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Degradation of Spent Radioactive Ion Exchange Resins and Its Mechanisms by Fenton Process

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Abstract: Spent IERs are released during the operation and decommissioning of nuclear facilities. The safe and efficient treatment of spent IERs is an emergent problem in nuclear industry. IRN77 is a typical ion exchange resin widely used in many nuclear power plants. Fenton process can degrade organic resins and reduce the radioactive residues volume and the disposal cost significantly. In this work, the IRN77 resin was selected as a model ion exchange resin and its treatment via Fenton process was investigated. The influencing factors for resin degradation, including catalyst dosage, reaction time, initial pH, temperature and oxidant dosage were investigated and optimized via the single-factor experimental method. Under the reaction temperature of 100°C and reaction time of 120 min at initial pH of 2, more than 97% COD was removed with 1.66 g H_2O_2 and 32 mg $FeSO_4 \cdot 7H_2O$ added to per gram of wet resin. The catalyst dosage and H_2O_2 dosage can decrease 78% and 50% respectively compared to previous results. SEM, FT-IR and ion chromatograph were employed to characterize the resin beads, soluble organics and intermediates during the degradation reaction. Based on the characterization results, the resin degradation pathway was discussed in detail and it is proposed to three stages including beads dissociation, styrene and divinylbenzene decomposition and carboxylic acids mineralization. During the IERs degradation, formic acid, acetic acid, propionic acid and oxalic acid were quantitatively monitored as main intermediates, and oxalic acid accounted for over 90% of COD in the final residue solution. Fenton process is suggested as a promising resin degradation method.

Keywords: Fenton; resin; degradation; mechanism

1 Introduction

In nuclear facilities water treatment systems, ion-exchange resins (IERs) have been usually used to minimize corrosion or the degradation of system components and to remove radioactive contaminants [1]. As usual, spent IERs are released during the operation of nuclear facilities, including the recycle of nuclear fuels, demineralization of the wastewater discharged, decontamination of nuclear installation, decommissioning of nuclear facility and so on. Spent IERs are one of the most important radioactive low-level wastes (LLW)

which are loaded by strontium-90, cobalt-60, cesium-137, uranium-235, uranium-238 and so on [2]. The spent resins must be treated as radioactive solid wastes due to the national regulations.

Traditional spent IERs treatment method is solidification but this method has the shortages of releasing explosive gases such as H_2, CH_4, corroding the equipment and container, and low resin mass containing ratio due to swelling property of resin [3]. Direct wet air oxidation, super- and sub-critical water oxidation needs high temperature and pressure and the efficiency is not sufficient [1,4]. Therefore, the treatment of spent IERs needs more efforts to improve its efficiency and safety. Advanced oxidation processes such as Fenton may degrade organic resins and reduce the radioactive residues volume and the disposal cost significantly.

Fenton process has been widely used to treat wastewater under laboratory conditions as well as real effluents from different resources, like olive-oil mill wastewater [5], textile wastewater [6], mining wastewater [7], pesticide wastewater [8], cosmetic wastewater [9], dye wastewater [10], landfill leachate [11], pharmaceutical wastewater [12], paper wastewater [13], pulp mill effluents [14,15] etc. The Fenton process is based on the formation of powerful oxidizing species (mainly ·HO), which are able to efficiently degrade the pollutants in wastewater [16].

IRN77 is a typical ion exchange resin widely used in many nuclear power plants. In recent years, several papers have been published regarding the degradation of spent radioactive resins by Fenton process [17–23]. However, the previous reports used a high catalysts/oxidants dosage and generated a large volume of final solution and sludge. Both the reaction mechanisms and optimized conditions are not clear and need further investigation. In addition, different types of resins may have different composition and characteristics, thus they may show different degradation performance. Though there are reports on other type radioactive resins degradation, there is few reports on IRN77 resins degradation till now. In this study, raw IRN77 resin is selected as model spent radioactive resin, the important parameters' effects (catalyst dosage, reaction time, initial pH, temperature and H_2O_2 dosage) are investigated via the single-factor experimental method. After that, the degradation mechanisms were proposed by characterizations of the intermediates during the reaction. It is believed that the results for raw IRN 77 resin degradation can be a reference for real spent radioactive IRN 77 treatment.

