

Research Article

Removal of Hardness of Earth Alkaline Metals from Aqueous Solutions by Ion Exchange Method

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An ion exchange process was introduced as an approach for softening of artificial hard water solutions. A strong acid cation exchange resin, Amberlite IR 120 [Na⁺], was used to reduce the hardness of water with the matrix of styrene-divinylbenzene copolymer having functional group as sulfonate. The ion exchange behavior of the ions of calcium and magnesium in synthetic solutions of hard water was investigated with the variables depending on pH, stirrer speed of the solutions and amount of the resin as a function of contact time between resin phase and hard water solution. The maximum ion exchange capacity was found to be 68 mg/g for Ca(II) and 12 mg/g for Mg(II) at pH 3.0. The method is a simple and efficient one to remove calcium and magnesium hardness from hard water solutions with the resin having more selectivity for calcium.

1. Introduction

The existence of the soluble Ca(II) and Mg(II) salts has caused unsuitable behavior of hard water solutions for drinking, watering, and the purposes of industrial. The ionic impurities can lead to problems in cooling and heating systems, steam generation, and manufacturing. The high calcium and magnesium concentrations have resulted in clogging of pipelines and heat exchangers through scaling as the form of carbonate, sulfate, or phosphate precipitates. Therefore the necessity of obtaining water having low level of hardness has taken place inside important occupational areas for industries such as leather production [1]. The common methods used for removal of the metals of earth alkaline from aqueous solutions and softening of water can be classified as chemical precipitation, ultrafiltration, reverse osmosis, electrodialysis, adsorption, and ion exchange [2–4]. Water softening using electrochemical techniques has gained attention [5]. The packed bed of polypyrrole/polystyrene sulfonate electrodeposited porous carbon electrode has been used for continuous water softening from flowing artificial hard water solutions [6].

The ion exchange resins have numerous commercial and industrial uses particularly in water purification and removal

of metal ions at very low concentrations in chemical process of industries [7]. Some polymeric resins having strongly acid sulfonic or weakly acid carboxylic functionalities are usually used in ion exchange processes [8–11]. A method has been focused on the combination of ultrasound and ion exchange for removal of hardness of calcium and magnesium from water [12]. Some methods have been conducted for removing Ca(II) and Mg(II) from water by using chelating resins with high selectivity [13–15]. The exchange of Ca(II) and Mg(II) from LiHCO₃ solution was studied with Amberlite IRC 747 [16]. Applications of polymeric resins containing iminodiacetate have been investigated from aqueous solutions for recovery of Ca(II) and Mg(II) [17–23]. Amberlite IRC 748 as K⁺ form was used from potassium chromate solution for removal of Ca(II) and Mg(II) [24]. A study was performed on softening of waste geothermal water using ion exchange resins [25].

There are some studies by ion exchange technology using the resin of Amberlite IR 120. Some of them for Ca(II) removal [26] and magnesium adsorption [27] have been investigated by using empirical kinetic models.

In the present work the main objective of the study is to remove the magnesium and calcium hardness from synthetic solutions of hard water. The selective adsorption equilibrium

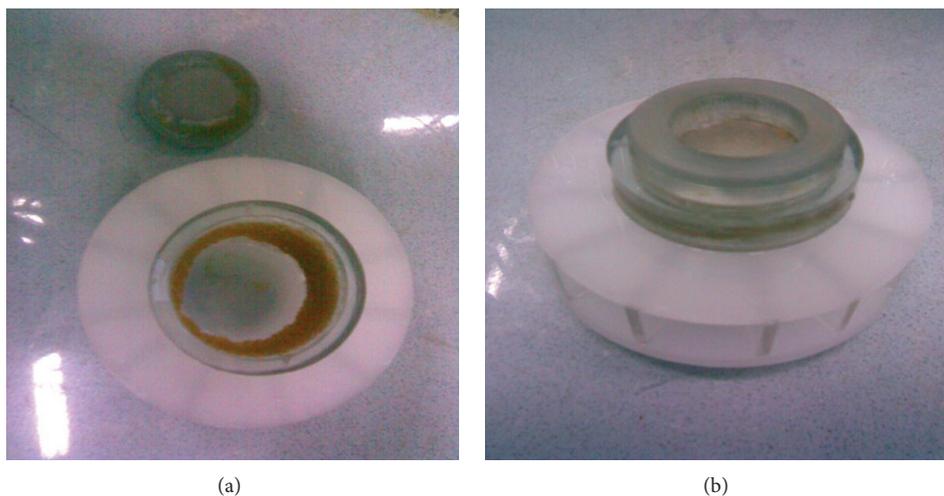


FIGURE 1: The basket of the resin.

