Environ Res Tec, Vol. 8, Issue. 2, pp. 245-254, June 2025

Environmental Research and Technology
https://dergipark.org.tr/en/pub/ert
DOI: https://10.35208/ert.1498026

Research Article

Sulfonated plastic waste: A solution for water hardness reduction

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ARTICLE INFO

Article history
Received: 09 Jun 2024
Revised: 24 Jul 2024
Accepted:20 Aug 2024

Key words:
Air bubble plastic resin, ground water, hardness, styrofoam resin, waste plastic

ABSTRACT

This study focused on the utilisation of waste plastic as an ion exchanger to reduce water hardness. The three identical glass columns R1, R2, and R3 were used, each containing different types of waste plastic resin, such as styrofoam resin (SR), air bubble plastic resin (AR), or a mixture of both resins (MR), respectively. The plastics underwent a sulfonation process after crushing and sieving; their sulfonation was confirmed using FTIR analysis. For lab-scale experiments, each glass column was filled with sand, gravel, and coconut fiber as supporting media and equipped with a pump and flow-control valves. Groundwater collected from an urban area was passed through the columns at different flow rates and contact times. The process takes place by exchanging the Na+ ions of resins with polluting ions present in hard water. The treatment parameters involved are total hardness, calcium hardness, magnesium hardness, chloride, TDS, and pH. During the treatment process, pH was maintained between 7 and 8.5. All the parameters were tested twice, and the average of the observations was noted. The results showed that the maximum total hardness reduction achieved in reactors R1, R2, and R3 was 63%, 52%, and 58.6%, respectively, at a contact time of 24 h.

Cite this article as: Patel M, Patel BB, Lad I. Sulfonated plastic waste: A solution for water hardness reduction. Environ Res Tec 2025;8(2) 245-254.

Nomenclature
AR- Air bubble plastic resin
Ca-Calcium
$CaCO_3$- Calcium carbonate
FTIR- Fourier transform infrared spectroscopy
H_2SO_4 - Sulfuric acid
LDPE-Low-density polyethylene
Mg-Magnesium
MR-Mixture of styrofoam and air bubble plastic resin
NaCl- Sodium chloride
SO_3 – Sulfur trioxide
SR-Styrofoam resin
TDS- Total dissolved solids
TH-Total hardness

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INTRODUCTION

Plastic waste has become a major environmental concern in recent years due to its non-biodegradable nature and the enormous amount of waste produced globally. Due to the fact that millions of tons of plastic garbage are produced each year, waste plastic has recently grown to be a significant environmental problem. A lot of the plastic waste still ends up in landfills or pollutes the environment, despite some nation's efforts to limit it by outlawing specific single-use plastics or putting recycling programs in place. Because plastic waste takes hundreds of years to disintegrate and releases hazardous chemicals that can pollute soil and water, it poses a serious risk to both the environment and human health when it is disposed of [1]–[3].

Despite efforts to mitigate its impact through legislation and recycling initiatives, the sheer volume of plastic produced annually continues to pose significant challenges. Of particular concern is polystyrene plastic, widely utilized in various industries for its versatile properties, including hardness reduction capabilities. An evaluation conducted by a previous study shows that the relative importance of reduce, reuse, repair, recycle, recover, and dispose of in a waste management strategy is shown by a six-level pyramid. Among all these factors, the most crucial one is lowering our own desire to purchase ostentatious plastic items [1], [4].

Ion exchange technology actually refers to the fact that when water passes through the ion exchange column, cations and anions in the water are exchanged with H+ ion of the positive resin and OH- ion of the negative resin ion, which is widely used for various purposes, especially pollutant reduction in water and wastewater treatment [5]–[10]. Table 1 highlights some of the previous studies using the ion exchange method, which have attempted to reduce the water hardness using different kinds of waste plastics.

Table 1. Types of waste plastic utilized by different studies with their summary results

Types of hard water sample	Column study or other	Type of waste plastic used	Procedure To check Sulfonation	Achieved hardness reduction	References
Synthetic hard water	Other	On-spec Polystyrene Sample, Waste Polystyrene Off-spec sample and waste plastic	FTIR	Ca: 47- 51% Mg: 68 - 74%	[11]
Synthetic hard water	Other	White coffee cup	FTIR	Ca: 60% Mg: 60%	[12]
Actual ground water	Column	Polystyrene foam	-	Total hardness: 42%	[10]
Synthetic hard water	Other	Virgin Polystyrene and Off spec Polystyrene	FTIR	Ca: 47 - 49%. Mg: 68 - 74%	[13]
Actual ground water	Column	Zeolite	FTIR for aluminium silicate and hydrogen bond	Total :81% Ca: 82% Mg: 84%	[14]
Wastewater	Other	Waste polystyrene	FTIR	Effective Lead and cadmium removal	[15]
Synthetic Hard water	Other	Rice straw	FTIR	Ca: 98%	[16]
Synthetic hard water	Other	LDPE	FTIR	Effective Lead and cadmium removal	[17]

