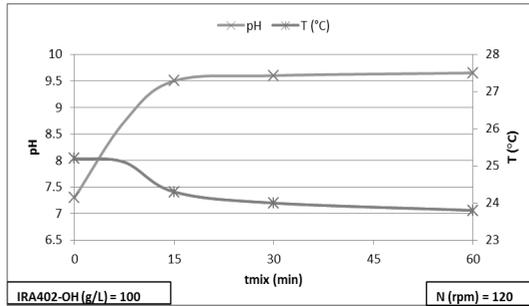


Figure (83): pH and temperature behavior curves by using variable mixing times for reuse of treated water.

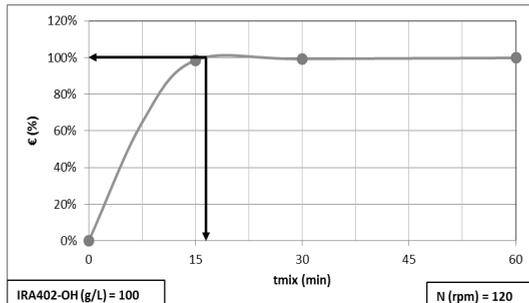


Figure (84): SO₄ removal efficiency curve by using variable mixing times for reuse of treated water.

Ion Exchange by using (IRA402 - OH)

For Reuse of Treated Water

For Disposal of Treated Water

SO₄ = 550 mg/L

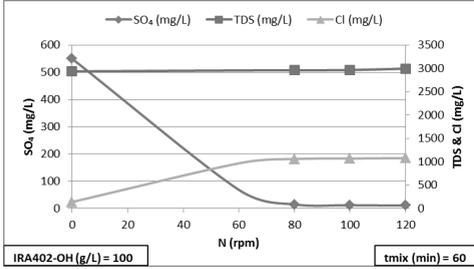


Figure (85): SO₄, TDS and Cl behavior curves by using variable mixing speeds for reuse of treated water.

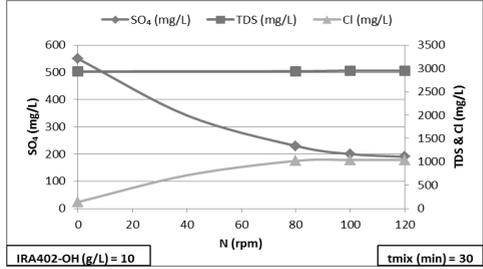


Figure (88): SO₄, TDS and Cl behavior curves by using variable mixing speeds for disposal of treated water.

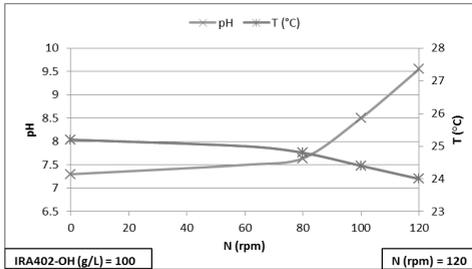


Figure (86): pH and temperature behavior curves by using variable mixing speeds for reuse of treated water.

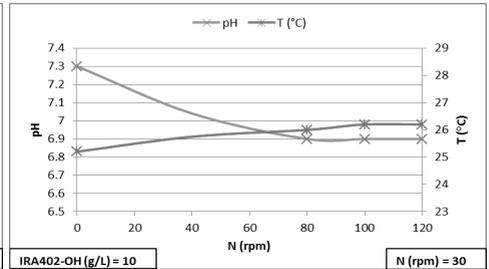


Figure (89): pH and temperature behavior curves by using variable mixing speeds for disposal of treated water.

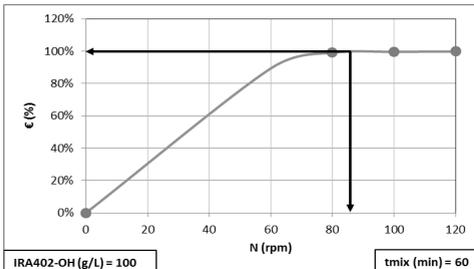


Figure (87): SO₄ removal efficiency curve by using variable mixing speeds for reuse of treated water.

