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A Comprehensive Method of Ion Exchange Resins Regeneration and Its Optimization for Water Treatment

Sameer Al-Asheh and Ahmad Aidan

Abstract

Ion exchange membranes, specifically resin technology, lie at the heart of electrolytically conductive systems used in the treatment of wastewater. This chapter deals with ion exchange deionization and the effect of resin amount as well as the concentration of acid and base on the product conductivity. The strong acidic cation polymeric exchanger resin is commercially called MERCK 104765 cation exchanger IV with capacity greater than 3.2 mmol/ml, while the strong basic anion polymeric exchanger resin is commercially called MERCK 104767 anion exchanger III with capacities greater than 1.0 mmol/ml. Water conductivity, as an indicator of regeneration efficiency, was monitored over time at the different conditions and scenario. In general, it was observed that the conductivity decreases with time until one point is reached and then starts to increase as a result of resin saturation. It was also noticed that the lowest conductivity is achieved when using 1-vol% NaOH and 5-vol% HCl in the cathodic and anodic resin tubes, respectively, and that water conductivity increases with the increase in the amount of water being used. The amount of resin significantly impacts the deionization efficiency; more ions are removed as the amount of resin increases.

Keywords: ionic resin, cationic resin, NaOH, HCl

1. Introduction and literature review

It is well known that such mineral as calcium and magnesium, in particular, along with iron and manganese cause water harness, the existence of which may cause scaling problems and serious failures in pipelines of boilers and heat-transfer equipment. Also, these divalent ions can react with soap anions and thus decreasing the cleaning efficiency resulting in high consumption of detergents. Temporary hardness is a type of water hardness caused by the presence of dissolved bicarbonate minerals (calcium bicarbonate and magnesium bicarbonate). When dissolved, these type of minerals yield calcium and magnesium cations (Ca^{2+} , Mg^{2+}) and carbonate and bicarbonate anions (CO_3^{2-} and HCO_3^-). The presence of the metal cations makes the water hard. However, unlike the permanent hardness caused by sulfate and chloride compounds, this “temporary” hardness can be reduced either by boiling the water, or by the addition of lime (calcium hydroxide) through the process of

lime softening. Boiling promotes the formation of carbonate from the bicarbonate and precipitates calcium carbonate out of solution, leaving water that is softer upon cooling. Permanent hardness (mineral content) is generally difficult to remove by boiling. If this occurs, it is usually caused by the presence of calcium sulfate/calcium chloride and/or magnesium sulfate/magnesium chloride in the water, which do not precipitate out as the temperature increases. Ions causing permanent hardness of water can be removed using a water softener, or ion exchange column.

Calcium is the most abundant mineral in the human body. Calcium plays vital roles in the structure and function of the human body [1]. It is substantial for intracellular metabolism, bone growth, blood clotting, nerve conduction, muscle contraction and cardiac function [2]. However, there is a significant association between calcium level in drinking water and colorectal, gastric and breast cancer [3]. Magnesium is a naturally occurring mineral that is found in food and other medical products. It is an essential component in the in bones and in muscles and other tissues. However, the too much supplement of magnesium may result in symptoms of toxicity, such as a fall in blood pressure, confusion, abnormal cardiac rhythm, muscle weakness, difficulty breathing and deterioration of kidney function [4].

Therefore, it is important to control the level of hardness in water. Several technologies are available for removal of water hardness (Ca^{2+} , Mg^{2+}), such as electrochemical processes [5], Enzyme catalyzed [6], Nanofiltration [7], electro-dialysis [8], ultrasound [9], ultra-filtration [10], ion exchange [11, 12], membranes [13] and pulsed spark discharge [14]. The two major methods which are typically used to remove hardness form water are lime soda softening and ion exchange softening. The high operating cost of lime soda softening, which is mostly used for municipal purposes [15], is attributed to the production of a large volume of sludge that requires post-treatment, excessive use of chemicals (e.g., lime soda ash, and caustic soda) and the addition of acids for pH adjustment [16]. The ion exchange process is primarily employed for residential water softening. Experimental studies have found the sodium level in softened water was 2.5 times higher than municipal water [17]. Adsorption is one of the few promising alternatives for this purpose, especially using low-cost sorbents. A three-dimensional super absorption polymer were used as a absorbents for heavy metal ions from water and other aqueous solution susficialy. Polymer like polyacrylic acid, polyacryliamide and its derivatives which have functional groups (such as carboxylic, hydroxyl and amide) can be used as absorbents for metal ions removal via the interaction between the metal ions and these groups [18].

