

## Desalting of papermaking tobacco sheet extract using selective electro dialysis

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**Abstract.** The inorganic components in tobacco sheet extract have significant influence on the sensory taste of the cigars and the harmful component delivery in cigarette smoke. To identify the contributions of the divalent inorganic components on harmful components delivery in cigarette smoke, a self-made selective electro dialysis was assembled with monovalent ion-selective ion exchange membranes. The influences of current density and extract content on the desalination performance were investigated. Result indicates that the majorities chloride, nitrate, and sulfate ions were removed, comparing with 50-60% of potassium and only less than 10% of magnesium and calcium ions removed in the investigated current density. The permselectivity of the tested cations across the Selemion CSO cation exchange membranes follows the order:  $K^+ > Ca^{2+} > Mg^{2+}$ . A current density of 15 mA/cm<sup>2</sup> is an optional choice by considering both the energy consumption and separation efficiency. When the extract contents are in the range of 7%-20%, the removal ratios the potassium ions are kept around 60%, while the removal ratios of the calcium and magnesium ions fluctuate in the range of 16-27% and 8-14%, respectively. The tobacco smoke experiments indicated that the divalent metal ions have dual roles for the harmful component delivery in cigarette smoke. The divalent potassium and calcium ions were unfavorable for the total particulate matter emission but beneficial to decrease the HCN delivery in the mainstream cigarette smoke. The selective electro dialysis is a robust technology to decrease the harmful component delivery in cigarette smoke.

**Keywords:** electro dialysis; monovalent ion-selective ion exchange membranes; reconstituted tobacco; papermaking tobacco sheet

### 1. Introduction

Papermaking reconstituted tobacco (PRT) technology is a mean for uniform control and modification of the chemical and physical properties of tobacco substantially superior to natural

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tobacco. In addition to chemical and structural modifications, the utility of PRT technology is an important tool for the cigarette smoke modification. Previous studies have demonstrated that PRT differs from natural tobacco leaf in the yield of particulate matter, nicotine and carbonyl compounds in the smoke (Wang *et al.* 2005). During the past 20 years, the use of PRT in cigarette has increased rapidly for the unique superiority in structural strength, combustion performance, and tar delivery (Wang *et al.* 2005). There are fourteen reconstituted tobacco sheet factories under the jurisdiction of China Tobacco Industry Co, Ltd and numerous private reconstituted tobacco sheet factories are under construction.

In comparison with natural tobacco leaf, PRT is rich in inorganic components due to the slather of tobacco stem. Numerous studies have indicated that the inorganic ions have significant influence on the sensory taste of the cigarette and the harmful component delivery in cigarette smoke (Seyler *et al.* 2013, Narkowicz *et al.* 2013). Generally, chloride and nitrate/nitrite ions are considered as undesirable components which affect the moisture absorptivity and combustibility of cigarette as well as harmful carbonyl compounds delivery in the mainstream smoke (Nunes-Alves *et al.* 2013, Johnson *et al.* 2009, Flischer *et al.* 1989). Nitrate and nitrite ions will lead to the generation of the carcinogen tobacco-specific nitrosamines (TNSAs). Potassium and ammonium ions are beneficial to the combustion behavior of cigarette (Chen *et al.* 2014). The monovalent or divalent alkali and alkaline earth metals are usually considered to be desirable components which have a catalytic role in the thermal degradation and char formation of biomass (Nowakowski and Jones 2008). Therefore, it is of significance to selectively manipulate the inorganic components of tobacco extract in PRT industry.

