

Decomposition of Spent Ion Exchange Resin Using Acid digestion, Fenton and Fenton-like Process

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ABSTRACT

This research used the Fenton-like oxidation method to decompose the cationic and anionic resins. Acid digestion and the fenton method were also studied to compare the results. Acid digestion and Fenton methods did not show desirable results, so the research was focused on Fenton like process. For this purpose, Cu^{2+} was used as a catalyst and H_2O_2 as an oxidant. Five parameters including temperature, pH, Cu^{2+} concentration, H_2O_2 addition flow rate, and time were studied to obtain the optimum value for the decomposition of fresh resin. It was found that the optimum values were as follows: temperature: 75°C , Cu^{2+} concentration: 0.3 M, H_2O_2 addition flow rate: 0.5 ml/min for cationic and 0.25 ml/min for anionic resin, and time: 2 hours. By optimization of the parameters decomposition weight percent of 88% was obtained for cationic resin and 83% for anionic resin. The optimum condition was applied for the actual sample, a mix of cationic and anionic resin from the Tehran research reactor. The decomposition percent of 81% was achieved. The resins were characterized before and after decomposition by FTIR and SEM, which showed the decomposition. COD analysis showed that only 2% of decomposed resin was maintained in the solution, and 98% was converted to CO_2 and H_2O .

Keywords: Fenton; Fenton like; Acid digestion; Decomposition; Resin

1. Introductions

In nuclear facilities' water treatment systems, organic ion-exchange resins have usually been used to reduce corrosion or system components' degradation and remove radioactive contaminants [1-3]. In other words, granular and powdery ion exchange resins are widely used in equipment such as condensate demineralizers, condensate filter demineralizer systems, reactor water purification systems, and effluent treatment systems [4].

Ion-exchange resins are a kind of large molecular polymer composed of three-dimensional mesh structures based on styrene monomer (C_8H_8) and cross-linked with divinylbenzene ($\text{C}_{10}\text{H}_{10}$), functional group, and the opposite charges on the group. They can be divided into cationic exchange resins and anionic exchange resins according to their functional groups [5]. Resins suffered from agents oxidizing, high temperature, the catalytic

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effect of metal ions, adsorbed species [6], and mechanical destruction, which could lead to deterioration of the structure of resins [7].

The spent radioactive ion exchange resins have been produced during the operation of the nuclear facilities in the nuclear industry. After a long time of usage, resins are considered as intermediate and low-level radioactive wastes, which have a variety of contaminants, including radionuclides (strontium-90, cobalt-60, cesium-137, uranium-235, uranium-238, and so on), metals (e.g., Ca, Fe, and Mn), and chemicals. Therefore, the treatment and disposal of spent radioactive resins is very important to minimize their potential hazard to human health and the ecological environment. The spent resins must be treated as radioactive solid wastes due to national regulations [8].

At present, many nuclear power plants temporarily store a large number of spent radioactive resins (the volume of spent ion exchange resins that arise from a typical nuclear power plant is 5–7 m³ reactor unit⁻¹ year⁻¹) that need to be processed in stainless steel barrels causing a large volume of the solid radioactive wastes. Because of the radiation effect and biological and chemical toxicity of wasted spent radioactive resins on the environment and animals, safe treatment and disposal is necessary to reduce the storage volume and stabilize the radioactive contaminants. In other words, two main strategies are for treating spent radioactive resins. First, the degradation of the organic materials to reduce the volume and produce an inorganic intermediate product, which may or may not be further conditioned, suitable for long-term storage and/ or disposal, and second direct immobilization in a matrix such as cement, bitumen, or polymers. The spent resins are radioactive wastes for which there is no satisfactory industrial treatment [9]. Solidification or immobilization (e.g., cementation, vitrification, bituminization, and plastic solidification) is a traditional treatment

method for spent ion exchange resins. However, solidification has some disadvantages, such as releasing explosive gases (H₂, CH₄), eroding the container, and swelling of resin [9].

Thus, several methods to reduce and stabilize the radioactive waste before immobilization have been studied, e.g., Dry oxidation [10] and Wet oxidation [10]. Dry oxidation, such as pyrolysis and incineration [10], performs well on waste volume reduction. However, a gas treatment system is needed to prevent the emission of harmful substances such as sulphuric or nitric oxides. Additionally, radionuclides (¹³⁷Cs, ¹⁰⁶Ru, etc), which are volatile at temperatures above 800 °C, must be recovered. Moreover, resins tend to form large clusters by melting before burning, thus causing corrosion problems with the refractory. Embedded resins may also undergo radiolytic or chemical degradation, which can result in the emission of hazardous species in the environment. Wet oxidation such as acid digestion [11], Fenton oxidation [12], supercritical water oxidation [13], radio-sensitive photocatalysis [14], the hybrid process of Fenton dissolution followed by sonication and/or wet air oxidation [15, 16] have been developed to overcome these issues. Some of these techniques have disadvantages such as special requirements of container (especially for acid digestion), high temperature and pressure required in supercritical water oxidation, extra energy consumption, high cost of treatment facility, etc.