Ion exchange resins are classified as cation exchangers, which have positively charged mobile ions available for exchange, and anion exchangers, whose exchangeable ions are negatively charged. Both anion and cation resins are produced from the same basic organic polymers. Classification of ion exchange resins based on their sources is illustrated in **Figure 1**.

In the early 1800s, several scientists discovered the ion exchange process. The ion exchange industry in the United States was born in the early 1900s when cation exchangers were first synthesized. Ion exchange resins are used for many water treatment applications. Of these applications, in terms of the volume of resins used, water softening and demineralization of water are the most significant. In 1905, the German chemist Gans used sodium aluminosilicate materials (*zeolites*) to soften water. These materials can absorb calcium or magnesium cations and liberate sodium ions, the basis of water softening. The zeolite could be regenerated by running concentrated NaCl solution through it to liberate CaCl_2 and MgCl_2 . While aluminosilicates are seldom used anymore, the term zeolite softener is occasionally used for any cationic exchange resin [19]. In 1944, cationic exchange resins were

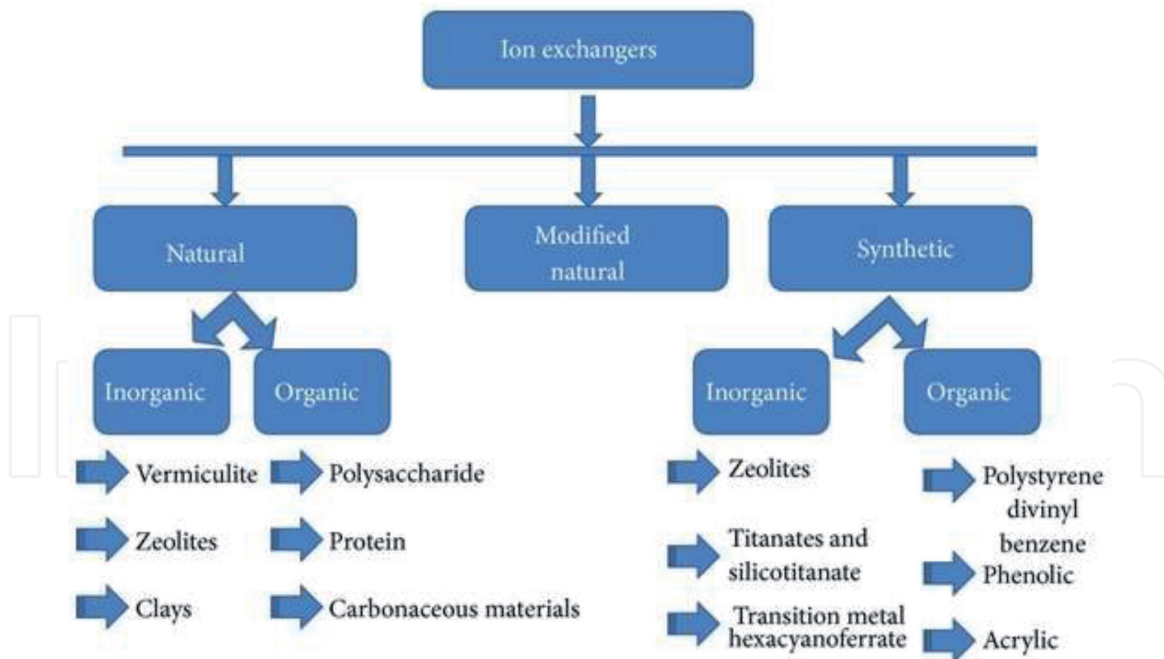


Figure 1.
 Classifications of ion exchange resins.

developed based on polystyrene cross-linked with about 6–8% divinylbenzene. This resin had a much higher capacity than previous resins, meaning that the same volume of resin could exchange many times more ions than previous materials. In 1948, the anion exchange analog was developed. Using these two resins in sequence allows the complete demineralization of water, instead of merely exchanging cations for sodium ions. The macroreticular anion resins that have discrete pores are used first in the sequence to remove organic materials from the raw water that would otherwise cause fouling of the standard gelular resins. Many modifications of the original polystyrene divinylbenzene have been made to accomplish a wide variety of separations [19].