2 Materials and Methods

2.1 Resin and Chemicals

The IRN77 resin is a strong acidic styrene-divinylbenzene copolymer with sulfonic functional groups made by ROHM & HAAS (USA). Its chemical formula is $C_{12}H_{12}SO_3$ and it has moisture content of 53.13%. The other chemicals used in this study are of analytical grade and are all from Tianjin Kermel Chemical Reagent Co., China.

2.2 Experimental Set-up and Procedures

Experiments were conducted in a 500 mL, three-neck round-bottom flask, with a magnetic stirrer. The reactor was immersed in an oil bath with controllable temperature. Raw IRN77 resin 10 g, $FeSO_4 \cdot 7H_2O$ powder as catalyst and deionized water 10 mL were added into the reactor directly. The initial pH was adjusted by 3.5 mol/L H_2SO_4 and 10 mol/L NaOH. Then the reactor was sealed and heated. When the set temperature was reached, 30 wt% H_2O_2 was added with a constant flow rate using peristaltic pump. The flow rate is determined by the reaction time and H_2O_2 amount to ensure the H_2O_2 was added continuously during the whole reaction period. The reaction started when H_2O_2 was pumped in. At the end of the experiments, the pH and volume of the solution were measured.

The removal ratio of COD was measured according to Eq. (1):

$$\text{Removal ratio of COD} \; = \; \frac{c_0 - c_1}{c_0} \times 100 \tag{1}$$

where c_0 is the original COD of the resin, and c_1 is the COD of the residue solution and condense after a certain reaction period. Considering the volume change by H_2O_2 addition during the reaction, the COD concentration was normalized to a constant volume of 70 mL to avoid the dilution effect. Because the resin is a kind of solid particle, c_0 cannot be measured directly by standard method and was estimated theoretically to be 159172 mg/L based on the resin's chemical composition. In order to eliminate the effect of catalyst and H_2O_2 on COD measurement, the COD contribution from catalyst and H_2O_2 in the residue solution is also measured and deducted from c_1.

The effect of reaction conditions was investigated by the single-factor experimental method, which varied one parameter and kept other parameters constant. The detailed experimental parameters would be given in proper sections.

2.3 Analytical Methods

The infrared spectra were obtained with Nicolet IS50 Fourier transform infrared spectrometer (Thermo Fisher Scientific, USA). The samples were vacuum evaporated at 60°C for 72 h to get solid samples, and then the solid samples were pressed into KBr pellets at room temperature for FT-IR characterization.

The scanning electron microscopy (SEM) analysis was done by using JSM-IT100 SEM of JEOL Company, Japan. It was used to characterize the morphology changes of the resins during the reaction process. The resin beads were collected from the solution and washed immediately with deionized water and vacuum dried at 60°C for 72 h before SEM characterization.

The intermediate organic acids were monitored with 930 ion chromatograph (Metrohm, Switzerland). The column was metrosep organic acids-250/7.8. The eluent was 0.5 mmol/L H_2SO_4 and the regenerated liquid was 10 mmol/L LiCl.

The chemical oxygen demand (COD) of the dissolved resin solution was measured with 5B-3A (Lianhua Technology Co., China). The pH value was obtained by a pH meter (Leici PHS-3E, China). The concentration of H_2O_2 was determined using a $Ce(OH)CO_3/H_2O_2/TMB$ reaction system [24].

3 Results and Discussions

3.1 Effect of Catalyst Dosage

The effect of catalyst dosage on resin degradation is studied by the single-factor experimental method with $FeSO_4 \cdot 7H_2O$ as catalyst and its dosage varies from 1 to 200 mg/g wet resin. The reaction temperature of 95°C, reaction time of 150 min, initial pH of 2 and the amount of 30% H_2O_2 of 72 mL (equal to 2.4 g H_2O_2/g wet resin) are kept constant. Residue COD and COD removal ratio are selected to assess the degree of resin degradation.