Therefore, for improvement in efficiency and safety, more efforts are needed. Today the most effective method for spent ion exchange resin treatment is the Advanced Oxidative Process because of its moderate operating conditions and significant volume reduction. The Fenton reaction (H₂O₂/Fe²⁺) is one of the most used Advanced Oxidation Processes due to its low cost and ease of operation [17, 18]. Fenton and Fenton-like

reactions degrade organic resins and reduce the radioactive residue volume and the disposal cost significantly [17, 19].

The treatment of the spent resins by Fenton's process may reduce costs of immobilization regarding final disposal and transport by 50% compared to direct immobilization [20]. The efficiency of Fenton and Fenton-like reactions (resin degradation) depends on the nature of the resins and operating conditions [21-23]. Fenton and Fenton-like reactions encompass peroxides (usually H_2O_2) reactions with metal such as iron or copper ions to form active oxygen species that oxidize organic or inorganic compounds. The oxidation of ferrous to ferric ions to decompose H_2O_2 into hydroxyl radicals is usually considered as the core of the Fenton chemistry. In other words, Fenton and modified Fenton reactions use a metal catalyst to generate hydroxyl radicals from hydrogen peroxide [24, 25], with various methods being used to deliver the catalyst into the reaction system. Anodic Fenton treatment (AFT) was developed to provide a continuous delivery of ferrous ions from a sacrificial iron electrode by electrolysis [26]. At the same time, hydrogen peroxide was simultaneously pumped into the system.

Investigation of Fenton reaction indicates resin degradation occurs through the interaction of functional groups, a cross-linking agent, and a styrene unit with Fenton's produced radicals [27]. In addition to resin degradation, the Fenton reaction was efficiently utilized in the wastewater treatment to remove many hazardous organics in recent years [28-29].

The recent work investigated the degradation of spent cationic and anionic resins by the Fenton-like method for chemical oxygen demand (COD) removal and weight reduction. The effects of various parameters such as Cu^{2+} concentration, initial pH, temperature, time, and H_2O_2 dosage on resin degradation were studied.

2. Materials and method

Strong acid and strong base resins were used in this work (polystyrene cross-linked with divinylbenzene). They were Purolite A400 and Purolite C100. The spent resins were also prepared from Tehran research reactor of. The other chemicals were analytical grade and were purchased from Sigma-Aldrich. The chemicals include sulfuric acid, nitric acid, hydrochloric acid, hydrogen peroxide, sodium hydroxide, Iron (II) sulfate hepta-hydrate, and copper (II) sulfate.

The resin was dried to a constant weight before using in the process. A 250 mL, three-necked round-bottom flask with a mechanical stirrer was used for the process (Figure 1). An oil bath was used to control the process temperature. A condenser was joint the flask to achieve the reflux condition. The other necks were used for checking the temperature by a thermometer and adding the hydrogen peroxide, which was added by a determined flow rate during the decomposition process by a peristaltic pump. The resin with determined weight and the catalyst solution were placed in the flask before raising the temperature. It was stirred for 30 minutes at room temperature, and then the temperature was increased. When the temperature reached the desired value, the peristaltic pump was turned on to transfer the H_2O_2 into the flask drop by drop. The released gases were collected or neutralized in a base solution after passing through a gas trap. After a determined time, the heater was turned off. When the mixture was cooled down, the resin was separated from the solution. The weight of residual was determined at the end of the process after drying as was done before the process.

The resin weight was compared with the weight before the process, and the degradation percent was calculated. The analytical methods used to investigate the decomposition process were SEM, COD, and FTIR. The chemical oxygen demand (COD) for the decomposed resin was determined

with Hack 1000. The SEM analysis was carried out by a Zeiss Evo 18 instrument. FTIR was recorded by a Vector-22 (Bruker) spectrometer. ICP analysis was done on a Varian vista-PRO spectrometer.

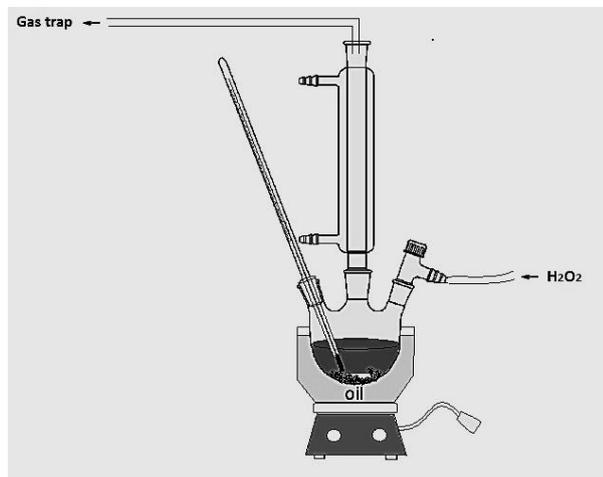


Fig. 1. Schematic diagram of experimental set-up for Fenton-like oxidation.