Water softening has been practiced commercially for a century or more, making use of a wide range of natural and synthetic products. As the variety of uses for purified water has increased, so has the need to soften and demineralize water. Demineralization has only been practiced since the discovery of synthetic anion exchange resins in the 1920s. Their usefulness increased greatly with the invention of strongly basic anion exchange resins, which can remove weakly acidic compounds such as silica and carbon dioxide, as well as mineral acids. This process of ion exchange can be used as a simple method to produce water of very high purity. In general, as industrial and domestic requirements have grown, specifications for water quality have become progressively more stringent, and regulations to enforce these have become more strict. Hence the choice of resin types for a particular application becomes increasingly complex [20].

The ion exchange (IX) technology, an already existing technology, is considered an economical and environmentally sustainable method for removing hardness (Ca^{2+} and Mg^{2+} ions removal) [21]. The theory behind the ion exchange (electro-chemical process) is to exchange positive and negative ions in water with hydronium (H^+) and hydroxide ions (OH^-). In order for this process to work, a 1-mm porous bed comprising of insoluble polymers and countless ion exchange sites must be used. When water passes through the bed, positive metallic ions (such as sodium, calcium and aluminum) are exchanged with H^+ , while others such as chloride, nitrate and sulfate will be replaced by OH^- [22]. It can also be used to remove nitrates [23], arsenic [24], dissolved organic carbon (DOC) [25] and

other heavy metal like cobalt [26], nickel, silver zinc and copper [27]. In general, the ion exchange method is vastly adequate in its ability to purify and separate a wide range of chemical compounds in the water treatment field. The medium used in most cases are IX polymer resin that is made of organic polymer structures [21]. Ion exchange process is considered a cost-effective solution and can remove microscale ion particles. However, it does not remove bacteria or pyrogens, and once all exchange sites are used up no ion exchange will occur. Furthermore, any ion exchange unit consists of two main types of resins: Cation exchange resins (CER) and Anion exchange resins (AER). CERs are used to remove cations from water and wastewater, particularly calcium and magnesium ions which are the two main contributors to water hardness [28].

The water from the main water supply enters the softening system and essentially flows through the bed of resin; the working principle of the system is such that the positively charged Ca and Mg ions are attracted to the resin beads [8]. An ion exchange then takes place wherein the Ca and Mg ions are replaced by weaker Na ions. Once the “hard” minerals are fully extracted, the water is termed as “soft” water [29]. As time goes by, the resin beads become saturated with hard ions and need to be regenerated to continue the ion exchange process [29]. Conventionally, the resins are made up of cross-linked polymer chains with ionically active sites and are usually in a bead-like form [30]. The cationic membranes are charged with a bulk of negative ions and available positive counter ions for the ion exchange process [30]. Characterization of these ionic exchange membranes and relating this to their different structures, including their physiochemical properties are reported elsewhere [31]; this involved procuring different types of ion-exchange membranes from different companies namely, Russia, Japan, USA and China. In addition, laboratory samples were also prepared from aromatic polymers and several perfluorinated compounds [32]. The chemical characteristics of the ion-exchange resins are demonstrated by studying the kinetics of a sodium-hydrogen ion exchanger on sulphonated cross-linked polystyrenes [33]. The rate of exchange was measured using an indicator and the results showed a higher rate of ion exchange at a Na^+ concentration greater than 1 N; however, at very high concentrations, the rate of exchange was found to be independent of the sodium concentration and inversely proportional to the particle radius [33].

Ion-exchange provides several advantages over other water treatment methods. Ion exchange process is environmentally friendly, is low-cost since the resin can be fervently regenerated and produces high output [34]. However, it has some disadvantages as well, adsorption of organic matter and calcium sulfate fouling are the most important ones [34]. Calcium sulfate precipitate blocks the resin beads and may also cause blocking of pipes in the system; adsorption of organic matter may reduce resin efficiency, and thus reducing quality of water. Therefore, eventually at one point the resin becomes saturated and ineffective in exchanging ions, and thus must be recharged or regenerated.