MATERIALS AND METHODS

The study's methodology comprises seven steps. Initially, the reduction of groundwater hardness involved selecting appropriate plastic materials capable of effectively exchanging ions post-treatment. This was achieved by reacting various plastic forms with concentrated H2SO4 to produce resin. Subsequently, FTIR testing was conducted to assess the extent of sulfonation. The ion exchange capacity of the prepared resins was then determined. Following this, lab-scale reactors were set up, and their performance was evaluated with varying contact times. All the parameters were tested twice, and the average of the observations was noted. Experimental work was undertaken to test parameters such as hardness, chloride, pH, and Total Dissolved Solids (TDS) removal.

Groundwater collection and characteristic

Groundwater is pivotal in tackling water scarcity in many developing nations due to its widespread availability, reliability, and cost-effectiveness, making it the preferred choice for various water needs. It is vital for drinking water, particularly in India, where it fulfils a significant portion of both rural and urban water demands. However, the increasing dependence on groundwater has led to its extensive and sometimes unregulated exploitation across the country, posing challenges [18]. Hardness concentrations in natural water vary, with levels ranging from 10 to 500 mg/L as $CaCO_3$ in drinking water [19].

The entire study was conducted using groundwater obtained from the western part of Ahmedabad, an urban area in Gujarat, India. Table 2 shows the characteristics of the collected groundwater.

Table 2. Characteristics of the collected groundwater

Characteristics	Noted Value
pH	7.8
Total dissolved solids (TDS)	1718 mg/L
Total hardness (as CaCO3)	650.3 mg/L
Chloride	653.4 mg/L

Selection of waste plastic and its characteristics

Low-density polyethylene (LDPE) is a lightweight and flexible variant of polyethylene commonly used in plastic bags, packaging films, and flexible bottles, offering moisture resistance. Air bubble plastic, or bubble wrap, contains tiny air pockets and is primarily used for cushioning items during packaging. It can sometimes be recyclable. Styrofoam, or polystyrene resin, provides excellent thermal insulation and chemical resistance but is non-biodegradable and has adverse environmental effects.

Styrofoam and air bubble plastic, both commonly employed in packaging, were sourced from domestic suppliers. They subsequently underwent a thorough cleaning process which aimed to remove impurities such as dust and discoloration.

Resin preparation methods

The preparation of all the three types of resins, i.e., styrofoam resins (SR) (Fig 1a), Air bubble plastic resin (AR) (Fig 1b), and a mixed resin (MR) of styrofoam and Air bubble plastic (Fig 1c), involved the following detailed steps:

1. The styrofoam and air bubble plastic were crushed to produce particles ranging between 0.2 and 0.3 mm in size, each weighing 20-30 grams. This crushing process increased the surface area of the waste plastic, facilitating better interaction with H_2SO_4 during the subsequent reaction.
2. A flask was charged with 100 ml of concentrated H_2SO_4 (98%) and 5 grams of dry crushed plastic. The crushed plastic was mixed with a sulfonating compound, concentrated H2SO4, to undergo sulfonation, which involves adding sulfonic acid groups to the resin [10], [17], [20]. The reaction mixture was agitated, and the duration of agitation was adjusted to optimize reaction conditions. Proper agitation ensured thorough mixing and distribution of the acid throughout the resin.
3. The reaction proceeded until a light brown colour was obtained in the resin, indicating the completion of the sulfonation process.
4. The resulting slurry was filtered using a funnel and washed with 250 ml of distilled water. Filtration separated the sulfonated resin from the reaction mixture, removing solid contaminants and undissolved particles. Subsequently, the sulfonated resin underwent rinsing with 250 ml of distilled water, which helped eliminate any residual H_2SO_4 and other contaminants. pH paper was used to test a portion of the filtrate to ensure complete removal of leftover H_2SO_4 after seven to eight washes.
5. The sulfonated resin was then dried at 40°C for 30 minutes to remove any remaining water content, ensuring that the resin was dry and ready for further processing or analysis.

After the resin, preparation, to check the resin properties and their ion exchange capacity, the degree of sulfonation in SR, MR, and AR was identified using FTIR analysis. As per the research study, the particle size has decreased and the degree of sulfonation has increased. Also, the sulfonation is increased, and the ion exchange capacity of resins is increased [4], [21].