3. Result and Discussion

The work was performed using fresh resins at first, and then the optimum condition was applied for spent resin. Acid digestion method was used to decompose the resins and the results were compared with Fenton and Fenton-like process. For this purpose, mixtures of two acids were used. Two processes were used to investigate acid digestion. In the first process, a mix of sulfuric and nitric acid was used, and in the other process, a mix of hydrochloric and nitric acid was used (The ratios of sulfuric and hydrochloric acid to nitric acid were 3:1). The process was investigated in various times and various temperatures up to 95°C. The maximum decomposition percents were obtained by a mix of nitric and hydrochloric acid which were below the 25%.

Fenton process was also studied for the decomposition of the resins. In this process, 2 g resin and 15 ml iron (II) sulfate solution (0.3 M) were placed in the flask, and 10 ml hydrogen peroxide was added with a flow rate of 0.5ml/min.

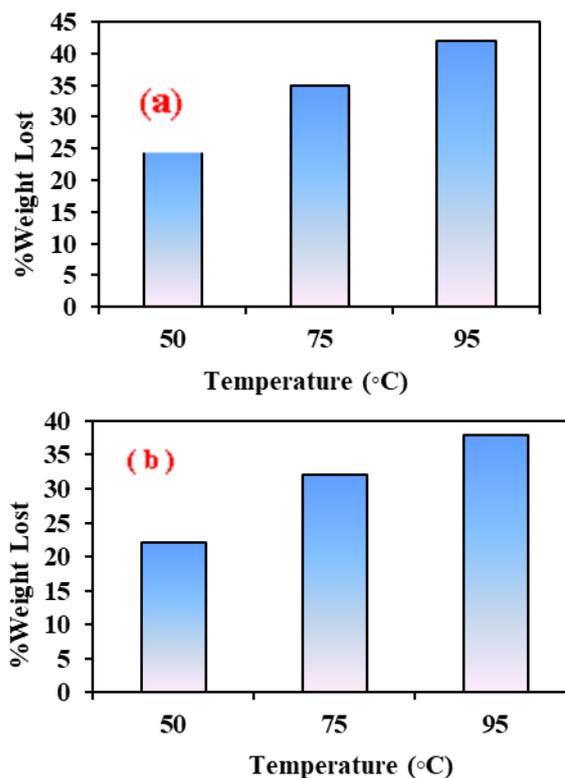
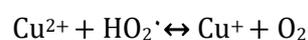
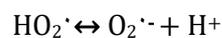
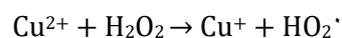


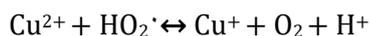
Fig. 2. The temperature effect on decomposition percent in Fenton process for cation, **a.** and anion, **b.** resins.

As it can be seen in the figures the maximum decomposition percent for cationic resin was about 43%, and for anionic resin was about 39%. Since the decomposition percents were not desirable compared with Fenton-like process (It was compared in primary experiments), the Fenton process was stopped, and the effect of parameters was not studied.

According to the result of preliminary experiments the Fenton-like process using Cu^{2+} as a catalyst showed more efficiency, so the work was continued by focusing on Fenton-like process.

The Fenton-like process is mainly based on the production of hydroxyl radicals ($\text{HO}\cdot$) which lead to starting a series of reaction as follows:





The organic structure of the resins can react with hydroxyl radicals ($\text{HO} \cdot$) and peroxide radicals ($\text{HOO} \cdot$). At last, the resins can be decomposed mainly into carbon dioxide and water.

The selected parameters for investigation were Hydrogen peroxide volume, temperature, pH, Cu^{2+} concentration, the flow rate of hydrogen peroxide addition, and time. Except for the pH, which was studied in three levels, the other parameters were

studied in four levels. It was some restriction for various pH ranges. As low pHs were not desirable in large scale process, they were not studied. In high pHs also, precipitation of catalyst was occurred.

Figure 3 (a-b) shows the hydrogen peroxide volume effect on the decomposition percent of the resins. The other parameter values were selected according to our previous experience. Cu^{2+} concentration was 0.3 M, pH was adjusted at 2, the process time was 2 hours, the temperature was 95°C , and the hydrogen peroxide addition rate was 0.5 ml/min.