Regeneration is the process at which ion exchanging capabilities of saturated resins are recovered. Available regenerators may include salts like NaCl and KCl, acids such as acetic and citric acids, and alkalis including NaOH and KOH. One of the methods to maximize the efficiency of the regeneration process is the flow pattern, in which the regenerator can be introduced from the bottom to the upper side of the ion exchange bed. As a result, the resins at the bottom are cleaner as they undergo more regeneration than the resins in the upper side of the ion exchange unit. Thus, the treated water leaving the exchanger (downward flow) will be in contact with cleaner less saturated resin, which increases the ion exchange efficiency and results in purer water [35]. Ion regeneration is the removal of the ions

that block or plug the internal exchange sites on resins; it includes the following three main steps: backwashing, regeneration and rinsing. The regeneration process is capable of restoring only 60–80% of the resin capacity, as some ions and hardness are retained on the resins. When service starts, these ions might leach off the resin and leave the bed with the treated water effluent, this case is known as leakage [36]. Thus, the regeneration cycle continues until the ions that were removed from the feed water during the service process are recovered from the resins or meet the allowable limits. This can be monitored by electrical conductivity measurements, where the change of electrical conductivity is measured at different volumes of rinsed water during succeeding resin washing cycles. It is expected that after each washing cycle, the electrical conductivity of water will decrease until it reaches distilled water conductivity [37].

Brining process referred to the one when a sodium cycle is used to soften water, where concentrated sodium chloride (NaCl) is passed through the bed to remove calcium and magnesium ions. It can be done in up flow or down flow mode. If sodium cycle is employed to soften water containing the iron ions, this may lead to lowering the resin capacity. This is because iron will be oxidized to the ferric insoluble form in which it blocks the exchange sites on the resin. This can be minimized by performing more frequent and quick regeneration so that iron would not have enough time to oxidize and blocks the sites. Moreover, the process can be enhanced by mixing sodium bisulfate with sodium chloride. This will reduce the insoluble ferric ion to its soluble form [38].

Deionized water has many applications: it can be used as a cooling medium, an agent for lab testing, in car engines and many more other applications [39]. In this work, tap water was deionized using HCl and NaOH as regeneration agents for the cathodic resin and the anodic resin, respectively. The effect of acid and concentrations, amount of water treated and mass of resins on the deionization process will be considered in this work.

2. Material and methods

2.1 Apparatus

The CE 300 Ion exchange demonstration unit (Gunt Hamburg, Germany) is used in this work. The apparatus facilitates tests relating to water softening and demineralization; it is equipped with both cation and anion exchangers with strong and weak basic or acidic contents. The unit layout is illustrated in **Figure 2**. CE 300 enables water deionization with the aid of cation and anion exchangers. The raw water is pumped from the tank into the top of the cation exchanger. In the softening process the water flows from there back into the collecting tank. To desalinate the raw water, it is then additionally routed through the anion exchanger. From there the treated water passes into the collecting tank. In the regeneration process, acid or caustic is fed into the ion exchangers from below using the same pump. The acid and caustic used is collected in the collecting tank. The flow rate of the pump is adjustable, and can be read from a flow meter before it enters the first ion exchanger. For continuous evaluation of the process, a conductivity sensor is installed upstream of the inlet into the collecting tank. The measured values can be read from a conductivity meter. Samples can be taken at all relevant points. Tap water can be used as raw water.

Commercial cation and anion exchangers were provided by Gunt Hamburg (Germany). The strong acidic cation polymeric exchanger resin is commercially called

