



# On-line mini-column flow injection electrochemical method for researching on resuscitation and dehydration performance of deeply-fouled cation-exchange resins



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## HIGHLIGHTS

- A mini-column FIA system was developed for studying cation-exchange resin (SCR).
- Examined the ion-exchange capacity of resuscitating and dehydrating the waste SCR.
- The system can become a special device for determining cation resin performance.
- Found ion-exchange capacity for dried SCR is higher than undried one.
- Proved fresh cation-exchange resin can be saved in drying state.

## ARTICLE INFO

### Keywords:

Deeply fouled resin  
Flow injection  
Dehydration  
Resuscitation  
Electrochemistry

## ABSTRACT

For rapidly and accurately obtaining the performance parameters of ion-exchange resins, an especial online column flow-injection electrochemical method and system (FIA-ECM) was designed. In the research, the waste strong acid type cation-exchange resin (WSCR) was used as test sample, which was again immersed in the saturated ferric trichloride solution and lubricant oil for long time and heated at 110 °C to further deteriorate its ion-exchange performance. And then, the orthogonal and the single factor experiments on WSCR were conducted by using the FIA-ECM system. Under optimized conditions, the resuscitation rate of WSCR reached to 90% which can reuse in industry for various water treatments. Besides, a new conclusion was also obtained by using the FIA-ECM system, namely, exchange capacity of SCR being in drying state is higher than that in wetting state, and new SCR can be transported and saved in drying state, thus can notably reduce transport costs. The conclusion has very important significance in water treatment industry using resins. Merits of FIA-ECM were higher in automation extent, lower in reagent consumption in the test, inexistent in personal errors, and conducive to environmental protection. It can be extended to automatically and rapidly determine performances of various resins or new developed materials with cation-ion exchange capacity and screening of various revitalizers for resins.

## 1. Introduction

Ion-exchange resin has been widely used in the purified-water preparation [1–4], biological product refining [5,6], impurity removal [7–9], industrial wastewater treatment [10], product purification [11,12], synthesis of organic compounds [13,14], desalination in nuclear power stations [15,16] and fossil power plants [17,18], removal of boron from geothermal water [19], and many fields [20,21]. It possesses many advantages that are convenient in manipulation, low in energy consumption, easy in separation after use, and low in itself cost.

The process of the resin's ion-exchange is reversible, but for various ions, its exchange capacity is different. Generally speaking, the ion-exchange resin easily exchanges with high-valence state ion. Using strong-acid type cation-exchange resin (SCR) as an example, it is very easy to exchange with such cation that the hydrated ionic radius is small. So, usual sequence for SCR exchanging cation is as follows [22,23]: Fe(III) > Fe(II) > Al(III) > Ca(II) > Mg(II) > K(I) > Na(I) > H(I).

However, the easier the cation is exchanged onto the SCR surface, the more difficult the cation is eluted off, especially is Fe(III) ion. Thus

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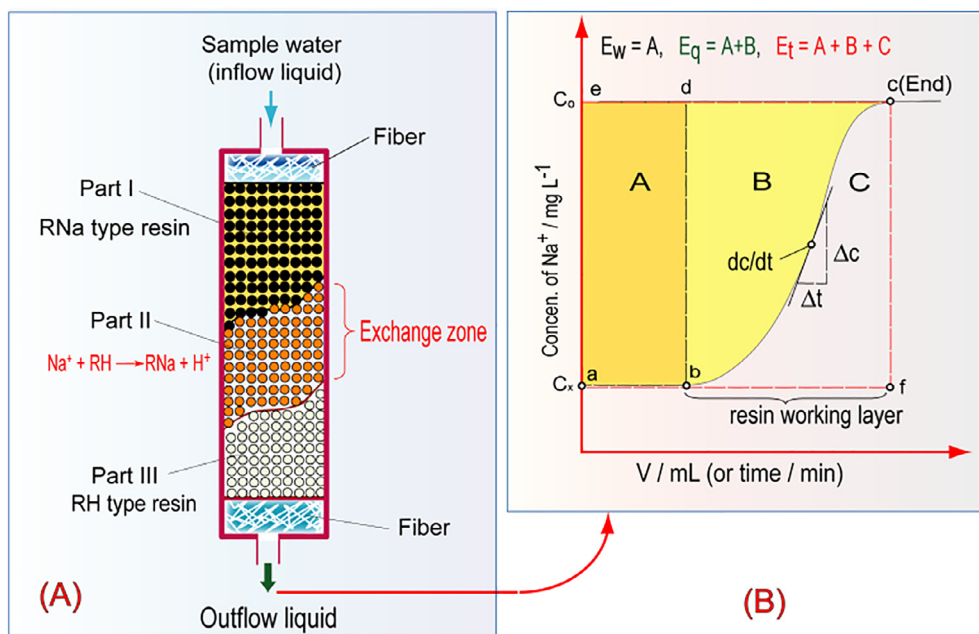