FTIR analysis of AR non-sulfonated (Fig 2a) and sulfonated (Fig 2b) revealed peaks to the polystyrene backbone, SO_3 present at 1190.18 cm-1 also the C-H stretching vibration at around 3032.04 cm-1 and the C=C stretching vibration at around 1465.22 cm-1.

FTIR analysis of SR non-sulfonated (Fig 2c) and sulfonated (Fig 2d) revealed peaks linked to the polyethylene backbone, SO_3 present at 1180.18 cm-1, also the C-H stretching vibration at around 3332.04 cm-1 and the C=C stretching vibration at around 1492.22 cm-1.

After preparing the resin, check the sulfonation of resin which is identified in the FTIR spectra analysis. The sulfonation reaction of SR and AR with sulfuric acid can be represented by the following chemical Eq (1).

After preparing the resin, check the sulfonation of resin which is identified in the FTIR spectra analysis. The sulfonation reaction of SR and AR with sulfuric acid can be represented by the following chemical Eq (1).

$$\underset{\text{Styrofoam}}{C_6H_5\text{-}CH(CH_3)\text{-}[CH_2]n\text{-}CH_2} + \underset{\text{Sulfuric acid}}{H_2SO_4} \rightarrow \underset{\text{Sulfonated styrofoam}}{C_6H_5\text{-}CH(CH_3)\text{-}[CH_2]n\text{-}SO_3H} \quad \underset{\text{water}}{H_2O} \quad \text{Eq (1)}$$

Sulfonic acid groups are found along the polymer chain of the resultant sulfonated styrofoam Resin (SSR). These sulfonic acid groups improve the ion-exchange characteristics of SSR, making it helpful in applications such as ion-exchange resins and catalyst supports. The sulfonation reaction facilitates the addition of sulfonic groups to polymer chains. The reaction mechanism involves the initial generation of sulfur trioxide (SO_3) from sulfuric acid. This process occurs at the interface between the organic solvent and sulfuric acid. Due to steric considerations, sulfonation of the phenyl rings predominantly occurs at the para position [22]. The LDPE structure is mostly intact during the sulfonation process, with sulfonic acid groups. ($-SO_3H$) added as attached groups along the polymer chain. These sulfonic acid groups are denoted as follows Eq (2).