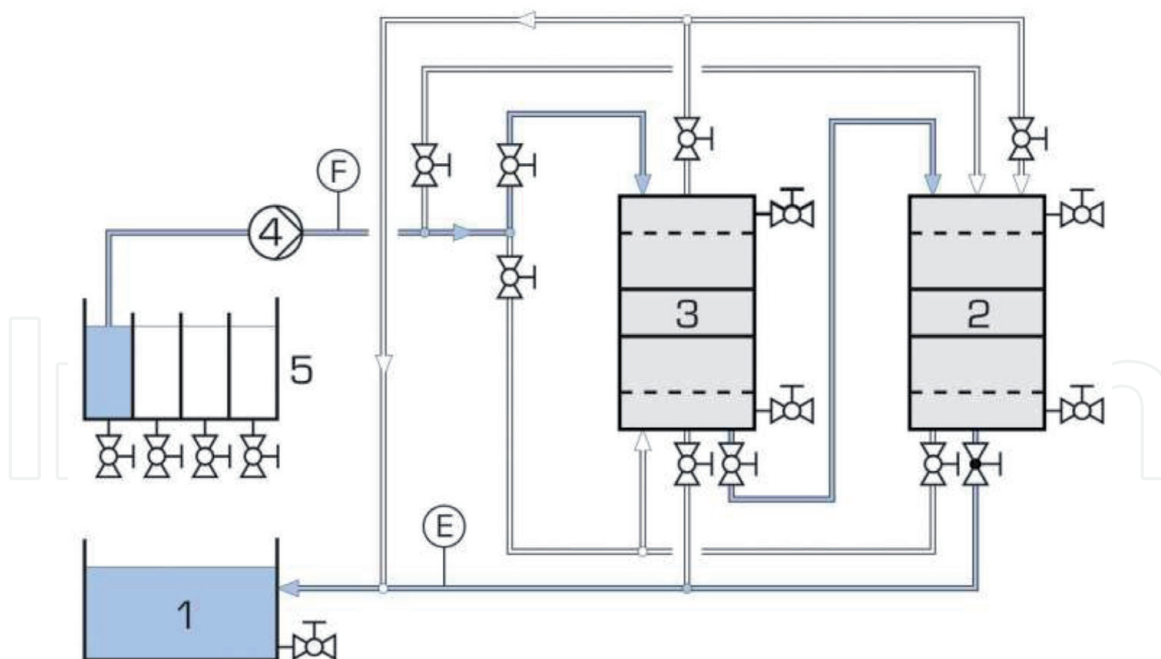


Figure 2.

CE300 unit diagram: Flow path with the two ion exchangers configured in series (desalination): 1 collecting tank, 2 anion exchanger, 3 cation exchanger, 4 pump, 5 raw water tank; E conductivity, F flow rate.

MERCK 104765 cation exchanger IV with capacity greater than 3.2 mmol/ml; while the strong basic anion polymeric exchanger resin is commercially called MERCK 104767 anion exchanger III with capacities greater than 1.0 mmol/ml.

2.2 Methodology

The set-up was prepared by opening/closing valves as described. Solutions of 5 vol% of HCl and 0.1 vol% of NaOH each in 100 mL distilled water were prepared. The cation tube was filled with the 5% HCl solution and the anion tube with the 0.1% NaOH solution; each containing 20 g polymeric resin. The process started by pumping the hard water through the column, and water was allowed to pass through the outlet tubes. Once a steady flow is passing through the outlet, the conductivity of the water at the outlet was recorded at time intervals of 10 seconds. The pump turned off when the conductivity values start to increase until reaching a steady state. The experiment was repeated with other concentrations of HCl and NaOH with 0.5 and 0.1 increments, respectively.

For the purpose of studying the effect of amount of water treated, the process was repeated at certain concentrations of HCl and NaOH with the tubes filled with different amounts of hard water. Also, the effect of amount of resin used was studied by pacing different amounts of resin at the different runs for given concentrations of HCl and NaOH.

3. Results and discussion

3.1 Effects of acid and base concentrations on deionazation

Resin generation is important from cost point of view as well as minimizing solid waste. In this work, resin regeneration using different combinations of acid in the cationic resin and base in the anionic resin, each at different concentrations, was accomplished. Each bed was fed with 20 g of polymeric resin and each filled

with 2 L of hard water. The hard water is municipality tap water with a hardness conductivity of 100,000 μS and a TDS of 600 mg/L. The experiment was started off by putting 0.5 vol% HCl and 0.1 vol% NaOH in the cathodic and anodic resin tubes, respectively; and in every different run the vol% was increased by 0.5 and 0.1 for each of the aforementioned resins. The results for water conductivity at different combinations of acid-base are shown in **Figure 2**, having in mind that the conductivity of purely deionized water is 1.1 micro Siemens (μS).

The experiment was started off by using 1-vol% NaOH and 5-vol% HCl in the cathodic and anodic resin tubes, respectively. It is seen (**Figure 3**) that conductivity starts to decrease indicating that the resin is deionizing the water until a point is reached where the conductivity starts to increase again, indicating that the resins have reached accumulation point. Accumulation point was reached after 5 seconds of running the experiment, and the maximum value of regeneration was found to be 1550 μS . When 0.9 vol% and 4.5 vol% of NaOH and HCl, respectively, were used the conductivity kept decreasing until a value of 1076 μS was reached in 45 seconds. The same trend was noticed when the NaOH and HCl vol% were decreased to 0.8 and 4% respectively; however, in this run a higher conductivity of 2270 was obtained in 45 seconds. This shows that as the amount of resin decreases, the conductivity of water increases indicating that fewer ions were removed. In **Figure 3**, a conductivity of 2140 μS was obtained at 45 seconds, when 0.9 and 4.5% NaOH and HCl, respectively, are used. More time was required to deionize the water for the case of 0.9 and 4.5% NaOH and HCl, respectively; at 45 seconds the conductivity was found to be 2360 μS . Furthermore, the conductivity was found to be 2260 μS at 45 seconds for the case 0.6 and 3.0% NaOH and HCl, respectively. Furthermore,