Residue COD and COD removal ratio with different catalyst dosage are depicted in Fig. 1. It can be seen that with the catalyst dosage increase, the COD removal ratio increases quickly and reaches 95% when the dosage is 32 mg/L. When the dosage of catalyst is 128 mg/g wet resin, residue COD is about 520 mg/L and the COD removal ratio can reach the highest value of 99.7%. However, when the catalyst is more than 128 mg/g wet resin, COD removal ratio decreases. This phenomenon is consistent with previous results that H_2O_2 and Fe^{2+} usually have an optimized ratio during Fenton reactions. Comparing to the published paper [18], the present dosage of catalyst decreases 78.0% to reach the same COD removal ratio. The present study doses catalysts in powder while the previous study dosed catalysts in aqueous solution. In the present study, the final solution volume and sludge will also significantly decrease, so the further treatment cost decreases, too.
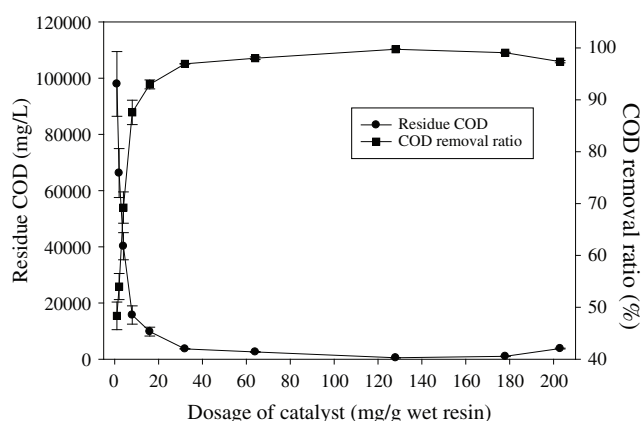
Figure 1: Effect of catalyst dosage on residue COD and COD removal ratio

Under the premise of ensuring high COD removal ratio, the dosage of the catalyst should be minimized in order to reduce the iron sludge amount in the residues. Thus in further experiments, the dosage of 32 mg/g wet resin is selected as the optimized catalyst dosage.

3.2 Effect of Reaction Time

Fig. 2 shows the effect of reaction time on resin degradation when the total reaction time is 30 min, 60 min, 90 min, 120 min, 150 min, 180 min and 210 min. The reaction temperature of 95°C, the initial pH of 2, the amount of 30% H_2O_2 of 72 mL and catalyst dosage of 32 mg/g wet resin are kept constant when changing the reaction time.
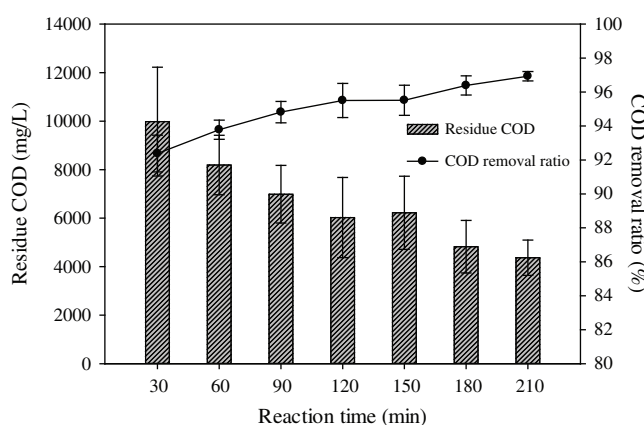

Figure 2: Effect of reaction time on residue COD and COD removal ratio

It can be seen from Fig. 2 that the residue COD decreases with the extension of the reaction time, and the COD removal ratio increases with the reaction time. When the reaction time extends from 30 min to 210 min, the residue COD decreases from 9982 mg/L to 4366 mg/L. COD removal ratio increases from 93.7% to 97.3%. Considering the treatment efficiency and performance, in further experiments the reaction time of 120 min is chosen.

3.3 Influence of Initial pH

The initial pH is adjusted to required values (pH = 1, 2, 3, 4, 5 and 6) with 10 mol/L NaOH and 3.5 mol/L H_2SO_4 solution. The reaction temperature of 95°C, the amount of 30% H_2O_2 of 72 mL, reaction time of
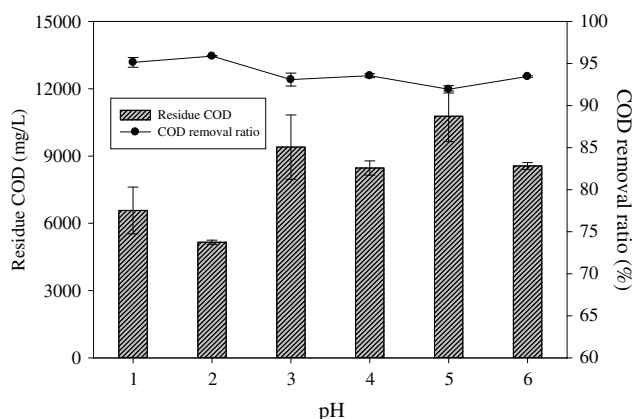
Figure 3: Effect of initial pH on residue COD and COD removal ratio

120 min and catalyst dosage of 32 mg/g wet resin are kept constant when changing the initial pH. Fig. 3 shows the effect of initial pH on resin degradation.