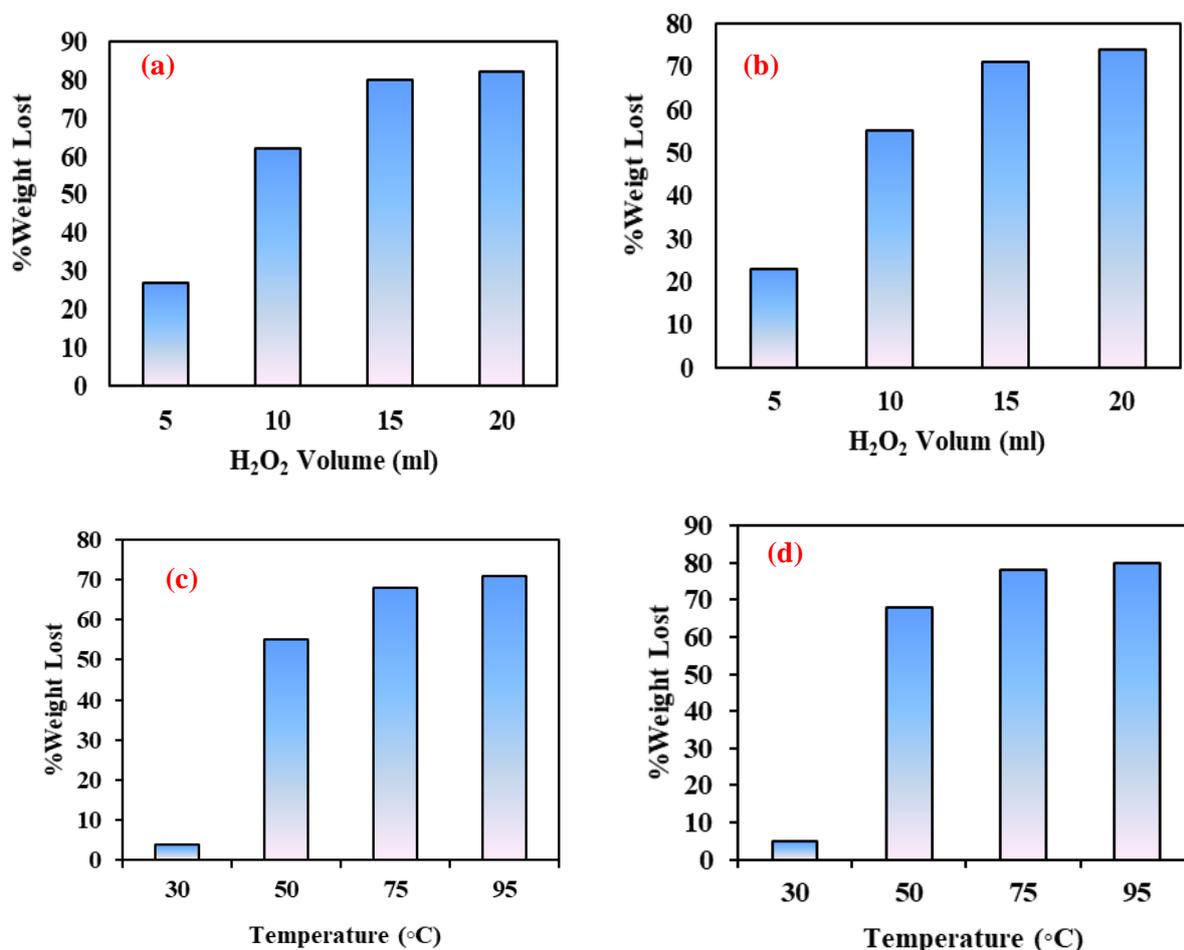


Fig. 3. The hydrogen peroxide effect on decomposition percent in Fenton-like process for cationic, **a.** and anionic, **b.** resins, the temperature effect on decomposition percent in Fenton-like process for cationic, **c.** and anionic, **d.** resins.

As can be observed in the figures, 15 ml of hydrogen peroxide is sufficient for the process, and more volume does not affect the decomposition percent remarkably. Although more volume may increase the decomposition slightly, it increases the process cost at a large scale. On the other hand, more hydrogen peroxide will produce more secondary waste.

The temperature effect on the process was studied in mentioned condition with 15 ml hydrogen peroxide. It was studied in four temperatures. Figure 3 (c-d) shows the results for both cationic and anionic resins.

According to the figures, increasing the temperature enhanced the decomposition percent, but. Still, it was not remarkable in the 75-95 °C

range, so the temperature of 75 °C was considered. According to the Arrhenius equation, increasing the temperature provides the energy required to overcome the activation energy and accelerate the reaction. It should be mentioned that in high temperatures, the decomposition of hydrogen peroxide is also accelerated, so the optimum temperature should be compromised.