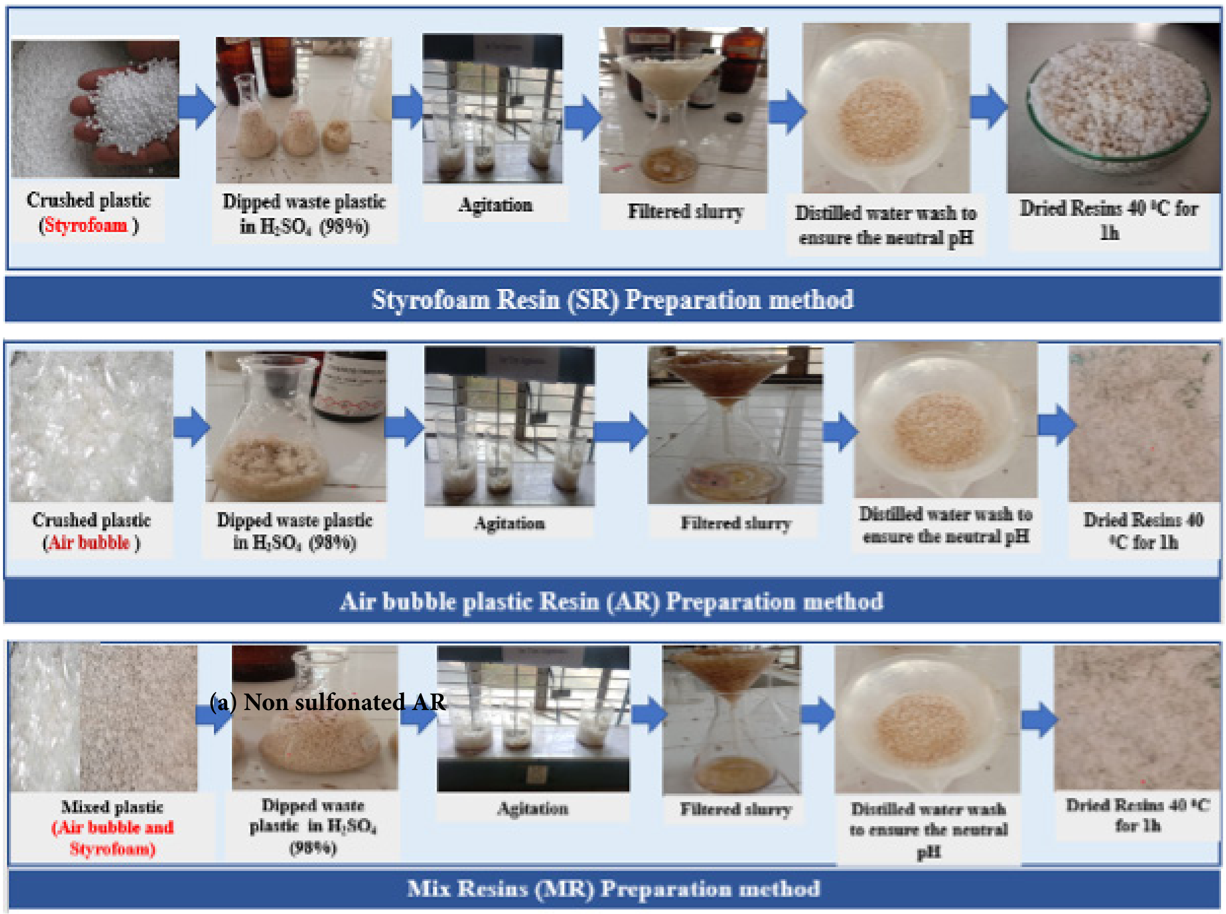
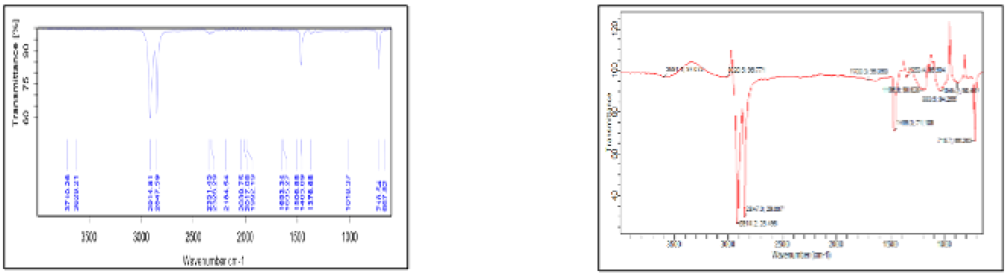
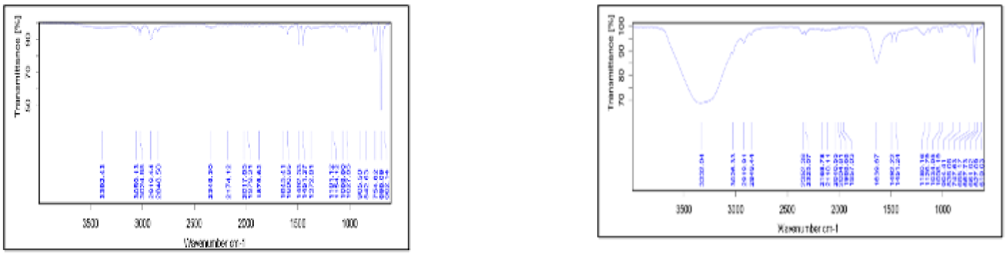


Figure 1. Waste plastic resin preparation method for (a) SR (b) AR and (c) MR



(a) Non sulfonated AR **(b) Sulfonated AR**



(c) Non sulfonated SR **(d) Sulfonated SR**

Figure 2. (a to d). FTIR Graphs showing comparison of non-sulfonated and sulfonated AR & SR

$$(C_2H_4)_n + H_2SO_4 \rightarrow (C_2H_3\text{-}SO_3H)_n \quad \text{Eq (2)}$$

Air bubble plastic   Sulfuric acid   Sulfonated air bubble plastic

Ground water passing from sulfonated and non-sulfonated resins

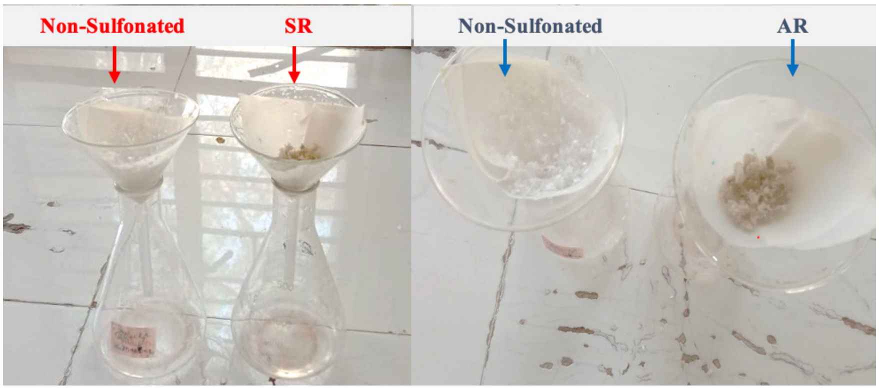


Figure 3. Groundwater pass through the sulfonated and non-sulfonated resins

To verify the efficiency of sulfonated resins, 10 ml of ground water has passed through the sulfonated and non-sulfonated resins (Fig 3). In non-sulfonated resins, only 2% total hardness reduction efficiency was noted, whereas in sulfonated resins, 7%, 9%, and 8% were noted in AR, SR, and MR, respectively, which is due to the sulfonation process of the resins.