It can be seen from Fig. 3 that when the initial pH is 2, the residue COD of 5166 mg/L is the lowest, and the COD removal ratio of 96.8% is the highest. It shall be noted that the pH here is the initial pH and during the reaction due to the release of sulfonic acid and carboxylic acid, the pH will drop down. This indicates that optimized pH is different from the pH in wastewater treatment [5]. This is possibly because in the present reaction the H_2O_2 concentration and reaction temperature is much higher than that in wastewater treatment. Under the present conditions, when pH is lower than 2, Fe^{2+} and Fe^{3+} exists as $Fe(H_2O)_6^{2+/3+}$, and the conversion of Fe^{2+} to Fe^{3+} is retarded. With higher pH, H_2O is partially replaced by HO^-, Fe^{2+} and Fe^{3+} exists in the form of $Fe(OH)(H_2O)_5^{+/2+}$, which facilitates the conversion of Fe^{2+} to Fe^{3+} [18]. Though the initial pH is different, during the reaction sulfuric acid will be released and pH will decrease to ensure the reaction going on smoothly. Thus in further experiment, the initial pH of 2 is selected as the optimized initial pH.

3.4 Effect of Reaction Temperature

Fig. 4 shows the effect of reaction temperature on resin degradation with temperature range of 60 to 100°C. The temperature range is chosen according to preliminary experiments and reference [18]. When changing the reaction temperature, the amount of 30% H_2O_2 of 72 mL, reaction time of 120 min and catalyst dosage of 32 mg/g wet resin and initial pH of 2 are kept constant.
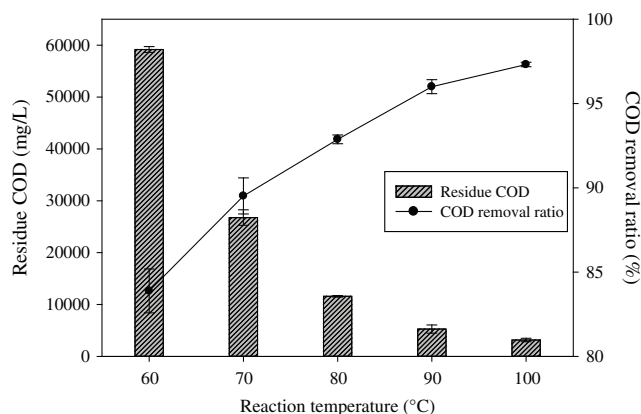

Figure 4: Effect of reaction temperature on residue COD and COD removal ratio

It can be seen from Fig. 4 that the residue COD decreases sharply with the increase of the reaction temperature. When the reaction temperature rises to 100°C, the residue COD of 3163 mg/L is the lowest with the highest COD removal ratio of 98.0%. According to the chemical reaction kinetics, the reaction ratio increases with increasing temperature, which is consistent with the results obtained by others [21,22]. It is noted that the H_2O_2 self-decomposition may happen under high temperature. However, the self-decomposition is believed to be lower than 6% under the present reaction conditions according to our preliminary results. This may affect the H_2O_2 utilization efficiency, but the optimized temperature will not be significantly affected considering the COD removal. Thus the reaction temperature is controlled at 100°C in further experiments.