for earth alkaline metals was investigated under different conditions such as pH, stirring speed of the solutions, and amount of the resin as a function of contact time between the phase of resin and synthetic hard water solutions. The examined method was applied for removal of calcium and magnesium hardness of tap water obtained from research laboratory of YILDIZ Technical University, Faculty of Science and Art, Analytical Chemistry Department, Esenler, Istanbul, Turkey.

2. Experimental

2.1. Materials. The inorganic chemicals including $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, HCl, and NaOH were obtained from Merck (Darmstadt, Germany) in analytical grade. All solutions were prepared using bidistilled water.

2.2. Ion Exchange Resin. The resin of Amberlite IR 120 [Na^+] [28] was used as a strongly acid cation exchanger having copolymer of styrene and divinylbenzene with functional groups of $\text{SO}_3^- \text{Na}^+$ in the physical form gel type. The properties and suggested operating conditions of the resin have been shown in Tables 1(a) and 1(b).

The resin was washed three times with the solutions of, respectively, 1.0 mol/L HCl and 1.0 mol/L NaOH before its use to remove or reduce possible organic and inorganic impurities. It was then washed with bidistilled water and converted to H^+ form from Na^+ by flushing in fixed bed with 1.0 mol/L HCl. The resin in H^+ form was finally washed with water and used throughout the experiments.

2.3. Preparing of the Solutions of Synthetic Hard Water. The synthetic solutions of hard water have been prepared as stock solution in the volume of 5.0 L. The volume of 950 mL of the stock solution was taken to the batch reactor and after the pH of the solution was adjusted to desired value the solution was transferred to the flask in the volume of 1.0 L by diluting to the volume and it was used as synthetic solution of hard water. The hardness of calcium and magnesium of the solutions with

TABLE 1: Manufacturer data of the resin [28].

(a) The properties of the resin, Amberlite IR 120 [Na^+]

Physical form	Amber spherical beads
Matrix	Styrene-divinylbenzene copolymer
Functional group	Sulfonate
Ionic form as shipped	Na^+
Total exchange capacity	>2.00 eq/L (Na^+ form)
Moisture holding capacity	45 to 50% (Na^+ form)
Shipping weight	840 g/L
Uniformity coefficient	<1.9
Harmonic mean size	0.600 to 0.800 mm
<0.300 mm	2% max
Maximum reversible swelling $\text{Na}^+ \rightarrow \text{H}^+$	< 11%

(b) Suggested operating conditions of the resin

Maximum operating temperature	135°C
Minimum bed depth	700 mm
Service flow rate	5 to 40 BV/h
Regenerant	HCl H_2SO_4 NaCl
Level (g/L)	50 to 150 60 to 240 80 to 250
Concentration (%)	5 to 8 0.7 to 6 10
Minimum contact time	30 minutes
Slow rinse	2 BV at regeneration flow rate
Fast rinse	2 to 4 BV at service flow rate

state of initial and equilibrium is shown as degree of French hardness (FH) in Tables 4, 5, and 6 and in Figure 1.

2.4. Preparing of the Solutions for Analysis. The volume of 0.5 mL of the solution of synthetic hard water was taken to the flask in the volume of 100 mL and diluted to the volume by distilled water.

2.5. Procedure. The batch experiments were carried out to determine the efficiency of removal of calcium and

TABLE 2: Instrumental parameters for determination of calcium and magnesium [29].

Instrumental parameters	Calcium	Magnesium
Wave length (nm)	442,67	442,67
Slit width (nm)	2,7	2,7
Fuel	Acetylene/air	Acetylene/air
Lamp current (mA)	20	20
Optimum working range	(0.5–2.5) mg/L	(0.2–1.0) mg/L

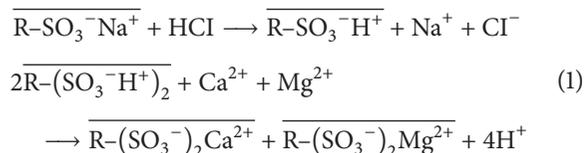
magnesium hardness from synthetic solution of hard water. The sample of 5.0 g of the resin in hydrogen form was put into basket of the resin. The basket of the resin was inserted to the mechanical stirrer with the model of Heildolph RZR 2021. The mechanical stirrer was placed into the vessel of reaction containing synthetic solution of hard water in the volume of 1.0 L. The basket stirrer was moved in the solution for one hour while the stirrer works at constant speed. The apparatus of the resin basket has been shown in Figure 1.