Electrodialysis (ED) is a mature but robustness technology for the selective ions removal by considering the permselectivity characteristic in the ion exchange membranes. As opposed to other separation techniques, ED does not suffer from major drawbacks, such as generation of large amounts of waste, use of hazardous solvents, short lifetimes of adsorbents, etc (Xu 2015, Huang *et al.* 2008, Strathmann 2011, Tanaka *et al.* 2012, Aghajanyan *et al.* 2013, Ali and Hamrouni 2016, Majewska-Nowak 2013). ED is thus considered an environmentally friendly and sustainable technology and may be a very competitive one when it uses in the tobacco industry. In our previous studies, the conventional ED has been used for the removal of the chloride and nitrate ions of tobacco sheet juice (Zhang *et al.* 2014). An improved sensory is obtained for cigarette made from the completely desalted tobacco sheet extract. The influences of current density and contents of tobacco extract were also investigated in our recent publication (Ge *et al.* 2016). The relative delivery of CO, NH<sub>3</sub>, HCN and phenol delivery in cigarette smoke are decreased compared with the control cigarette without the ED treatment (Ge *et al.* 2016). The previous studies were conducted with the conventional ED and all the dissociated ions are removed. As a consequence, it is still unknown whether the decrements in the harmful carbonyl compounds delivery in the mainstream smoke are caused by the monovalent inorganic ions or the divalent and multivalent ions. Therefore, the main objective of this study is to desalt the tobacco sheet extract using selective electrodialysis with monovalent ion-selective ion exchange membranes. The selective electrodialysis is an electro-membrane process, which has a special function to separate the monovalent ions from multivalent ones with the same sign of charge on the base of its original permselectivity (Lambert *et al.* 2006, Zhang *et al.* 2014). This new technology may help clarifying the contribution of monovalent and divalent inorganic ions on the harmful component delivery in the mainstream smoke.

Table 1 The main characteristics of the membranes used in the experiments

Properties	Selemion CSO	Selemion ASV
Thickness/ $\mu\text{m}$	100	120
Resistance/ $\Omega \cdot \text{cm}^2$	2.3	3.7
Transfer number/%	>97	>97
Break stress/MPa	1.5	2.0

## 2. Experimental

### 2.1 Materials

Berley tobacco sheet extract (solid content ~40wt%) used in this experiment was supplied by China Tobacco Anhui Industrial CO., Ltd, Hefei, Anhui Province, China. This tobacco extract was pretreated by a centrifugation at 3000 rpm for 5 min to remove the suspended solids before the ED process. The membranes used for the electro dialysis (ED) experiments were monovalent ion-selective anion exchange membrane (Selemion ASV, Asahi Glass Co., Ltd., Japan) and monovalent ion-selective cation exchange membrane (Selemion CSO, Asahi Glass Co., Ltd., Japan), and their properties are listed in Table 1.

### 2.2 ED set-up

A schematic diagram of ED set-up is illustrated in Fig. 1. The ED set-up was composed of (1) a cathode and an anode were respective fixed on two organic glasses plate each with a rectangular notch; the electrodes were made of Iridium-Tantalum with a thickness of 1.5 mm. The electrodes were fixed into the notch with epoxy glue to make an even surface on the organic glasses. A direct current power supply (WYL1703, Hangzhou Siling Electrical Instrument Ltd.) was connected on the electrodes. The voltage drop across the stack was directly read from indicators on the power supply. (2) Eleven pieces of cation exchange membranes and ten pieces anion exchange membranes, which are alternatively arranged. The effective area of each membrane was  $189 \text{ cm}^2$ . (3) Sealing spacers made from PE with a thickness of 0.75 mm was used to separate the membranes; (4) Beakers to store the feed. Each beaker was connected with a submersible pump (AP1000, Zhongshan Zhenghua Electronics Co. Ltd., China, flow rate of 22 L/h) to form a circulating loop. In our experiments, three circulating loops, namely, electrode chamber, concentrate chamber, and diluted chamber were established in this ED stack. A 400 mL  $\text{Na}_2\text{SO}_4$  solution (0.3 mol/L) was fed into the electrode chambers as the rinse electrolyte. A 400 mL tobacco sheet extract was fed into the dilute chambers. A 400 mL tap water was fed in the concentrate chamber. Before the experiment, each chamber was circulated for 30 min to eliminate the visible bubbles. ED experiment was initially operated at a constant current mode but later conducted at a constant voltage mode. Because the resistance in the feed chamber was continuously increased during the experiments, ED would automatically be shifted to voltage constant mode when the stack voltage reaches the maximum value of the current supplier. All the experiment runs were kept at room temperature. It should be noted that the solution temperature increase by Joule effects in the experiments is not pronounced due to a short experiment time and a small stack.