Neutralization of the resins

The process of neutralizing sulfonated polystyrene resin involved treating it with a 1 M sodium chloride (NaCl) solution. During this process, sodium ions replace the sulfonic acid groups, resulting in the resin being converted into its sodium form. Neutralization was done by stirring the dried resin in a 500 mL 1 M NaCl solution for 2 h for to provide contact between the resins, and solution for neutralization to occur similarly to the previous studies [11], [16], [23]. Sulfonated Styrofoam Resin is then neutralised after sulfonation by treating it with sodium chloride (NaCl), which is a commonly used base for neutralisation in Eq (3).

$$C_6H_5\text{-}CH(CH_3)\text{-}[CH_2]n\text{-}SO_3H + NaCl \rightarrow C_6H_5\text{-}CH(CH_3)\text{-}[CH_2]n\text{-}SO_3Na + H_2O \quad \text{Eq (3)}$$

Sulfonated Styrofoam   Sodium Chloride   Neutralized Styrofoam   water

The sulfonic acid group (-SO3H) is transformed into a sulfonate ion (-SO3Na) in this reaction by accepting a sodium ion (Na+) from the base Eq (4). As a by-product, water (H2O) is generated.

$$(C_2H_3\text{-}SO_3H)_n + NaCl \rightarrow (C_2H_3\text{-}SO_3Na)_n + HCl \quad \text{Eq (4)}$$

Sulfonated AR   Sodium Chloride   Neutralized AR

The earlier study also attempted the sulfonation of crushed waste polystyrene, which attaches the sulfonic groups to polymer chains. The noted degree of sulfonation was 16%, and the cation exchange capacity was 0.8 meq/100 g of clay. The adsorption isotherms of resin indicated that the cation exchange capacity of polymers was 50 and 130 meq/100 g for lead and cadmium, respectively, with simulated wastewater. Observed greater than 70% lead and cadmium removal [24].

Fabrication of the three glass reactors

The experimental investigation utilised a lab-scale arrangement comprising three identical glass reactors or columns labelled R1, R2, and R3, interconnected in parallel. The columns, with a height of 50 cm and a diameter of 3 cm, were partitioned into inlet storage, media storage, and outlet storage sections. Various resins, namely SR, AR, and MR, were employed within the media compartments to address water hardness via ion exchange. Specifically, R1 was packed with SR, R2 with AR, and R3 with MR.

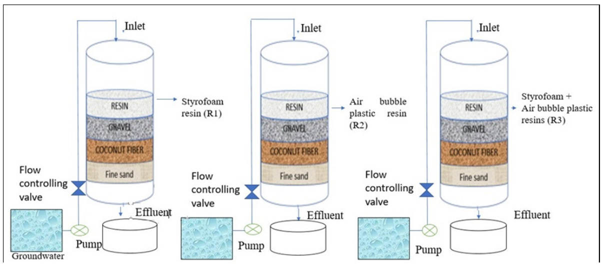


Figure 4. Experimental Set up for the study

Fig 4 is the drawing of reactor where the inlet is provided through the pump and the flow control valve (nob) is provided. Where the 5 cm of each media is filled with resin. The outlet is also provided, which is collected in a container.

Types of media used for resin support

The support media in the experiment included sand (Fig 5a), gravel (Fig 5b), coconut (Fig 5c), and cotton. These materials were chosen because of their substantial surface area, adsorption capacity, ion exchange capability, and chemical inertness.

(a) Gravel (8.25 mm) (b) Sand (1.25 mm) (c) Coconut Fiber

Figure 5. Types of support media used in the reactors to fill the prepared resin

Experimental run hardness reduction

In this study, three identical columns were used: R1 with SR, R2 with AR and R3 with MR to simulate the treatment of groundwater. As support, cotton was placed at the bottom and then layers of sand, coconut husk, and gravel were put in equal column heights of 5 cm each, as shown in Fig 6. To maintain a consistent flow of ground water through the resin column, a pump (DC 6-12V) was employed. Plastic containers were placed at the inlet and outlet. A flow monitoring valve was used to regulate the groundwater's flow rate, and a pump was utilized to move the water through the tank's intake. The experimental work was done in the batch process considering different time intervals of 2, 3, 5, 6, 8, 10, 12, 14, 18 and 24 h. Parameters like total hardness, calcium (Ca) hardness, magnesium (Mg) hardness, chloride, pH, and TDS were measured from the inlet and outlet. All the parameters were tested twice, and the average of the observations was noted.