3.5 Effect of H_2O_2 Dosage

The dosage of H_2O_2 affects the final resin degradation ratio, residue volume and also the processing cost. Too small dosage of H_2O_2 results in incomplete degradation and low degradation ratio. Excessive use of H_2O_2 not only causes higher cost, but also increases the final residue volume. Fig. 5 shows the effect of H_2O_2 dosage on resin degradation when the H_2O_2 dosage varies from 0.71 to 3.08 g/g wet resin. When changing the H_2O_2 dosage, the reaction temperature of 100°C, initial pH of 2, reaction time of 120 min and catalyst dosage of 32 mg/g wet resin are kept constant.
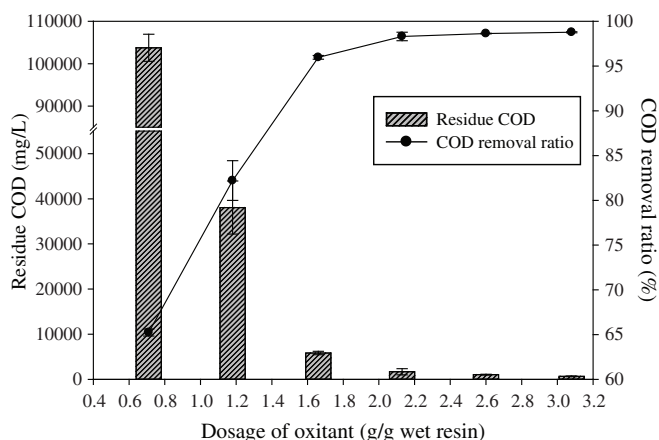

Figure 5: Effect of oxidant dosage on residue COD and COD removal ratio

It can be seen from Fig. 5 that the residue COD decreases with the increase of H_2O_2 amount. With H_2O_2 dosage increases, the residue COD decreases and COD removal ratio increases quickly. When the H_2O_2 dosage reaches 1.66 g/g wet resin, the residue COD value of 5483 mg/L and COD removal ratio of 97.1% can be reached. Though the degradation efficiency is not the highest under the present experimental conditions, the optimized oxidant dosage is assigned as 1.66 g/g wet resin by both considering the residue solution volume and COD removal. This optimized dosage is almost 50% lower than previous report [18], which indicates that the residue volume may also reduce ca. 40%. Thus the present technology is not only cost effective, it also produces much less residues and sludge for further disposal.

Stoichiometrically, it consumes 2.0 g H_2O_2 to completely mineralize 1 g wet resin. It is noted that the optimized H_2O_2 amount used here (1.66 g/g wet resin) is something lower than the stoichiometry. The higher COD removal ratio than stoichiometry may be mainly caused by volatile organic compounds (such as benzene, furan, ketone etc.) evolution from aqueous phase. These volatile organic compounds are already detected in the exhaust gas but their concentration and amount shall be further analyzed.

3.6 Degradation Mechanisms

In order to study the mechanisms of resins degradation, the residues during a typical degradation process are characterized.

During the resins degradation, the dissolution of the resin beads can be observed at the very beginning and after ca. 15 min, the beads can be dissolved completely and the reaction mixture changes to solution without apparent beads or solids. So the resin samples are collected and characterized with SEM at 5 min, 10 min and compared with the fresh resin beads (Fig. 6a). The cracks quickly appear on the beads surface in the first 5 min and the resin beads become smaller and starts to agglomerate (Fig. 6b). After 10 mins, the resin beads are further dissociated and collapse to smaller particles and agglomerate densely (Fig. 6c).
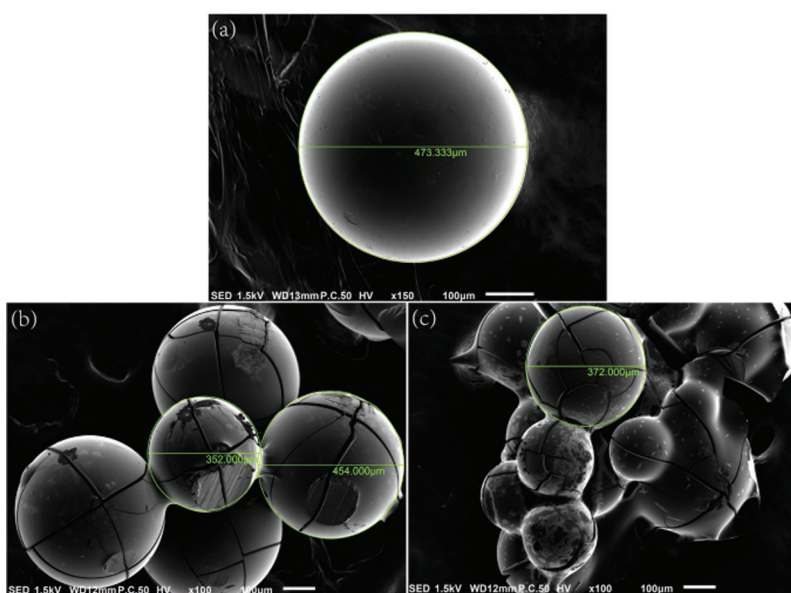