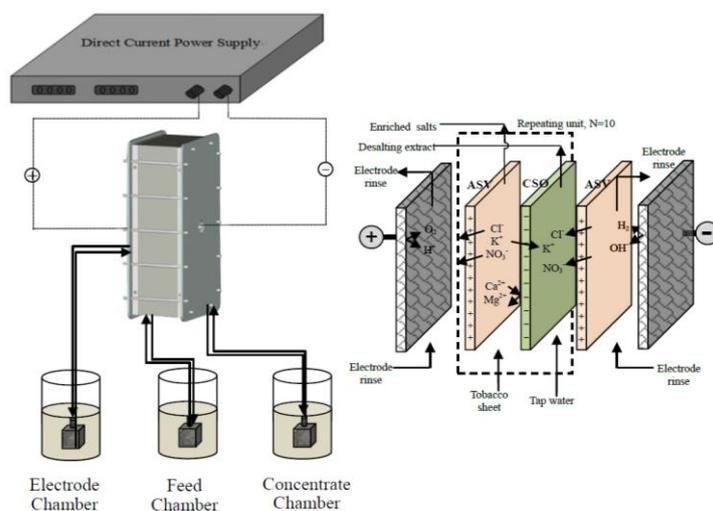


Fig. 1 Schematic diagram for the experiment stack and the main principle of electrodiagnosis process

### 2.3 Energy consumption

The energy consumption ( $E$ ,  $\text{kWh}\cdot\text{L}^{-1}$ ) of the ED process was calculated as Eq. (1),

$$E = \int_0^t \frac{UI dt}{V} \quad (1)$$

$U$  (V) is the voltage drop across ED stack;  $I$  (A) is the current applied;  $V$  is the volume of feed compartment; Removal rate of a particular  $i$  ions ( $R_i$ ) was calculated as Eq. (2),

$$R_i = \frac{C_{i,0} - C_{i,t}}{C_{i,0}} \times 100\% \quad (2)$$

$C_{i,0}$  and  $C_{i,t}$  are the concentration of  $i$  ions in the feed chamber at time 0 and  $t$ , respectively. The permselectivity ( $T_B^A$ ) of  $A$  ions against  $B$  ions during the electrodiagnosis process was calculated as Eq. (3),

$$T_B^A = \frac{\frac{J_A}{C_A}}{\frac{J_B}{C_B}} \quad (3)$$

Where  $J_A$  and  $J_B$  ( $\text{mol}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$ ) are the fluxes of  $A$  and  $B$  ions transported from feed into the concentrate chamber, respectively;  $C_A$  and  $C_B$  ( $\text{mol}\cdot\text{L}^{-1}$ ) are the initial concentration of  $A$  and  $B$  ions in the feed solution, respectively.

### 2.4 Analytical methods

The conductivity of the feed solution (dilute chamber) was monitored by a conductivity meter

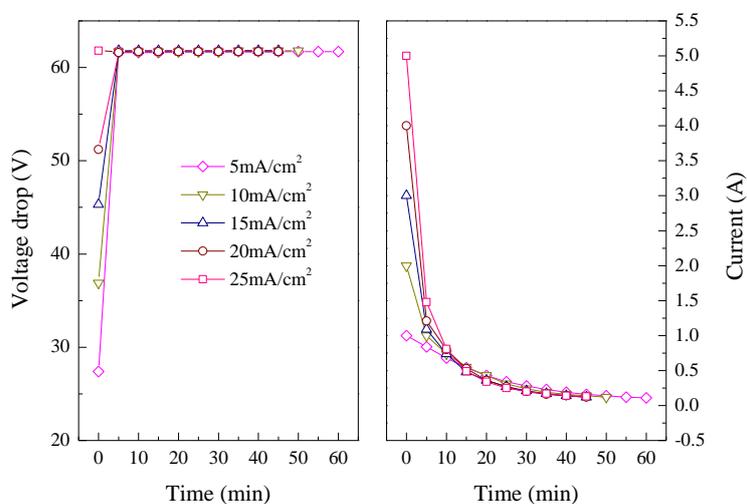


Fig. 3 Effect of initial current density on the voltage and current drops in ED stack

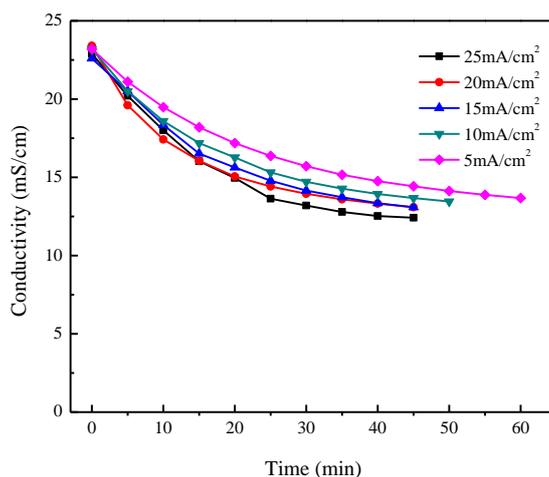


Fig. 4 The conductivities changes in the dilute chamber under different initial current density