The process was also studied at three pHs to investigate the effect of media acidity on the efficiency of decomposition. The pH of prepared copper sulfate was 3.5, which was one of the studied points. The other points were 2 and 3. It was observed that the optimum pH was 3 for both cationic and anionic resins, as can be seen in figure 4 (a-b).

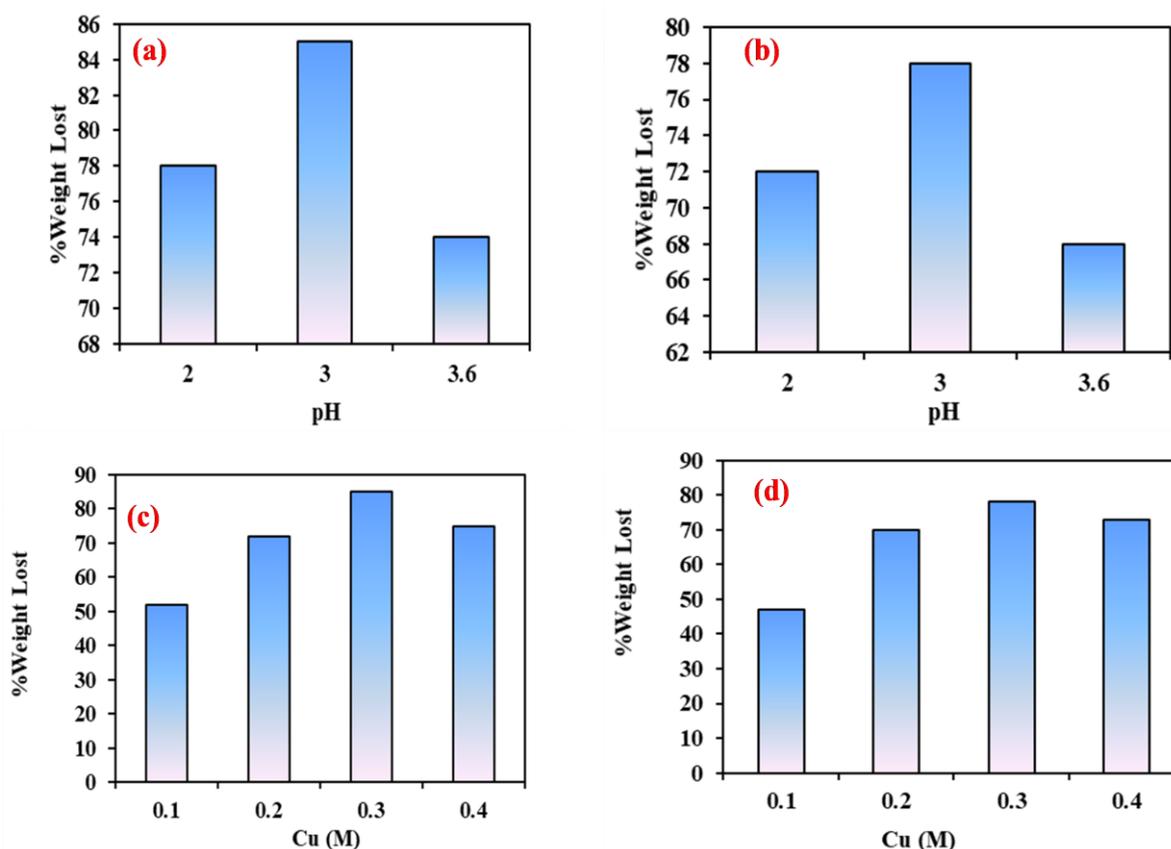
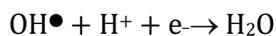


Fig. 4. The effect of media pH on decomposition percent in Fenton-like process for cationic, **a.** and anionic, **b.** resins, the effect of Cu^{2+} concentration on decomposition percent in Fenton-like process for cationic, **c.** and anionic, **d.** resins

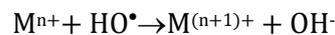
In many research studies [30], the pH range of 2.5-3 was obtained as the optimum range. According to the literature, by decreasing the pH (below the 2.5) the effect of radical elimination by H^+ as below occurs more severe:



In contrast, by increasing the pH more than 3, hydrolysis and precipitation of cations could decrease the catalyst capacity. In higher pHs oxyhydroxide or hydroxide precipitate formation leads to a decrease in the active metal species, which causes to decrease in radical production and decomposition rate. Considering these events, determining the pH effect could be somewhat complicated, so it should be identified experimentally for each process.

The other parameter affecting efficiency of resin decomposition is the Cu^{2+} concentration studied at four levels. The results are shown in figure 4(c-d). According to the figures, the concentration of 0.3 M of Cu^{2+} is the optimum value for both resins.

Increasing the catalyst increases the decomposition rate, but it could not be done unrestricted. Increasing the catalyst concentration can affect the radical concentration by eliminating it as below:



Moreover, increasing the catalyst will increase the cost of the process clearly, and the amount of produced sludge will also be increased, which needs subsequent treatment.