Figure 6: SEM images of resin beads after different reaction time (a) 0 min, (b) 5 min, (c) 10 min

In order to characterize the degradation process, the liquid samples during degradation are collected and evaporated to get solid samples. The solid samples are characterized with FT-IR as shown in Fig. 7 and the bands information is listed in Tab. 1. In Fig. 7 the methylene groups for crosslinking (675 cm^{-1}, 775 cm^{-1} and 2926 cm^{-1}) disappear in ca. 15 min indicates the quickly dissociation of resin beads to styrene and divinylbenzene monomers. The absorption bands at 833 cm^{-1} and 1412 cm^{-1} are the signals for benzene rings and they diminish with reaction going on. While the band at 1219 cm^{-1} is for carbonyl groups in carboxylic acids and it is strongest at reaction time of ca. 60 min. It can be proposed that styrene and divinylbenzene are oxidized to carboxylic acids with benzene rings opening during reaction time of ca. 15–60 min. In the later stage after 60 min, the carboxylic acids are further mineralized together with residue benzene rings opening, which is consistent with the results obtained by others [22].

Except FT-IR, the quantitative analysis of intermediates such as oxalic acid, formic acid, acetic acid and propionic acid is also performed, because these carboxylic acids are usually intermediate products of aromatic compounds degradation [20,22]. As shown in Fig. 8, oxalic acid shows the highest concentration level of 73400 mg/L at the reaction time of 30 min, while the others show concentration level lower than 11000 mg/L. The propionic acid concentration is almost zero. It can also be seen that the carboxylic acid concentration increases quickly at the first 30 min, which indicates quickly degradation of
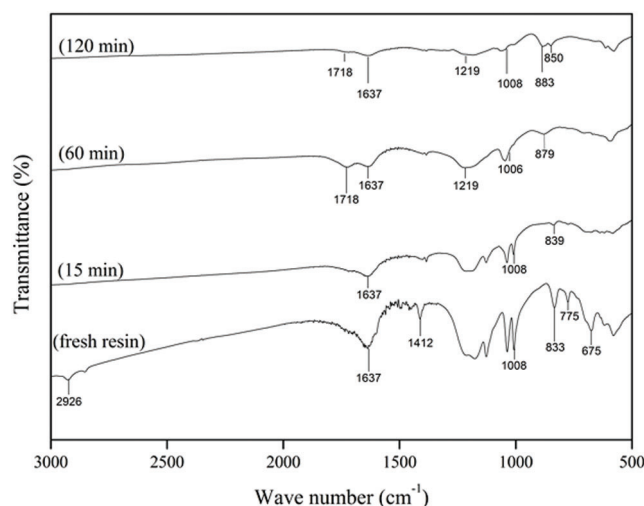
Figure 7: The changes of the FT-IR spectrum during resins degradation

Table 1: Summary of FT-IR bands and tentative assignments during resins degradation

Assignment	Bands position at different reaction time (cm^{-1})			
	0 min	15 min	60 min	120 min
C-H bending (methylene)	675			
	775			
C-H deformation (benzene ring)	833	839		850
			879	883
SO$_4^{2-}$ symmetry stretching	1008	1008	1006	1008
−COOH			1219	1219
C-H vibration (methylene)	1412			
C=C stretching	1637	1637	1637	1637
−C=O stretching			1718	1718
−CH$_2$-stretching (methylene)	2926			

resins. As the reaction proceeds, the contents of the four carboxylic acids reach the maximum during 30–60 min and then decrease. This indicates resins can be degraded into carboxylic acids in 30–60 min and then these carboxylic acids are further oxidized in later stage. The concentration changing trend of formic acid and oxalic acid in our results is consistent with other study [19]. It is still difficult to quantitatively determine all intermediates during IRN77 degradation because of their complexity. According to the quantity of oxalic acid and COD, it is estimated that oxalic acid contributed over 90% of COD in the final residue solution.