(DDS 307, Shanghai INESA Scientific Instrument Co. Ltd., China). The concentrations of inorganic ions were determined by Ion Chromatography (ICS3000 multifunctional ion chromatography and ED electrochemical detector, DIONEX Company, USA). To determine the yields of CO, HCN, NH<sub>3</sub>, phenol, crotonaldehyde and total particulate matter (TPM) in cigarette mainstream smoke, cigarettes made from PRT were smoked by a smoking machine (Borgwaldt RM200A), according to the selected smoking conditions (35 mL puff volume, 2 s duration, 1 puff per min) of International Standard (ISO) recommendations (ISO 4387 1991). The harmful components in cigarette mainstream smoke were determined according to previous study (Zhou *et al.* 2014).

### 3. Results and discussion

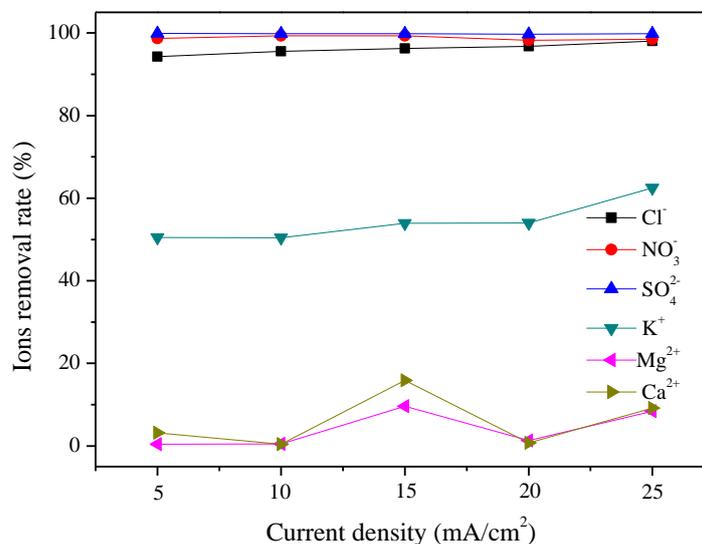


Fig. 5 The removal rate of individual ion under different initial current density

### 3.1 Effect of current density

The applied current density is the most prominent factor affecting the performance of electro dialysis process. A high current can save the capital cost of the ED project, but high current density will reduce the lifespan of membranes. A low current is beneficial to the current utilization and reducing the energy consumption. Fig. 3 shows the effect of initial current density on the voltage and current drops in ED stack for 20 wt% tobacco sheet extract. It is clearly indicated that voltage drops are increased sharply to reach a plateau. Meantime, the higher the current density is, the higher the initial voltage will be. This is consistence with the Ohm's law. The applied currents decreased with the elapse of time. Fig. 4 presents the conductivities changes in the dilute chamber under different initial current density. The experiments were stopped when the conductivities in the feed solution are not decrease in more than one minute. It can be seen that the conductivities in the feed solution are decreased with the elapse of time, indicating the removal of charged ions during the electro dialysis process. The conductivities at high currents are slightly lower than that at low current densities. It requires less time for the higher currents compared with the lower currents. It is logically truth since the driven force of an electro dialysis is proportional to the applied current. There is no pronounced difference in the desalination performance for initial current density in the range of 15-25 mA/cm<sup>2</sup>. There is due to a similar actual current applied at the ED stack even with different set values of initial current.

Fig. 5 shows the removal ratios of different ions for different initial current density. It is clearly indicated that the majorities of inorganic anions are removed with the removal ratios higher than 90% during the ED process. But things are different for the inorganic cations. The removal ratios for potassium ions can reach 50-60% and slightly increase with an increase in the initial current density. But the removal ratios for divalent cations such as magnesium and calcium ions are fluctuated less than 10% under the investigated current density. According to the characteristics of mono-selective ion exchange membrane, it allows the transport of monovalent counter-ions and retards the transport of co-ions and divalent and multivalent counter-ions. It should be noted that