This procedure was applied by using synthetic solutions of hard water having comparable amount of calcium and magnesium hardness. The samples of 500 μL were taken from solution to measure concentration of the calcium and magnesium for each 10 min, 20 min, 30 min, 40 min, 50 min, and 60 min. The samples were diluted to 100 mL and acidified with solution of nitric acid. The concentrations of calcium and magnesium were determined by an atomic absorption spectrophotometer with a model of Perkin Elymer and air-acetylene flame. The balance of the model of Sartorius A 200 S was used for the all weights. Table 2 shows the instrumental parameters for determination of calcium and magnesium by atomic absorption spectrophotometry.

3. Results and Discussion

3.1. Method for Removal of Calcium(II) and Magnesium(II) Hardness. The ion exchange process was developed to remove calcium and magnesium hardness from synthetic hard water solutions by using the resin of Amberlite IR 120 in H^+ form.

The following reactions show that the resin matrix has been converted to the H^+ form from Na^+ and the exchange reaction of calcium and magnesium has been summarized:



The resin's behavior is different for sorption of calcium and magnesium ions in solution and it is more selective for calcium than magnesium and exchange capacity is more high for calcium. Figures 2 and 3 show this phenomena.

The method of atomic absorption spectrophotometric was used for determination of initial and equilibrium concentrations of ions of calcium and magnesium in solution of hard water by using the equations of calibration curve as it is shown in Table 3.

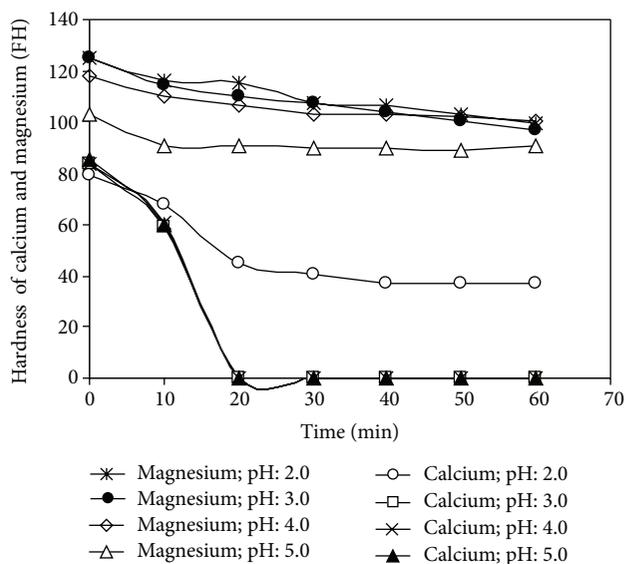


FIGURE 2: The exchange of hardness of calcium and magnesium as a function of initial pH of the solution (the amount of resin: 5.0 g; the stirring speed of the solution: 115 rpm).

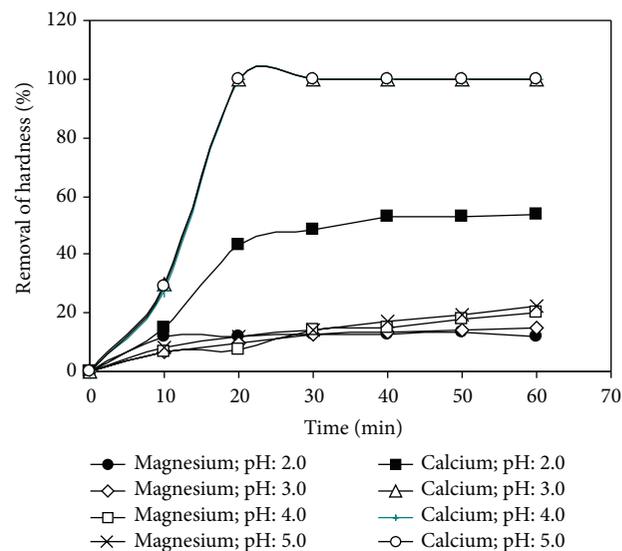


FIGURE 3: The removal of the hardness of calcium and magnesium as a function of initial pH of the solution (the amount of resin: 5.0 g; the stirring speed of the solution: 115 rpm).

