

Desalination of graphite oxide by electro dialysis



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HIGHLIGHTS

- Electro dialysis is introduced to the desalination of GO dispersions.
- A new ED system is developed to solve the membrane fouling.
- This new ED system can ensure the continuous desalination of GO dispersions.

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ABSTRACT

Graphite oxide (GO) has got more and more attention for its excellent performance. However, the desalination of GO dispersions is a challenge because of its easy swelling and electronegativity. A new electro dialysis (ED) system was developed specially by adding an interception medium (IM), to solve the membrane fouling of conventional ED system and achieve efficient desalination. The salt removal rate and efficiency were achieved about 85% and 88%, respectively, for GO by this designed ED system under the optimum conditions. The results showed that the designed ED system was more suitable for the desalination of GO dispersions, which intercepted charged particles and ensured the continuous work of anion exchange membrane (AEM). These results provide important technical basis for a large-scale procedure of GO and the desalination of charged colloid dispersions.

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1. Introduction

Graphite oxide (GO) has attracted a wide interest of the chemical community since synthesis technology becomes more mature [1–5]. GO still keeps a layered structure after oxidation, and has the characteristics of strong polarity, large specific surface area, and high ability to exchange particles [6–11]. Recently, it has been widely applied in the electronic devices [12], sensors [13], lithium ion batteries [14–16] and super capacitors [17]. However, the desalination of GO dispersions after preparation is a challenge because it is easy to swell to charged colloidal dispersions during the process, which limits its purity and some applications such as catalysts [18].

Nowadays, electro dialysis (ED) is used as an alternative to conventional industrial separation methods even for colloids as the result of the dramatic improvements of membrane properties and the advantages of highly selective separation, separation without any auxiliary materials, continuous and automatic operation, and economical operation also in small units [19,20]. While the only problem to solve is the membrane fouling [21], which causes a decrease of membrane permeability as a result of the accumulation of colloidal and particulate matter [22–24]. In this paper, a new ED system is designed by adding an

interception medium (IM) based on the conventional electro dialyzer, to control membrane fouling. GO dispersions are used to investigate the performance of the designed ED system, the results will demonstrate proof that this ED system is much more suitable for the desalination of GO dispersions.

2. Material and method

2.1. Experimental installation

The designed ED system setup mainly consisted of an electro dialyzer. This electro dialyzer (Fig. 1b) was divided into four cells by anion exchange membrane (AEM), interception medium (IM), and cation exchange membrane (CEM). AEM and CEM were commonly used nowadays and made of polyethylene. IM is a kind of interception medium that is made of polypropylene. These were all acid-proof and corrosion-resistant. Compared with the conventional electro dialyzer (Fig. 1a), IM was added into the electro dialyzer, to separate intermediate chamber and dilution chamber. Dispersions to be purified flowed into the dilution chamber, and deionized water or a certain concentration of electrolyte solution flowed into intermediate chamber. IM was used to intercept charged particles that easily caused membrane fouling, and meanwhile the intermediate chamber provided a channel for ions to pass, which was a guarantee of the ongoing electro dialysis process.

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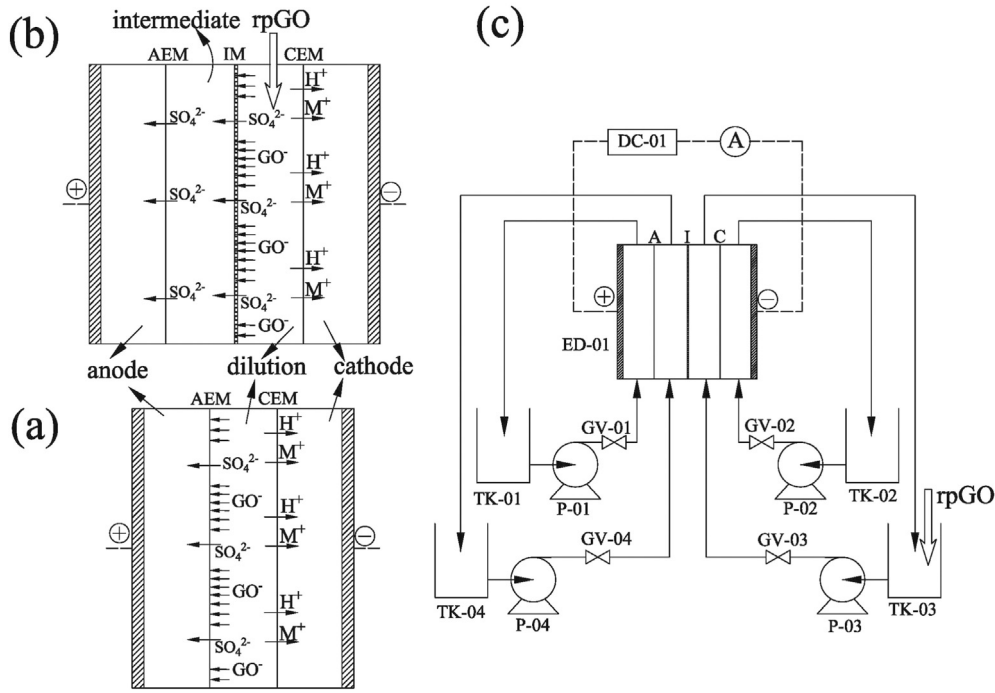