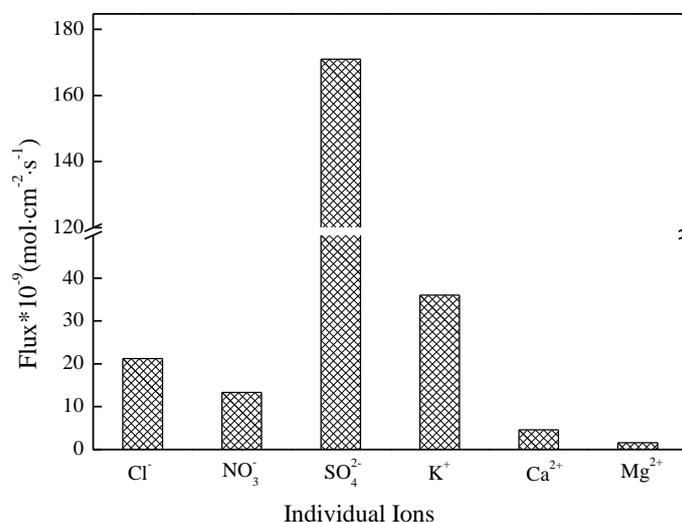


Fig. 6 The fluxes of individual ions transporting across the ion exchange membranes during the selective electro dialysis process at initial current density of 15 mA/cm<sup>2</sup>

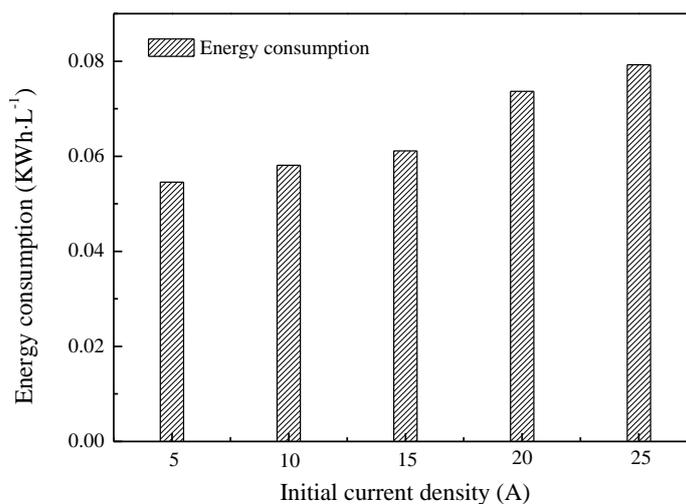


Fig. 7 The energy consumption of the electro dialysis process under different initial current density

the permselectivity of monovalent ions is actually a relative concept, a completely separation of monovalent and divalent/multivalent ions is not feasible in practical situation. Nevertheless, the objective of study is achieved in this experiment since the most undesirable chloride and nitrate ions were removed while the desirable magnesium and calcium divalent ions were mainly retained in the feed solution. But it seems that the permselectivity of the Selemion CSV cation-selective membranes is superior to Selemion ASO anion-selective membranes by considering the divalent sulfate ions are also completely removed compared with a small fractional of divalent metal ions are removed. To further compare the selectivity of individual ions, the fluxes of ions transport during the electro dialysis process at initial current density of 15 mA/cm<sup>2</sup> are plotted in Fig. 6. It

can be seen that the fluxes of sulfate ions is nearly 10 times of the chloride and nitrate ions considering the high content of sulfate ions in the tobacco sheet extract. In case of the cations, the flux of potassium ions is two magnitudes higher than the divalent ions. The permselectivity coefficients of monovalent ions against the divalent ions are calculated in the following Eqs. (4.1)-(4.4). It is clearly indicated that there are similar permselectivities for monovalent anions against the divalent sulfate ions in the ASV anion exchange membranes. There are high permselectivities for the monovalent potassium ions against the divalent magnesium and calcium ions. The permselectivity of the tested cations across the CSO cation exchange membranes follows the order:  $K^+ > Ca^{2+} > Mg^{2+}$ .