TABLE 3: The characteristics of the calibration curves.

Earth alkaline metal	Equation of calibration curve	Coefficient of regression (R^2)
Calcium	$A = 0.0328C - 0.0018$	0.9994
Magnesium	$A = 0.3895C + 0.0145$	0.9985

The sorption process of the resin for calcium and magnesium was investigated under experimental conditions such as contact time between resin and aqueous solution, pH, stirring speed of the solution of hard water, and amount of the resin.

TABLE 4: Experimental conditions depend on effect of pH for removal of hardness of calcium and magnesium.

pH	Stirring speed (rpm)	Resin dosage (g)	Initial hardness of calcium (FH)	Hardness of calcium in equilibrium (FH)	Initial hardness of magnesium (FH)	Hardness of magnesium in equilibrium (FH)
2	115	5	79.12	36.7 (60 min)	124.89	99.83 (60 min)
3	115	5	83.75	0.00 (60 min)	124.89	96.80 (60 min)
4	115	5	83.75	0.00 (60 min)	118.28	100.77 (60 min)
5	115	5	85.25	0.00 (60 min)	102.90	90.50 (60 min)

TABLE 5: ((a), (b), and (c)) The exchange of pH of the solution in equilibrium depends on contact time with the resin according to experimental conditions.

(a)							
Contact time with the resin (min)	0	10	20	30	40	50	60
pH (resin dosage: 5.0 g) (stirring speed: 115 rpm)	2.00	1.77	1.74	1.73	1.72	1.72	1.72
pH (resin dosage: 5.0 g) (stirring speed: 115 rpm)	3.00	2.03	1.98	1.96	1.95	1.94	1.93
pH (resin dosage: 5.0 g) (stirring speed: 115 rpm)	4.00	2.22	2.18	2.17	2.16	2.14	2.11
pH (resin dosage: 5.0 g) (stirring speed: 115 rpm)	5.00	2.35	2.29	2.27	2.25	2.20	2.11
(b)							
Contact time with the resin (min)	0	10	20	30	40	50	60
pH (resin dosage: 5.0 g) (stirring speed: 78 rpm)	3.00	2.84	2.25	2.23	2.22	2.21	2.21
pH (resin dosage: 5.0 g) (stirring speed: 148 rpm)	3.00	2.18	2.17	2.17	2.16	2.16	2.14
(c)							
Contact time with the resin (min)	0	10	20	30	40	50	60
pH (resin dosage: 7.5 g) (stirring speed: 115 rpm)	3.03	2.05	2.04	2.03	2.02	2.02	2.01
pH (resin dosage: 10.0 g) (stirring speed: 115 rpm)	3.02	2.00	1.95	1.93	1.92	1.91	1.91

3.2. *Effect of Contact Time between Resin and Synthetic Solution of Hard Water on Removal of Hardness of Calcium and Magnesium.* The contact time is an important factor in the process of removal of calcium and magnesium from solution

of synthetic hard water. The batch experiments were carried out at different contact times as 0, 10, 20, 30, 40, 50, and 60 min with 5.0 g of the resin and stirring rate of the solution as 115 rpm in the pH range between 2.0 and 5.0. The equilibrium was established for 60 min.

The uptake of Ca(II) and Mg(II) ions and removal of hardness of calcium and magnesium increase rapidly during, respectively, 20 min and 10 min at first then the removal of magnesium hardness increases slowly until the equilibrium state was reached as the active sites on the sorbent were filled by ions of calcium. The obtained results has shown that the equilibrium is reached after 20 min under experimental conditions as mentioned. Figure 2 shows the exchange of hardness of magnesium and calcium from synthetic solutions of hard water with the pH range between 2 and 5.

3.3. *Effect of pH on Removal of Hardness of Calcium and Magnesium.* The pH of the solution of hard water has significant effect on affinity of the resin for calcium and magnesium ions. The sorption of ions of Ca(II) and Mg(II) and removal of hardness of calcium and magnesium from solution of hard water were studied with the resin of Amberlite IR 120 [H⁺] at initial pH range between 2.0 and 5.0. The initial pH of the solution of hard water was controlled with pH meter by adding solutions of hydrochloric acid and sodium hydroxide. In the experiment the temperature, stirring speed, and maximum contact time were kept constant at 298 K, 115 rpm, and 60 min. Figure 3 shows that the removal amount of hardness of calcium and magnesium from synthetic solutions of hard water depends on contact time with the resin phase in the pH range of 2.0 and 5.0.