The flow rate of hydrogen peroxide addition is another parameter that could affect decomposition rate. Increasing the hydrogen peroxide drop by drop leads to moderate distribution in media. Moreover, delay in the decomposition of hydrogen peroxide is another reason for enhancing the efficiency. Gradual increase of hydrogen peroxide prevents intense increase of hydrogen peroxide concentration, so the side reactions are prohibited. As can be observed in figure 5(a-b), the optimum flow rate of hydrogen peroxide addition for the cationic resin is 0.5 ml/min and for the anionic resin is 0.25 ml/min.

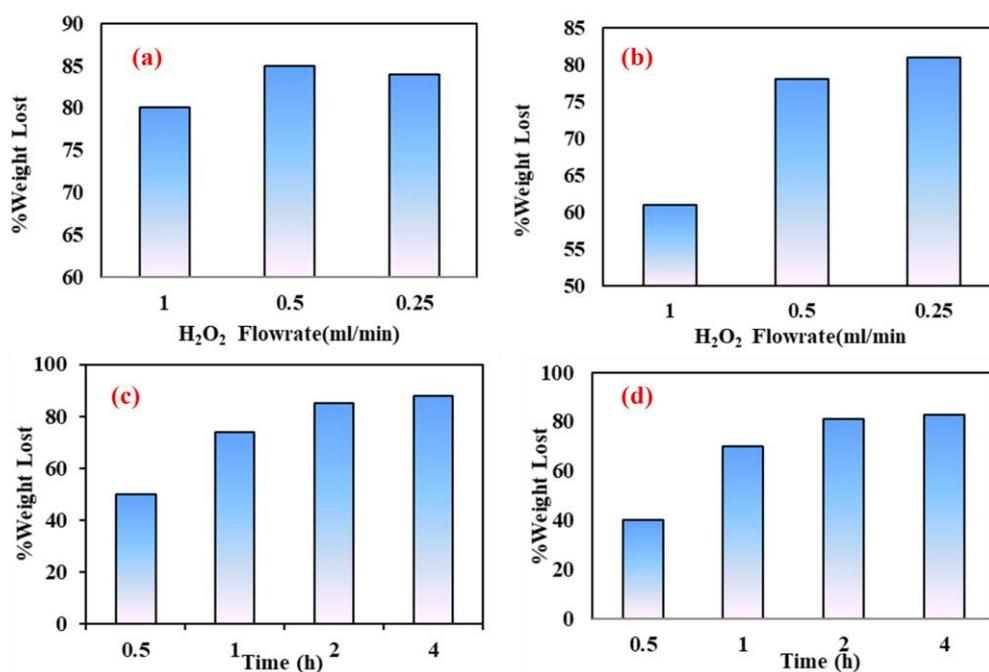


Fig. 5. The effect of hydrogen peroxide flow rate on decomposition percent in Fenton-like process for cationic, **a.** and anionic, **b.** resins, Decomposition percent of cationic, **c.** and anionic, **d.** resins by the Fenton-like process at various times.

At a higher flow rate, especially for anionic resin, foaming of reaction media occurs, which leads to exiting the resin from the top of the condenser and undesirable contact of reacting materials and consequently low efficiency of decomposition. The foam formation is shown in figure 6.

For each parameter, investigation the optimum conditions obtained in previous experiments was applied.

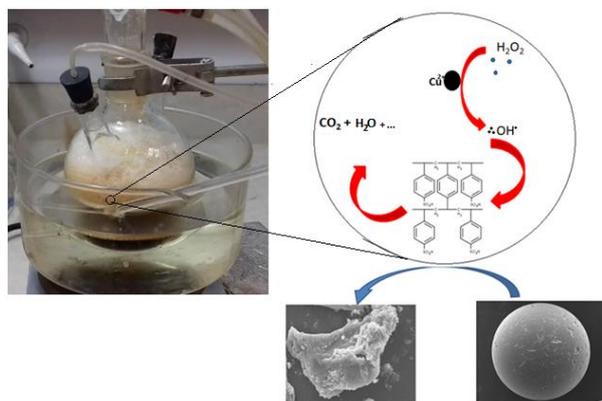


Fig. 6. Foam formation in anionic resin decomposition. Resin decomposition mechanism by Fenton process (inset).

At the end stage of optimization of effective parameters, the optimum time for the reaction was studied (Figure 5(c-d)).

It was found that by increasing the reaction time in a range of 0.5-4 hours, the decomposition percent increases, but the increase in the range of 2-4 hours was insignificant. So the optimum time can be 2 hours. No increase of efficiency by time can be because of generation of acidic products and decrease of pH. As mentioned before, decreasing the pH leads to the formation of stable oxonium species, which decreases decomposition efficiency.