$$T_{SO_4^{2-}}^{Cl^-} = \frac{\frac{J_{Cl^-}}{C_{Cl^-}}}{\frac{J_{SO_4^{2-}}}{C_{SO_4^{2-}}}} = \frac{21.2 \times 10^{-9} (mol \cdot cm^{-2} \cdot s^{-1}) / 0.281 (mol \cdot L^{-1})}{171 \times 10^{-9} (mol \cdot cm^{-2} \cdot s^{-1}) / 2.187 (mol \cdot L^{-1})} = 0.96 \quad (4.1)$$

$$T_{SO_4^{2-}}^{NO_3^-} = \frac{\frac{J_{NO_3^-}}{C_{NO_3^-}}}{\frac{J_{SO_4^{2-}}}{C_{SO_4^{2-}}}} = \frac{13.3 \times 10^{-9} (mol \cdot cm^{-2} \cdot s^{-1}) / 0.170 (mol \cdot L^{-1})}{171 \times 10^{-9} (mol \cdot cm^{-2} \cdot s^{-1}) / 2.187 (mol \cdot L^{-1})} = 1.00 \quad (4.2)$$

$$T_{Ca^{2+}}^{K^+} = \frac{\frac{J_{K^+}}{C_{K^+}}}{\frac{J_{Ca^{2+}}}{C_{Ca^{2+}}}} = \frac{36.0 \times 10^{-9} (mol \cdot cm^{-2} \cdot s^{-1}) / 0.852 (mol \cdot L^{-1})}{4.6 \times 10^{-9} (mol \cdot cm^{-2} \cdot s^{-1}) / 0.372 (mol \cdot L^{-1})} = 3.42 \quad (4.3)$$

$$T_{Mg^{2+}}^{K^+} = \frac{\frac{J_{K^+}}{C_{K^+}}}{\frac{J_{Mg^{2+}}}{C_{Mg^{2+}}}} = \frac{36.0 \times 10^{-9} (mol \cdot cm^{-2} \cdot s^{-1}) / 0.852 (mol \cdot L^{-1})}{1.63 \times 10^{-9} (mol \cdot cm^{-2} \cdot s^{-1}) / 0.216 (mol \cdot L^{-1})} = 5.60 \quad (4.4)$$

Fig. 7 shows the effect of initial current density on the energy consumption of the selective electro dialysis process. It can be seen that the energy consumptions are increased with an increase in the applied current density. This suggested that low currents have better current utilization compared with the high currents. This conclusion is consistency with the results in the literature (Strathmann 2011). The selective electro dialysis process is preferable to be operated at a low current for the terms of current efficiency. But it is difficult to calculate the current efficiency due to the complex of ions transport in the tobacco sheet extract. In fact, there is often a trade-off to be made between high and low current during the practical applications of the ED process. Higher current densities are not attractive due to high energy costs related to increased ohmic drop and lower current densities are not attractive due to the large required membrane area. A current density of 15 mA/cm<sup>2</sup> is an optional choice by considering both the energy consumption and separation efficiency.

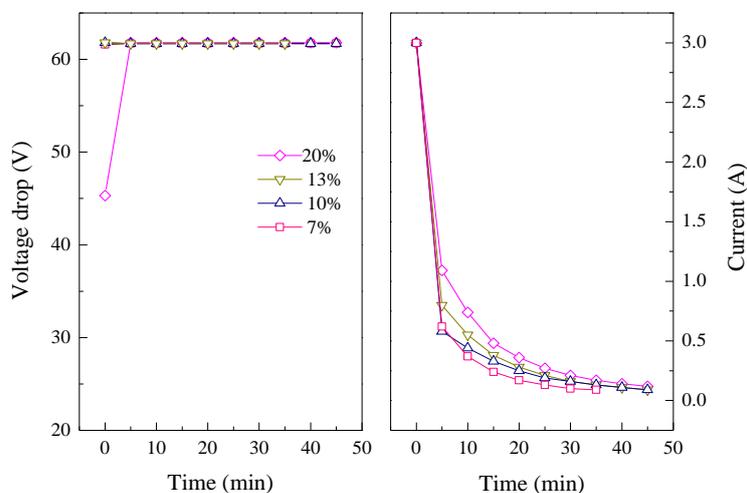


Fig. 8 Effect of tobacco sheet extract contents on the voltage and current drops in ED stack