While the amount of removal of calcium hardness has remarkably increased from 40% to 100%, the amount of removal of magnesium hardness is more restrictive up to about 12% for equilibrium of 20 min at higher pH than 2.0. The maximum uptake capacity for calcium and magnesium has been determined, respectively, as 68 mg/g (pH: 3.0) and 12 mg/g (pH: 3.0). This result has shown that the resin has higher capacity of uptake and selectivity for adsorption of calcium than magnesium. Therefore there is a consideration that the adsorption of calcium ions with anionic functional groups on the surface of the resin has a priority to compare

TABLE 6: Experimental conditions depend on effect of stirring speed for removal of hardness of calcium and magnesium.

pH	Stirring speed (rpm)	Resin dosage (g)	Initial hardness of calcium (FH)	Hardness of calcium in equilibrium (FH)	Initial hardness of magnesium (FH)	Hardness of magnesium in equilibrium (FH)
3	78	5	92.50	59.56 (60 min)	134.35	108.35 (60 min)
3	115	5	83.75	0.00 (60 min)	124.89	96.80 (60 min)
3	148	5	80.05	0.00 (60 min)	124.25	95.00 (60 min)

TABLE 7: Experimental conditions depend on effect of amount of the resin for removal of hardness of calcium and magnesium.

pH	Stirring speed (rpm)	Resin dosage (g)	Initial hardness of calcium (FH)	Hardness of calcium in equilibrium (FH)	Initial hardness of magnesium (FH)	Hardness of magnesium in equilibrium (FH)
3	115	5.0	83.75	0.00 (60 min)	124.89	96.80 (60 min)
3	115	7.5	97.90	0.00 (60 min)	122.50	82.25 (60 min)
3	115	10	93.40	0.00 (60 min)	119.65	33.60 (60 min)

with magnesium and the sorption of magnesium has a disadvantage with less number of binding sites. Tables 4 and 5(a)–5(c) show, respectively, experimental conditions concerned with effect of pH for removing of hardness of calcium and magnesium from synthetic solutions of hard waters and pH values of solution in equilibrium depending on initial pH of the solution and contact time with the resin phase according to experimental conditions.

3.4. Effect of Stirring Speed on Removal of Hardness of Calcium and Magnesium. The effect of stirring speed of the synthetic solution of hard water on removal of hardness of calcium and magnesium was examined with agitation speed between 78 and 148 rpm at pH of the solution of 3.0 with the resin amount of 5.0 g. The results show that while stirring speed of the solution increases the hardness of calcium and magnesium has reduced and as seen in Figure 4 the removal amount of hardness of calcium and magnesium has risen. The results indicate that the agitation speed of the solution of hard water has an impact on adsorption of calcium and magnesium and removal of hardness degrees.

Table 6 shows experimental conditions concerned with effect of stirring speed for removing of hardness of calcium and magnesium from synthetic solutions of hard waters.

3.5. Effect of Amount of the Resin on Removal of Hardness of Calcium and Magnesium. The removal amount of hardness of calcium and magnesium (Figure 5) from synthetic solutions of hard water was examined in relation to the amount of the resin between 5 and 10 g under experimental conditions such that pH of the solution is 3.0 and the stirrer speed of the solution is 115 rpm.

According to the results obtained, the removal efficiency of calcium and magnesium hardness increases with rising