Fig. 1. Cation-exchange reaction and a flow-out liquid curve in mini-exchange SCR column. a, Schematic diagram for cation-exchange reaction; b, Variation of sodium ion concentration in the outflow liquid.

if SCR was used to ion-exchange for long time, Fe(III) would be gradually kept onto its surface, where Fe(III) was changed into Fe(OH)<sub>3</sub> colloid which will block up interface of SCR ion-exchanging, and when Fe(III) ion on the SCR surface or inside exceeds a certain limitation, the ion-exchange capacity of SCR will sharply decrease to result in failure. Therefore, in order to prolong the service life and repeated utilization rate of SCR [24,25], some studies have been conducted in the deeply fouled WSCR (DF-WSCR) recovering [26–32], in which the research purpose is all to try to restore the ion-exchange capacity and reuse in industrial production.

Presently, studies on the resuscitation of DF-WSCR were mainly conducted by using the manual operation, in which its disadvantages are that the volume of resin sample used for testing is very large, the treating process is extremely tedious and very time-consuming, and the human error is easily brought, etc. Besides, because the manual operation is difficult to obtain the continuously dynamic data on ion-exchanging [33,34], so it is also impossible to obtain the dynamic information in the whole resuscitation process of DF-WSCR.

The flow-injection analysis (FIA) [35–38] is a very characteristic analytical means that it is easy in operation and has very high accuracy and low reagent consumption, so far, it had been used for various fields [39–51]. Consequently, taking advantage of FIA, an online ion-exchange mini-column flow-injection electrochemical method and system (FIA-ECM) were developed, which can be used for studying performances of SCR ion-exchange and DF-WSCR resuscitation, and effects of various constituents in revitalizer on the DF-SCR resuscitation were also examined.

## 2. Experiment and method

### 2.1. Apparatus

A FIA-3100 model apparatus (Jitian instrument limited company, Beijing, China) was used as a flow-injection processor which was connected with a DWG-9323A model meter and a 102 model sodium ion selective electrode as well as a 001S model calomel electrode. Besides, a TDS-110 model electrode was used as a temperature sensor (Tianshi Instrument Co., Shanghai, China). A computer was used for treating the recorded signal in the research. Ultra-purified water was prepared by a

KL-UP-IV-10 model device (Kangling factory, Chengdu, China).

### 2.2. Preparation of reagent and standard solutions

All chemicals in the research were analytical-reagent grade. Ultra-purified water (0.065 μS cm<sup>-1</sup>) was used to prepare various solutions which were stored in polyethylene vials. The waste 732 (001 × 7) styrene type SCR were used as testing sample, which is the same in performance with Amberlite IR-120 and Dowex-50 (U.S.), Lewatit-100 (Germany), Diaion SK-1 (Japan) and Allassion CS (France).

#### 2.2.1. Basification liquid

60 mL of di-isopropylamine was transferred in 100-mL measuring cylinder and diluted to the mark with purified water.

#### 2.2.2. 2.0 g L<sup>-1</sup> of sodium-ion stock solution

The saturated NaOH solution was taken 2.50 mL to transfer into 1000-mL volumetric flask and diluted to the mark with water. And then the Na<sup>+</sup> content in the stock solution was demarcated by the potassium hydrogen phthalate, where 10 g L<sup>-1</sup> phenolphthalein solution was used as indicator, and obtained demarcation result was 2.0013 g L<sup>-1</sup> for Na<sup>+</sup>. A certain volume of sodium stock solution was taken to dilute and prepare the standard working solution of sodium ion.

### 2.3. Making of online mini-column

Distribution of DF-WSCR in particle size was not uniform. It was proved that when the particle size of ion-exchange resin was approximate same, the resin exchange capacity was the highest [52]. So, the resin sample was sieved by using Taylor standard screens before the experiment to reduce test error coming from the non-uniformity and the resin between 20 and 40 mesh was used for next test, which was about 0.68 mm in grain diameter.

The DF-WSCR sample sieved was placed in a desiccator for 30 min to constant weight. 0.200 g DF-WSCR was taken to fill into an organic glass column which was 10 cm in the length and 3.0 mm in the inner diameter, two ends of the column was sealed with 100 mesh nylon net (pore size: 150 μm), which was to prevent escaping of the resin within the column and to give an elastic buffer space where permitting the

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