Table 3 shows the different parameters and media used in this study, as well as the different contact times for the experimental runs.

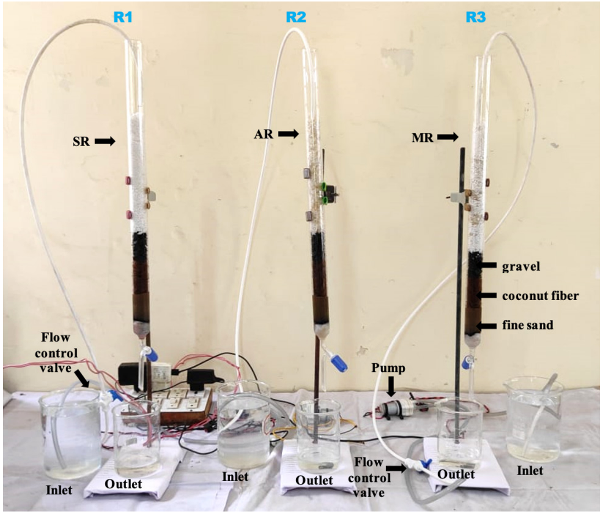


Figure 6. Working reactor during experimental work

Table 3. Experimental work details

Sr. no.	Parameters	Value
1	Flow rate (ml/h)	50-709
2	Contact time (h)	24,18,14,12,10,8,6,5,3,2
3	Reactor depth (cm)	50
4	Reactor diameter (cm)	3
5	Volume of reactor	1413 cm^3
6	Filter media (as per length)	5 cm
	Sand	5 cm
	Coconut Husk Gravel	5 cm
	Resin	5 cm

RESULTS AND DISCUSSIONS

Experimental runs for hardness reduction

The experiments for hardness reduction involved varying contact times and flow rates. The study began with a 24 h contact time and progressively reduced it to 2 h. Flow rates ranged from 59 to 709 ml/h, managed through flow control valves and pumps. The experimental run has been done in two trials, in the first trial (T1), which was started with 24 h and reduced to 10 h. At the 10 h contact time, the resin capacity is exhausted, as the observed water quality of the inlet and outlet was similar. Therefore, after 5 experimental runs, fresh resins were placed, and a further second trial (T2) from 8 h to 2 h contact time has been taken.

In trial T1, with contact times ranging from 24 h to 10 h, Reactor R1 (filled with SR resin) achieved the highest hardness reduction, starting at 63% at 24 h and decreasing to 5% at 2 h. Reactor R3 (MR resin) followed with 58.6% at 24 h, dropping to 3.9% at 2 h, and Reactor R2 (SR resin) reduced total hardness by 52% at 24 h, falling to 2.5% at 2 h. Trial 2, with shorter contact times of 8 h to 2 h, mirrored these results: R1 showed a 50.71% reduction at 8 h, decreasing to 5.9% at 2 h; R3 achieved a 50.6% reduction at 8 h, dropping to 3.9% at 2 h; and R2 recorded a 42.5% reduction at 8 h, falling to 2.5% at 2 h. These findings highlight the significant impact of contact time on the effectiveness of the resins, with SR resin consistently outperforming the others across both trials. In the past column study, wastes of polystyrene were prepared from the foam of food packaging, and air bubble plastic also showed a 40–45% reduction in the hardness of ground water [19].

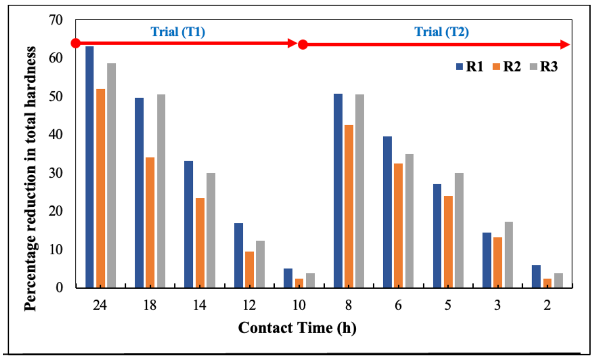


Figure 7. Effect of various contact time with percentage total hardness reduction

During the entire experiment, pH observed into the neutral range. Fig 8(a) represents the capacity of calcium hardness reduction in R1, R2, and R3. In the first trial (T1), the maximum calcium hardness reduction observed in R1, R2, and R3 was 58.6%, 49%, and 36.45%, respectively, at 24 h of contact time. In the second trial (T2), started with a contact time of 8 h. In trial 2 (T2), with an 8 h contact time, reactor R1 reduced calcium hardness by 36.16%, R2 by 33.47%, and R3 by 33.58%.