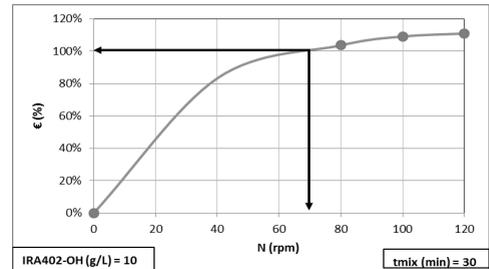


Figure (90): SO₄ removal efficiency curve by using variable mixing speeds for disposal of treated water.

Ion Exchange by using (IRA402 - OH)

For Decision Making

For Decision Making

SO₄ = 550 mg/L

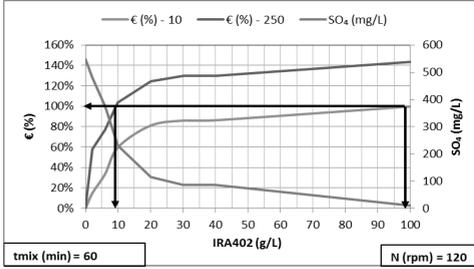


Figure (91): Overall SO₄, reuse & disposal removal efficiency behavior curves by using variable IRA402 dosages in (60 min) retention time.

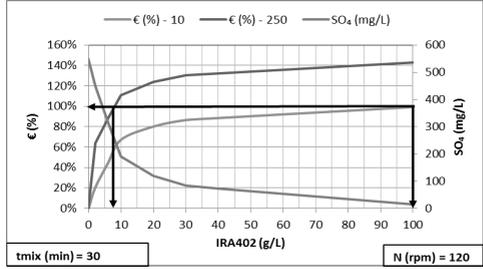


Figure (94): Overall SO₄, reuse & disposal removal efficiency behavior curves by using variable IRA402 dosages in (30 min) retention time.

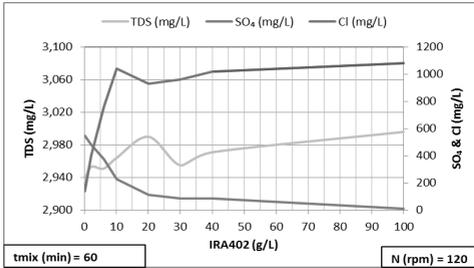


Figure (92): Overall SO₄, TDS and Cl behavior curves by using variable IRA402 dosages for reuse or disposal in (60 min) retention time.

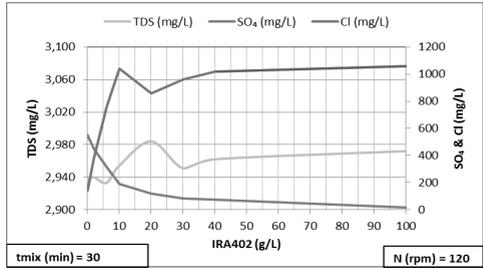


Figure (95): Overall SO₄, TDS and Cl behavior curves by using variable IRA402 dosages for reuse or disposal in (30 min) retention time.

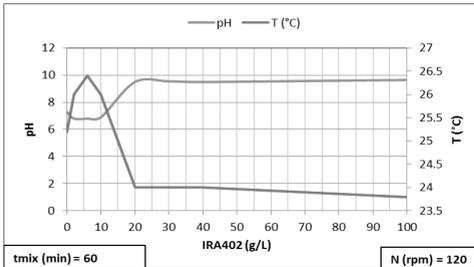


Figure (93): Overall pH and temperature behavior curves by using variable IRA402 dosages for reuse or disposal in (60 min) retention time.

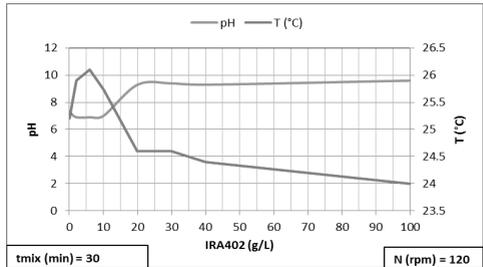


Figure (96): Overall pH and temperature behavior curves by using variable IRA402 dosages for reuse or disposal in (30 min) retention time.