By optimizing all parameters for fresh resins, the maximum decomposition percent of 88% was obtained for cationic resin in the optimized parameters and 83% for anionic resin.

The real sample, a mix of anionic and cationic spent resin from the Tehran research reactor, was decomposed at the optimized level of studied parameters. For this purpose, 2 g resin was placed in the flask, and pH of the catalyst solution was adjusted to 3, the temperature was adjusted to 75 °C, and the hydrogen peroxide (15 ml) was added at a flow rate of 0.25 ml/min. In this condition, the decomposition percent of 81% was obtained, which was desirable for our purpose.

Figure 7(A) shows the FTIR spectrum of fresh cationic resin and the FTIR spectrum of residual material after decomposition.

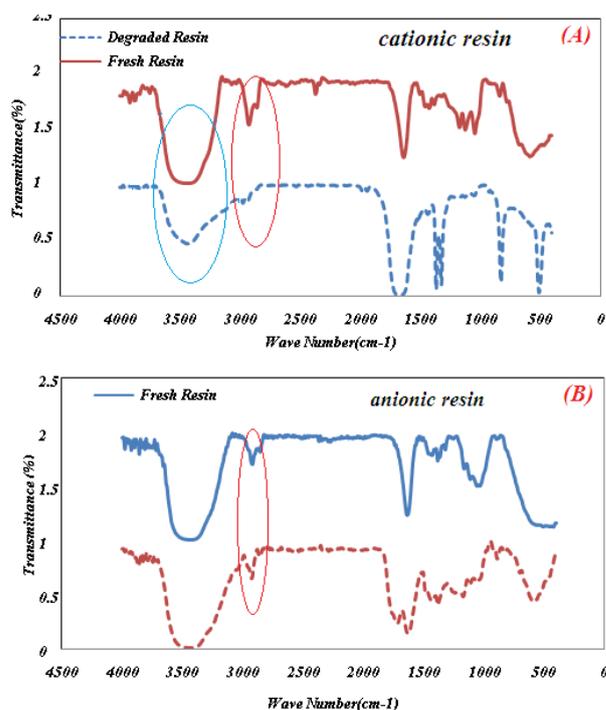


Fig. 7. A. FTIR spectrum of fresh cationic resin and the residual after decomposition, **B.** FTIR spectrum of fresh anionic resin and the residual after decomposition.

As can be seen in the spectrum, the intensity of the broad peak at 3500 cm^{-1} , which is for O-H and sulfonic bands decreased, because of the decomposition of resin. Also, the intensity of the peak observed at 2800-3000 cm^{-1} , which is for polystyrene structure, decreased because of

decomposition. Other peaks have also been changed because of the decomposition of resin.

Figure 7(B) shows the FTIR spectrum for anionic resin. As can be seen, the peak observed at 2900 cm^{-1} , which can be attributed to tertiary amine, decreased and shifted to lower wavenumbers because of decomposition. Other peaks have also been changed, proving the decomposition and structure changes [31].

Figure 8(a-c) shows the fresh and decomposed resin's SEM image. The SEM images indicated that the resin beads varied and deformed completely during the Fenton-like process. The sphere-like beads became small sheet-like particles which was a result of resin oxidation. The fresh resin has spherical particles sizing from 490 to 840 μm .

After decomposition, the particles were crashed with a disordered shape and the size of below 20 μm . The images (c, d) also show that the particle's surface became wrinkled due to the attack of hydroxyl radical and high pressure of CO_2 .

The residual solution was analyzed by ICP to determine the elements dissolved in the solution. Table 1 shows the result of this analysis.

To evaluate the decomposition efficiency, the COD of the solution was determined after separation of solid residual. According to the results of COD analysis and calculations based on the resin weight, it was concluded that about 2% of decomposed resins entered the solution as organic products, and 98% converted to CO_2 and H_2O .