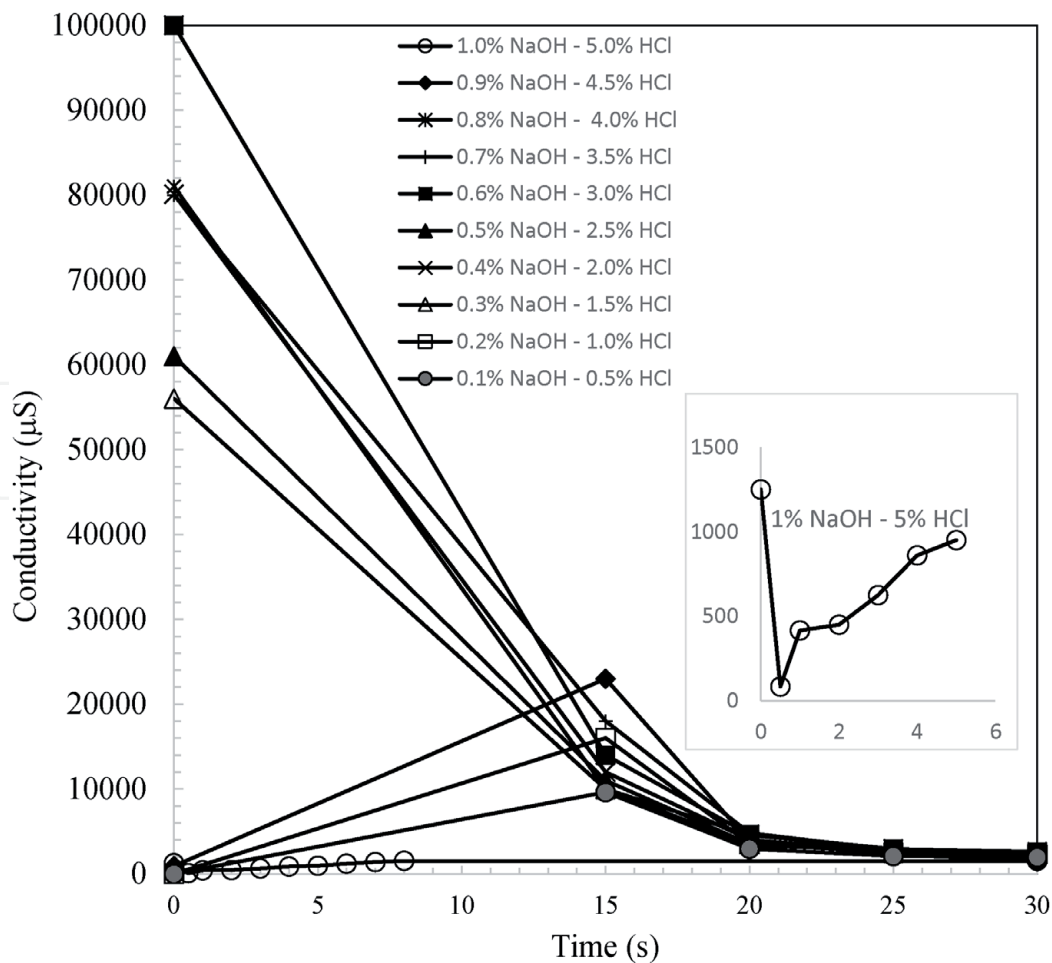


Figure 3.
 Effect of acid-base concentrations on resin regeneration; resin amount: 20 g in both tubes; volume treated: 2 L.

as the vol% decreased, the conductivity started giving similar results at 45 seconds. For example, for the cases of 0.3% NaOH—1.5% HCl, 0.2% NaOH—1.0% HCl, and 0.1% NaOH—0.5% HCl, the conductivity was found to be 2300, 2100, and 1867 μS , respectively, which are close to each other. In conclusion, to have best results, the resin vol% should be high.