In trial 1 (T1), after 5 runs, reactor R1, filled with SR, showed the highest magnesium hardness removal capacity at a 24 h contact time, achieving a 54% reduction. In contrast, R2 with AR achieved a 45% reduction, and R3 with MR achieved a 49% reduction under the same conditions. In trial 2 (T2), with an 8 h contact time, R1 achieved a 36.13% reduction, R2 a 30.13% reduction, and R3 a 34.26% reduction in magnesium hardness removal capacity (Fig 8(b)).

Fig 9(a) illustrates chloride reduction in reactors R1, R2, and R3 with varying contact times. In trial 1 (T1), after 5 runs, the highest chloride removal capacity was observed in R1 filled with SR, achieving a 39.38% reduction at a 24 h contact time. In comparison, R2 with AR achieved a 24.6% reduction, and R3 with MR achieved a 22.77% reduction under the same conditions. In trial 2 (T2), with an 8 h contact time, R1 achieved a 14.08% reduction, R2 a 12.20% reduction, and R3 a 12.60% reduction in chloride reduction. These results suggest that longer contact times lead to more effective removal, as observed in the experiments.

Fig 9(b) displays TDS reduction in three reactors with varying contact times. In trial 1 (T1), after taking 5 experiment runs, the highest TDS removal capacity was observed in R1 filled with SR, achieving a 20.52% reduction at a 24 h contact time. In comparison, R2 achieved a 16% reduction, and R3 attained a 14.02% reduction under the same conditions. In trial 2 (T2), with an 8 h contact time, R1 achieved a 23.52% reduction, R2 a 14% reduction, and R3 a 15.02% reduction in TDS removal. These results indicate that the second trial yielded the best results in TDS reduction.

Breakthrough point of resin

At the experiment's outset, the outlet hardness level was lower than the inlet due to the initial high efficiency in the three columns. However, over time, the outlet hardness increased, likely due to the decreasing ion exchange capacity. Eventually, the outlet hardness equaled the inlet, indicating that the ion exchange capacity was depleted, with the resins fully saturated with Ca+2 and Mg+2 ions. The breakthrough point, when the outlet concentration reaches 650 mg/L (groundwater hardness), signifies the need for resin replacement. In this study, the second trial with different or reduced contact times aimed to assess the resin's removal capacity.

Table 4 demonstrates that this study surpasses previous research because, typically, ion exchange resin exhaustion occurs after 2 to 3 cycles. However, in the present study, it's evident that the resin loses its ion exchange capacity after completing five cycles or experimental runs.

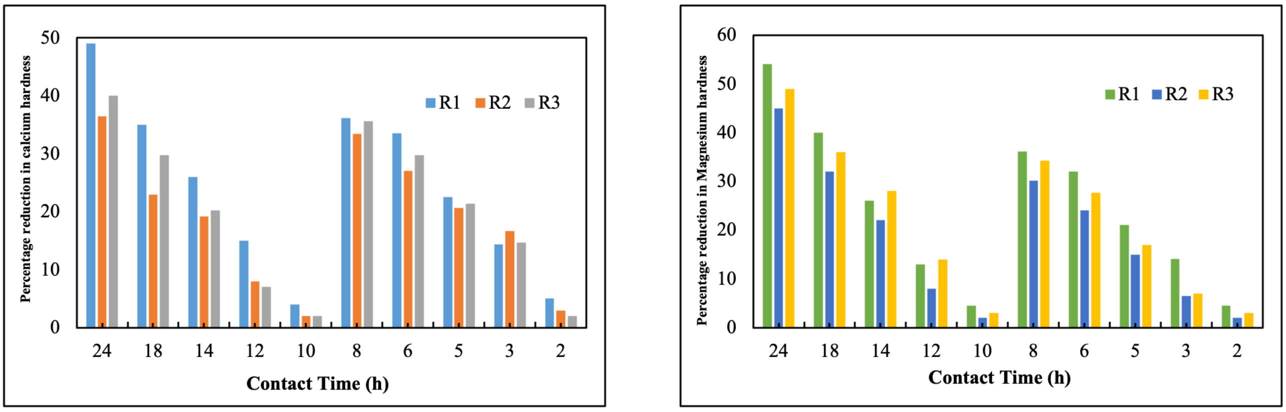


Fig 8. Percentage reduction at varied contact time (a) Calcium hardness (b) Magnesium hardness