### 3.2 Effect of tobacco sheet extract content

The extract content has a significant impact on the desalination performance of the electro dialysis process. In most case, ED is preferred to be operated at a high concentration of electrolytes not only to decrease the resistance of stack but also to alleviate the subsequent condensation process. But high solid contents cause poor flowability in the narrow flow passage of the ED stack. A low linear velocity of the flow may accelerate the fouling of membranes by considering the complex composition in the tobacco sheet extract. Therefore, the content of tobacco sheet extract must be carefully considered. Fig. 8 presents the voltage and current changes in the ED stack under different tobacco sheet extract with the initial current density of  $15 \text{ mA/cm}^2$ . It shows the voltages sharply increases to the maximum value of the current supplier in less than 5 min for the 20 wt% contents of extract solution. The other contents of tobacco sheet extract operated at the voltage-constant mode since the initiation of the experiments. The current drops sharply with the elapse of time. The current of the high extract content is slightly higher than that of the low extract content after 5 minutes. This is easily understood since a high concentration of electrolyte causes a low resistance in the solution. Fig. 9 shows the conductivities changes in the dilute chamber under different tobacco sheet extract contents. It can be seen that the conductivities in the feed solutions are decreased with the elapse of time, indicating the desalination of the tobacco extract caused by the ED process. This is consistent with the voltage drop curves. But the desalination rate is less than 50% using the selective electro dialysis with the monovalent ion-selective membranes. In comparison, a completely desalination of the tobacco sheet extract is achieved using the conventional electro dialysis process (Zhang *et al.* 2014). Fig. 10 indicates the removal rate of individual ion under different contents of the tobacco sheet extract. The removal ratios of the anions can reach 90% for all the investigated contents of the tobacco sheet extract. The removal ratios the potassium ions are kept at about 60%. The removal ratios of the calcium and magnesium ions fluctuate in the range of 16-27% and 8-14%, respectively. The desalination ratio of the tobacco sheet extracts are not improved by diluting the extract. A 20% tobacco sheet extract is an appropriate content by considering both the desalination efficiency and subsequent condensation process.

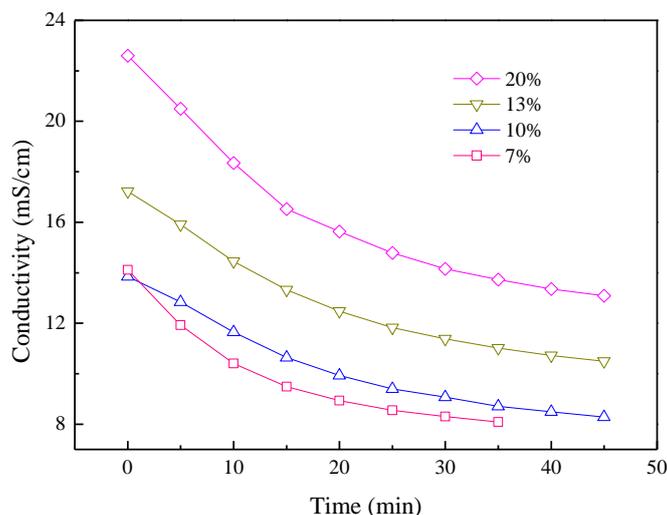


Fig. 9 The conductivities changes in the dilute chamber under different tobacco sheet extract contents

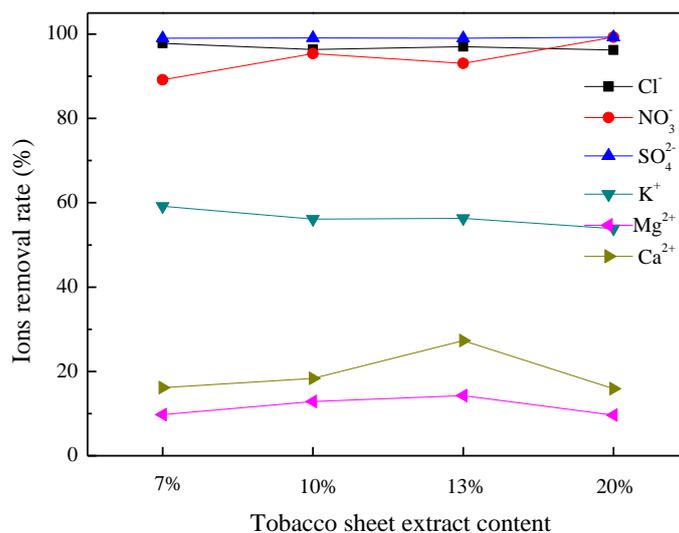
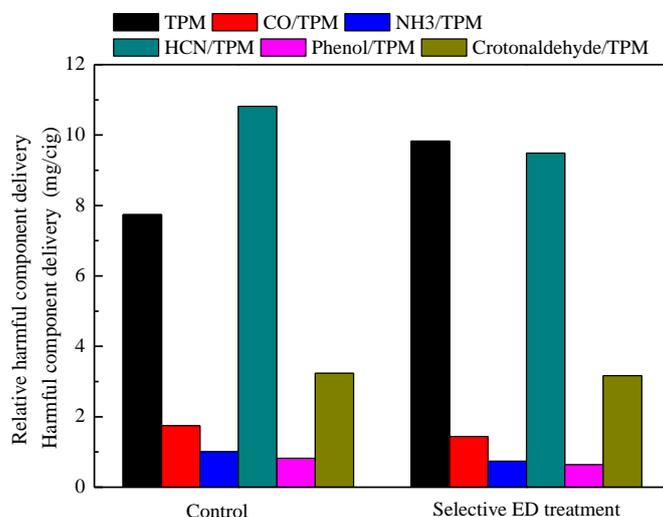