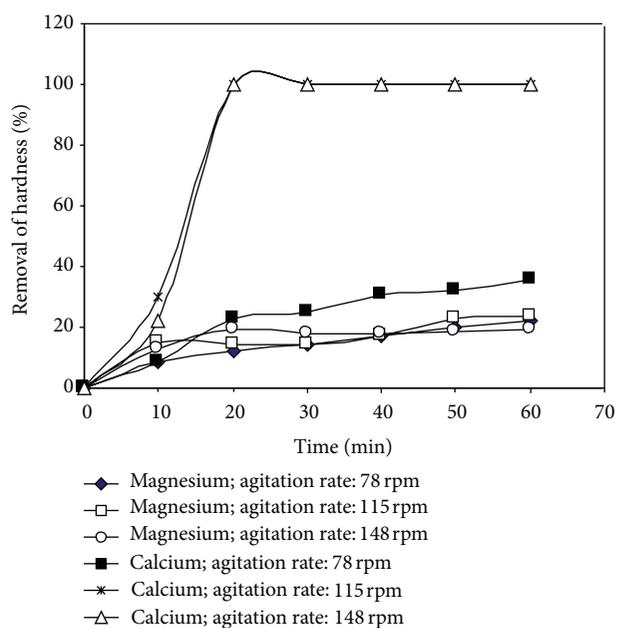


FIGURE 4: The removal of the hardness of calcium and magnesium as a function of agitation speed of the solution (the amount of resin: 5.0 g; the initial pH of the solution: 3.0).

amount of the resin. The amount of the resin provides a great of ion exchange sites to replace of earth alkaline metals for a fixed initial concentration of calcium and magnesium in the solution of hard water. The amount of the resin is an important parameter to obtain the quantitative uptake of calcium and magnesium ions. Table 7 shows experimental conditions concerned with effect of resin dosage for removing hardness of calcium and magnesium from synthetic solution of hard waters.

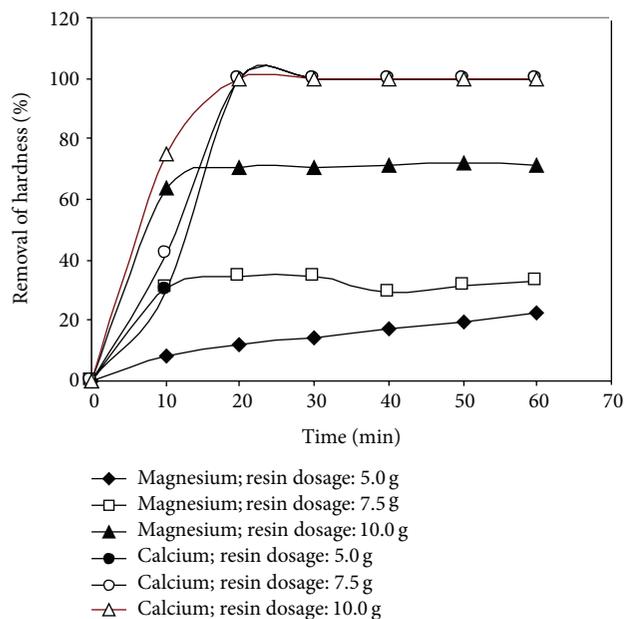


FIGURE 5: The removal of the hardness of calcium and magnesium as a function of amount of the resin (the initial pH of the solution: 3.0; the stirring speed of the solution: 115 rpm).

4. Conclusion

Ion exchange technique has proved to be advantageous over other technologies. Amberlite IR 120, a strong acid cation exchange resin in sodium form, is a low cost ion exchange resin and the experiments have shown that the resin is effective for removal of hardnesses of calcium in the presence of magnesium and softening of waters.

In this study the effects of pH and stirring speed of the solution and dosage of the resin were searched to remove of hardness level of calcium and magnesium as a function of contact time between the resin and solution of hard water. The optimum operation conditions were determined as: pH of the solution is 3.0, agitation speed of the solution is 115 rpm, amount of the resin is 10 g, and ratio of resin/solution is 1 g/100 mL. The maximum removal efficiency was achieved for calcium and magnesium, respectively, as 100% and 70% with contact time of 20 min. The results have shown that the ion exchange rate and capacity of the resin are higher for calcium than magnesium as a competition of sorption of calcium and magnesium and hydrogen ions has occurred for resin sites and exchange of calcium has formed in preference to ions of magnesium and hydrogen. The difference of selectivity of the resin for sorption of calcium and magnesium ions has great effect on removal of hardness of calcium with the pH range higher than 2.0.

This study has been introduced as a method for removal of calcium from aqueous solutions in the presence of magnesium and pH range between 2.0 and 5.0. The feasible results have been reached for removal of hardness of calcium and magnesium of tap water obtained from Research Laboratory of YILDIZ Technical University, Analytical Chemistry Department, Istanbul, Turkey. The resin of Amberlite

IR 120 [Na⁺] can be used for treatment applications including softening.

Conflict of Interests

The author declares that there is no conflict of interests regarding the publication of this paper.

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