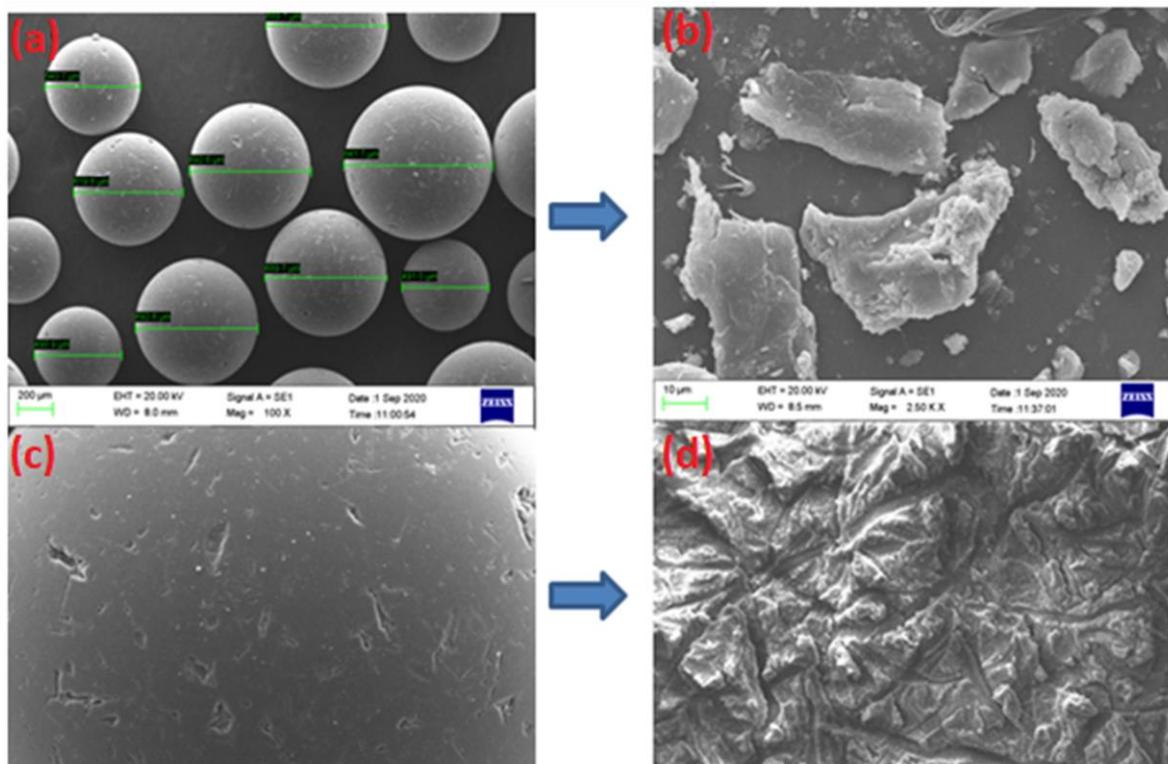


Fig. 8. SEM image of fresh resin (a and c) and decomposed resin (b and d).

Table 1. ICP analysis of residual solution.

Element	Concentration in	
	diluted to 100 ml solution (ppm)	mg/g in resin
Zn	23	1.15
Mg	1.2	0.06
Fe	0.4	0.02
Cd	0.34	0.017
Al	0.4	0.02
B	0.5	0.025
Cr	0.05	0.0025
Mn	0.05	0.0025
Na	220	11
K	1.2	0.06
Ca	7	0.35
Ba	0.03	0.0015
Si	1.1	0.05
Ti	0.5	0.025
Th	0.07	0.0035
U	<0.5	<0.025
Pb	20	1

4. Conclusion

In the current work, three methods were studied for the decomposition of spent resins produced in the Tehran Research Reactor. The methods include acid digestion, Fenton, and Fenton-like oxidation. The main goal of this work was volume reduction of spent resin by degradation. Then it will be suitable for long-term storage and/ or disposal. The effect of some factors, including pH, temperature, type of catalyst and its concentration and the flow rate of H₂O₂ were studied. Scanning electron microscopy and FTIR spectroscopy were used to show the morphological changes and the degradation of the resins. The SEM images indicated that the resin beads varied and deformed completely during the Fenton-like process, and the -sphere-like beads turned to sheet-like particles resulting from resin oxidation. The resin beads were disintegrated into small pieces resulting from the attack of hydroxyl radical and high pressure of

CO₂. The FTIR declared useful information about the decomposition of styrene, cross-linked agents, and functional groups of resins during the oxidation process. The results show Fenton-like process with Cu²⁺ as a catalyst is an effective method compared with acid digestion and the Fenton process for the disintegration and dissolution of cationic and anionic spent resins. It was found that the optimum values in this process were as follows: temperature: 75° C, Cu²⁺ concentration: 0.3 M, H₂O₂ addition flow rate: 0.5 ml/min for cationic and 0.25 ml/min for anionic resin, and time: 2 hours. At this condition, 15 ml H₂O₂ (30%) could degrade 2 g wet resin. Under optimum conditions the maximum degradation was 81 % for Tehran Reactor radioactive spent resin. The Fenton-like process proposed in the present work can be potentially used for the significant volume reduction of spent radioactive resins.

5. Conflict of interest

The authors declare that there is no conflict of interest.

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How to cite this article

H. Ghasemi Mobtaker, T. Yousefi, R. Yavari, *Decomposition of Spent Ion Exchange Resin Using Acid digestion, Fenton and Fenton-like Process*, Journal of Nuclear Science and Applications, Vol. 3, No. 1, P 47-58, Winter (2023), Url: https://jonra.nstri.ir/article_1482.html, DOI: 10.24200/jon.2023.1005.



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