It is also believed that OH· radicals play a very important role for resin degradation. However, it is still difficult to monitor the OH· continuously in the aqueous phase due to the high concentration of organic matters and the high temperature of the solution via traditional methods. It is worthy further study on free radicals monitoring method later.
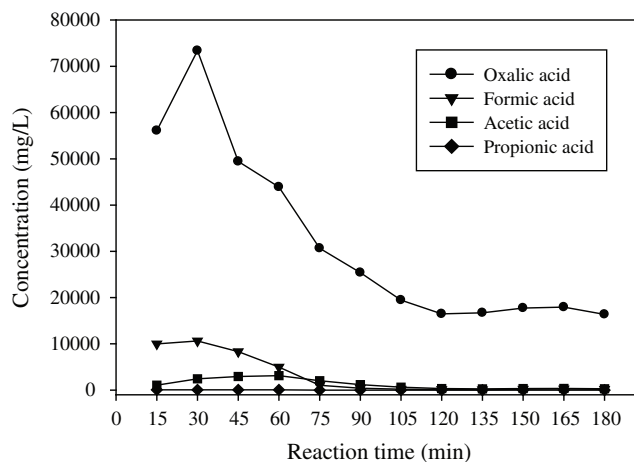
Figure 8: Carboxylic acids concentration change during resins degradation

From the above experimental results, it can be proposed that in the early stage of the degradation (before ca. 15 min), the main reaction is the dissociation of resin beads by breaking down the crosslinking bonds to form soluble styrene and divinylbenzene (Step ① in Fig. 9). In addition, some of the benzene rings are also broken down during this stage (Step ② in Fig. 9). During the middle stage (ca. 15–60 min), the main reactions are mainly the breaking down of the sulfonic functional groups, benzene rings and the long carbon chains (Steps ③ and ④ in Fig. 9), which respectively form sulfuric acid and carboxylic acids. The final stage of the reaction (after ca. 60 min) is mainly the mineralization of carboxylic acids, which are completely degraded into CO_2 and H_2O.
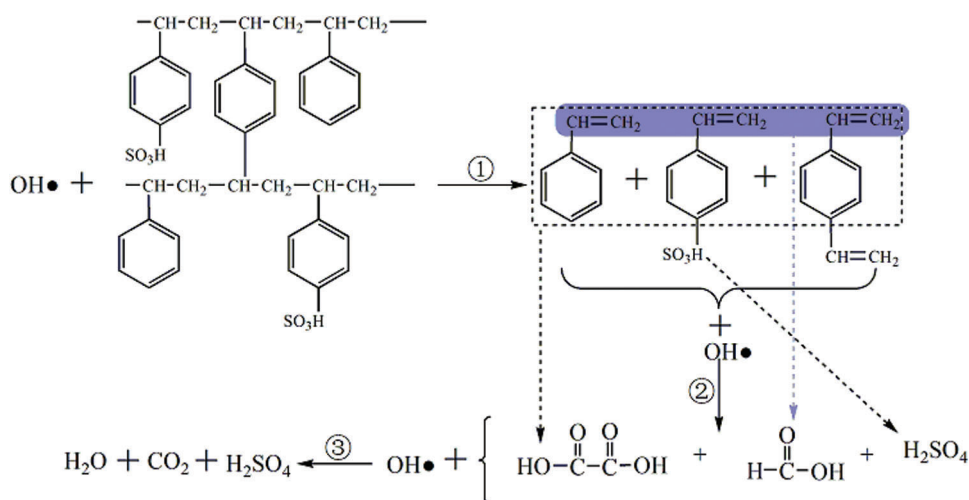

Figure 9: Illustration of the resins degradation pathway

4 Conclusions

By using IRN77 resin as a model, the degradation of spent radioactive ion exchange resin is studied by Fenton process. The influencing factors for resin degradation, including catalyst dosage, reaction time, initial pH, temperature and oxidant dosage are investigated and optimized via the single-factor experimental

method. Under the optimized condition, more than 97% COD is removed, while the catalyst dosage and H_2O_2 dosage can decrease 78% and 50% respectively compared to previous results.

The degradation pathway of resins is proposed to three stages. Firstly, the resin beads are dissociated into soluble styrene and then the styrene is decomposed to carboxylic acids. The final stage is mainly the mineralization of carboxylic acids. The oxalic acid accounts over 90% of COD in the final residue solution. This research suggests Fenton process to be a promising method for resins degradation.

Conflicts of Interest: The authors declare that they have no conflicts of interest to report regarding the present study.

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