Fig. 10 The removal rate of individual ion under different contents of the tobacco sheet extract

### 3.3 The influence of desalting tobacco sheet extract using selective electrodialysis on the delivery of harmful mainstream tobacco smoke

According to health concerns, CO, NH<sub>3</sub>, HCN, Phenol, and crotonaldehyde are classified as toxic and carcinogenic constituents (Zhou *et al.* 2011). Fig. 11 shows the influence of desalting tobacco sheet extract on the delivery of five harmful components in the tobacco smoke. The control experiment is conducted with tobacco sheet extract without the ED treatment. It indicates that the removal of inorganic ions lead to an increase in the total particulate matter (TPM). The relative tobacco smoke components such as CO, NH<sub>3</sub>, HCN and phenol delivery are decreased compared with the control cigarette. It seems the inorganic components have no influence on the



generation of crotonaldehyde. In comparison with our previous work (Ge *et al.* 2016), it is found that TPM emission in the cigarette made from the desalting tobacco extract using selective electro dialysis is less than that using the conventional electro dialysis. The relative values of CO, NH<sub>3</sub>, and Phenol (except HCN) delivery in the selective electro dialysis are lower than that in the conventional electro dialysis. These results indicate that the divalent metal ions have dual roles for the harmful component delivery in cigarette smoke. The divalent potassium and calcium ions are unfavorable for the total particulate matter emission but are beneficial to decrease the HCN delivery in the mainstream cigarette smoke. But the detailed mechanism for the influence of divalent metal ions on the delivery of HCN is still unknown and should be further studied. Nevertheless, the present study indicates that the removal of monovalent inorganic components using selective electro dialysis is beneficial to decrease the harmful component delivery in cigarette smoke.

#### 4. Conclusions

Laboratory-scale experimental set-up was used to desalt the tobacco sheet extract by using selective electro dialysis with monovalent ion-selective ion exchange membranes. Results indicated that removal ratios for potassium ions can reach 50-60% and slightly increase with an increase in the initial current. But the removal ratios for divalent cations such as magnesium and calcium ions fluctuate less than 10% under the investigated current density of 5-25 mA/cm<sup>2</sup>. The energy consumption were increased with an in the current. High currents could improve the desalting efficiency but cause higher energy consumption. Low currents have high current utilization but require higher capital cost. The permselectivity of the tested cations across the Selemion CSO cation exchange membranes follows the order: K<sup>+</sup>>Ca<sup>2+</sup>>Mg<sup>2+</sup>. A current density of ~15 mA·cm<sup>-2</sup> is an optional choice by considering both the energy consumption and desalting efficiency. When the extract contents are in the range of 7%-20%, the removal ratios the potassium ions are kept around 60%, while the removal ratios of the calcium and magnesium ions fluctuate in the range of 16-27% and 8-14%, respectively. The tobacco smoke result indicates that the removal of inorganic

ions lead to an increase in the total particulate matter (TPM). But the relative contents of CO, NH<sub>3</sub>, HCN and phenol delivery are decreased compared with the control cigarette. It is interesting to find the divalent metal ions may have dual roles for the harmful component delivery in cigarette smoke. The divalent potassium and calcium ions were unfavorable for the total particulate matter emission but beneficial to decrease the HCN delivery in the mainstream cigarette smoke. But the detailed mechanism for the influence of divalent metal ions on the delivery of HCN is still unknown and should be further studied. Nevertheless, our serious studies indicate that ED technology is a potential technology not only to improve the smoking quality of cigarette but also to decrease the harmful component delivery in cigarette smoke.

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