3.2 Effect of treated volume on deionization

Effect of treated volume of water was also investigated using same amount of resins in each bed (20 g) and same eluants concentrations in the cationic and anionic resins (5 vol% HCl and 1 vol% NaOH, respectively). The results for water conductivity at different amounts of treated water are shown in **Figure 4**. As shown in **Figure 3** for the case of 1 L, the conductivity started to decrease indicating that the resins are deionizing the passing water. It started off with a conductivity of 864 μS which is the conductivity of tap water and ended with a conductivity of 73.19 μS . When the volume increased to 2 L, the lowest conductivity was found to be 70 μS after 140 seconds have passed. On the other hand, when 3 L of water was added, 1.14 L of water was treated and the lowest conductivity was found to be around 77.2 μS ; after this point, the conductivity increased again and that happened at 70 seconds (**Figure 4**). For the case of 4 L of water being added, an amount of 1.63 L of water was treated. The lowest conductivity was found to be 242 μS and then it started to increase at 70 seconds. This shows that as more water is being treated, the conductivity increases.

3.3 Effect of amount of resin

The effect of amount of resin used in cationic anionic resin tubes on deionization is also studied by putting different amounts of resins in each tube with

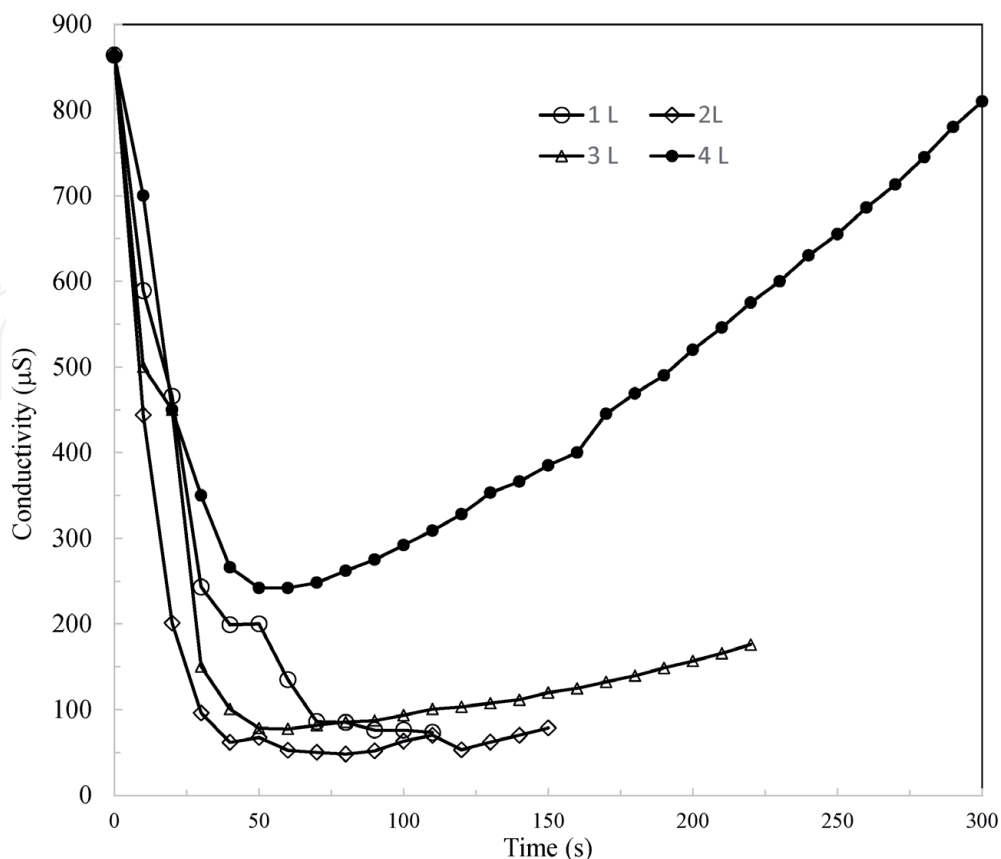


Figure 4. Effect treated water on resin regeneration; resin amount: 20 g in both tubes; 5 vol% HCl—1 vol% NaOH.