Fig. 1. Structure of conventional ED (a), new ED (b) and the whole new ED system setup(c).

For this designed electrodesion cell, the thickness and effective area of each cell were 3 mm and $100 \times 100 \text{ mm}^2$, respectively. If the structure was top opening, the desalination was intermittent and dispersions in dilution chamber should be stirred well at regular intervals. Otherwise the electrodesion cell was airtight, it needed a set of accessories to make the liquid in each cells recirculating flow such as feed tanks, pumps and globe valves, as shown in Fig. 1(c). In order to prevent the permeation of solution in the intermediate chamber by concentration difference, the inlet pressure of dilution chamber was higher than that of the intermediate chamber.

2.2. Material

The GO dispersions prepared by a modified Hummers method were used in this experiment. Now, it is well known that GO was prepared by oxidation of graphite with $\text{KMnO}_4/\text{H}_2\text{SO}_4$ and subsequent hydrolysis according to the method of Hummers–Offeman [18,25]. However, during the preparation strong oxidizing agents were excessive, a large number of ions such as K^+ , Na^+ , Mn^{2+} , H^+ , SO_4^{2-} remained in GO samples. GO dispersions were washed with deionized water and separated by centrifugation (1500 rcf, 20 min) twice. The total ion content in GO dispersions after washing that swelled to stable colloidal dispersions was about 2.75%. Subsequently, this colloidal dispersion was flowed into the electrodesion cell designed for further desalination.

2.3. Analytical method

A conductometer (DDS-11A) was used to measure the conductivity in the intermediate and dilution chamber. According to the research by Kohlrausch, the relation of molar conductivity and concentration for strong single electrolyte solution can be expressed as

$$A_m = A_m^\infty - A\sqrt{c} \quad (1-1)$$

where, A_m is molar conductivity, $\text{S} \cdot \text{m}^2/\text{mol}$, A_m^∞ is limiting molar conductivity, $\text{S} \cdot \text{m}^2/\text{mol}$, c is concentration, mol/L , A is a constant. The electric conductivity of Na_2SO_4 solution with different concentrations was measured and fitted. Then the relation of conductivity and

concentration was obtained, as

$$\frac{\kappa}{c} = 260 - 430\sqrt{c} \quad (1-2)$$

where, κ is conductivity, mS/cm , and c is concentration, mol/L . Desalination rate and current efficiency were measured. Considering that ions in GO dispersions were various, salinity was calculated by milliequivalent (mEq). The conversion relation of mg and mEq for this dispersion was expressed as follows.

$$\gamma = \frac{m(\text{K})}{39} + \frac{m(\text{Na})}{23} + \frac{m(\text{K})}{27.5} \quad (1-3)$$

where, γ is salinity, mEq, m is the mass of various ions in GO dispersions, so desalination rate, and current efficiency can be expressed as

$$\varphi = \frac{\gamma_0}{\gamma_d} \times 100\% \quad (1-4)$$

where, φ is desalination rate, γ_0 and γ_d are feed and dilute salinity, mEq, respectively.

$$\eta = \frac{F \cdot (\gamma_0 - \gamma_d) \cdot 10^{-3}}{It} \quad (1-5)$$

where, η is current efficiency, F is Faraday constant (96,500), and It is the total actual consumption electricity in the electrodesion process [21]. The content of ions in the GO dispersions was obtained by Inductively Coupled Plasma (ICP) after the sample drying, calcining in muffle furnace and acid dissolution.

3. Results and discussion

3.1. Ion transportation

GO dispersions contained more Na^+ than other positive ions, so we chose Na_2SO_4 solution to study the ion transportation law between each chamber in the designed and conventional ED system. In Fig. 2, the

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