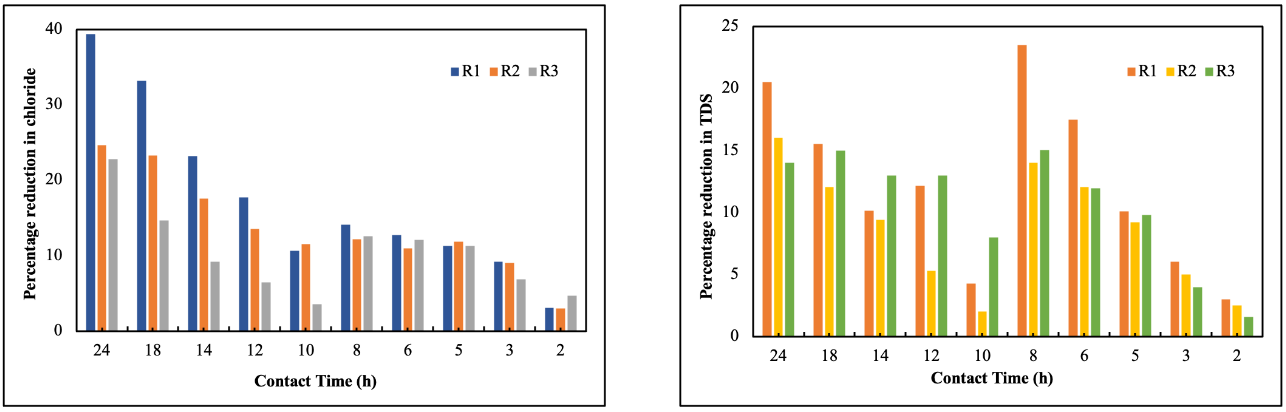


Fig 9. Percentage reduction at varied contact time (a) Chloride (b) TDS

Table 4. Comparison of resin exhausted their ion exchange capacity

Sr. No.	Resin	Flow	Exhausted resin	References
1	Food packaging	1 l/min	After 1 cycle 42.5% reduction in TH	[10]
2	White coffee cup	2.5 l/h	After 3 cycles	[15], [25]
3	Polystyrene	51 and 34 ml/min	After 2 cycles 42.5% reduction in TH	[26]
4	Air buble and styrofoam	50-140 ml/h	After 5 cycles	This study

The study concluded that reactor R1, filled with SR resin, was the most efficient in reducing groundwater hardness, followed by R2 and R3. These results suggest that using waste plastic for hardness reduction is a promising approach, offering a sustainable solution for both plastic waste management and water quality improvement.

CONCLUSIONS

The experimentation involved activating three types of resins (SR, AR, and MR) using styrofoam and air bubble plastic waste through sulfonation, as confirmed by FTIR analysis. The sulfonated compounds were neutralized by sodium salt structures. Three reactors (R1 with AR, R2 with SR, and R3 with MR) were analyzed at various contact times, i.e. starting at 24 h and being reduced to 2 h to determine hardness reduction efficiencies. After five experimental runs, resin replacement was necessary due to hardness reduction capacity exhaustion.

Results showed maximum total hardness reduction efficiencies at 24 h contact time (63% in R1, 42% in R2, and 58.6% in R3). Subsequent runs with an 8 h contact time revealed total hardness efficiencies of 50.71% in R1, 42.5% in R2, and 50.6% in R3. Initial calcium hardness reduction capacities were 49% in R1, 36% in R2, and 40% in R3, with subsequent runs yielding 36.16%, 33.47%, and 35.58%, respectively. Magnesium hardness removal capacities were highest in the first trial (54% in R1, 45% in R2, 49% in R3) and lower in the second trial (36.13%, 30.13%, and 34.26%, respectively).

A neutral pH range was noted throughout the entire experiment run. The most significant chloride reduction was achieved in R1, which was filled with SR, showing a 39.38% reduction at 24 h of contact time. In comparison, R2 with AR achieved a 24.6% reduction, and R3 with MR achieved a 22.77% reduction under identical conditions. These findings indicate that longer contact times enhance chloride reduction efficiency in all three reactors. Regarding TDS reduction, R1 with SR demonstrated the highest, achieving a 23.52% reduction at 8 h contact time. R2 showed a 14% reduction, and R3 exhibited a 15.02% reduction in TDS.

The results suggest that waste plastic can serve as a viable solution for hardness reduction while concurrently addressing the challenge of plastic waste management.

DATA AVAILABILITY STATEMENT

The author confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

USE OF AI FOR WRITING ASSISTANCE

Not declared.

ETHICS

There are no ethical issues with the publication of this manuscript.

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