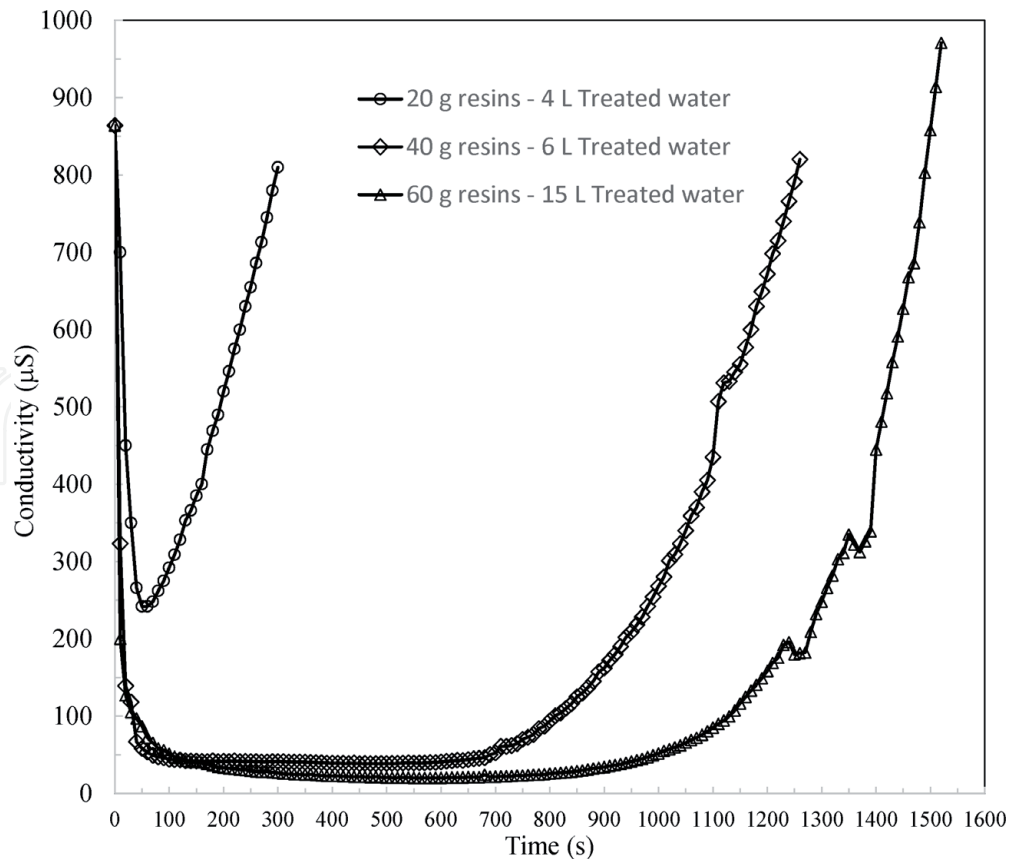


Figure 5. Effect amount of resins on resin regeneration using 5-vol% HCl in cationic resin tube and 1-vol% NaOH in anionic resin tube.

different volumes of hard water as 3 using 5 vol% HCl in the cationic resin tube and 1 vol% NaOH in anionic resin tube. The results are shown in **Figure 5**. The case of 20 g resin is the same as the described in the previous section. When the amount of resin was increased to 40 g, the same trend was obtained as that of the 20 g resin. However, more water was deionized in this case because more resin was used. In other words, it required around 480 seconds to saturate the resin and to stop the deionization process. This shows that increasing the resin amount, helps in increasing the deionization efficiency. The resin amount was again increased to 60 g. The water was being deionized until 560 seconds were reached and a conductivity of 19.8 μS was obtained. After this point, the conductivity started to increase again indicating that the resin has been saturated. As the resin amount increased, the apparatus was able to give better results in terms of removing more ions from the water. For example, at 500 seconds the conductivity was found to be 38.6 and 20.4 μS for the 40 and 60 g, respectively. This shows that the amount of resin is related to the deionization efficiency. As the amount of resin increases, more ions are removed.

4. Conclusions

Water conductivity decreases with the increase in resins concentrations; the lowest conductivity is achieved when using 1-vol% NaOH and 5-vol% HCl in the cathodic and anodic resin tubes, respectively. The results of this work show that water conductivity increases with the increase in the amount of water being used. The amount of resin significantly impacts the deionization efficiency; more ions are removed as the amount of resin increases. The optimization implemented in this

work is considered superior compared to other deionization techniques due to life time and efficiency of the reused resins.

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