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# Removal of inorganic contaminants in sugar refining process using electrodeionization

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#### ABSTRACT

This paper reports performance of an electrodeionization (EDI) system during removal of inorganic contaminants in sugar refining process. Diluate compartments of EDI stack were filled with mixed ion-exchange resins (strong acid cation-exchange and strong base type I anion resins). Experiments were then conducted at both batch and continuous operation mode. The results showed that EDI is able to remove inorganic contaminants as well as colour body from sugar solutions. At relatively high sugar concentration, significant decrease in ions removal is observed. The increase of current density and applied voltage had little impact on ions removal and lead to current efficiency and pH reduction. The colour removal may be associated to complex bond (organic–inorganic) breakage due to water dissociation inside the diluate compartment.

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#### 1. Introduction

Inorganic contaminant which is present in sugar solution can cause many problems in sugar refinery. High concentration of inorganic ions (calcium ion) cause scales in evaporators and crystallizers (Arslanoglu and Tumen, 2012). Hence, it will reduce heat transfer efficiency and increase power consumption. Moreover, high concentration of inorganic contaminants lead to lower yield of sugar crystallization because they hold sugar in mollases (Arslanoglu and Tumen, 2012). Therefore, it is important to reduce quantity of inorganic contaminants.

Since 1970s, numerous patents for refining sugar solution have been granted (Farag and Norman, 1978; Rousseau and Lamotte, 1982; Stringfield et al., 1990; LaBrie and Bharwada, 1992). Basically, most of these patented methods are based on adsorptive separation using ion-exchange resins. In a certain case, it is combined with activated carbon. The ion exchange resins are periodically regenerated by concentrated acid and caustic solutions. Despite of high chemical consumption and labor intensive, this technology is also producing a large volume of wastewater.

Recently, Electrodialysis (ED) has been used in removal of melassigenic ions, especially  $Na^+$  and  $K^+$  from cane sugar juice (E1 Khattabi et al., 1996). It was able to removed up to 85% of ionic

content. Results also showed that the membranes have long life and needed a simple pretreatment to avoids contamination of membranes by organic components. However, the pH has fallen to an unacceptable value which can cause a decomposition of sugar.

Conductive ED, well known as electrodeionization (EDI), is an electrically driven membrane process combining ion exchange resin and electrodialysis. An EDI stack consists of diluted compartments, concentrated compartment, and electrode compartments, in which the diluted compartments are filled with mixed ionexchange resins. Due to an influence of electrical field, cations and anions in solution are attracted to cathode and anode respectively with the mixed resins as a conducting media. A relatively high concentration of H<sup>+</sup> and OH<sup>-</sup> ions are produced due to water splitting reaction which will regenerate the mixed resins continuously. Hence, EDI can be continuously operated without chemical regeneration. Since dilute solution has relatively high electrical resistance, using electrically active media (ion exchange resin filler) as the bridge over current is a strategy to decrease the resistance and the energy consumption of the ED stack (Huang et al., 2007). By adopting this strategy, low concentration solution can be treated by using EDI process with high efficiency and higher removal rate.

After the first commercial unit introduction in 1987 (Ganzi et al., 1987), EDI has continued to be an attractive deionization process with various advantageous than conventional ion exchange deionizaton (IX) in production of Ultrapure Water







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(UPW) from technological and economical standpoint (Wood et al., 2010; Fedorenko, 2004). The main reason for EDI commercial success is its capability on eliminating regeneration process and associated hazardous chemical. As a chemical free operation or environment-friendly technology, this process has dominant the choice of alternative technology for large scale UPW production (Wood et al., 2003; Ho and Wood, 2006; Hernon et al., 2010). EDI is mainly applied to water and wastewater treatment, but it has also shown great potential to be applied in a number of different applications. The use of this technique in biotechnology and organic acid removal is also challenging (Widiasa et al., 2004; Arora et al., 2007; Boontawan et al., 2011).

The use of EDI for ions removal from glucose/maltose syrup was investigated by Widiasa et al. (2002) and Widiasa and Wenten (2003). They found that the quality of the syrups product was stable with ionic impurities less than 1 ppm. Subsequently, Widiasa and Wenten (2007) evaluated the combination of RO-EDI for sugary wastewater treatment. They discovered that the hybrid process allowed almost total sugar recovery and significant reduction of salts content. The objectives of the present study is to further investigate the potential of EDI process in sugar refining. Various process parameter were assessed to study the behavior of EDI during decolorizing and desalting sugar solution.

#### 2. Experimental

#### 2.1. EDI system set up

The EDI stack had two diluted compartments, one concentrated compartment, one anode compartment, and one cathode compartment. Commercially, cation-exchange membrane (MC-3470) and anion-exchange membrane (MA-3475) were used as ionic selective barriers of the EDI stack (Table 1). The effective surface area of each membrane was 140 cm<sup>2</sup>. All diluted compartments were filled with mixed-bed ion-exchange resins (purolite strong acid cation-exchange, C-100E and strong base type I anion resins, A-400) with the properties as summarized in Table 2. Platinum and stainless steel were used for anode and cathode, respectively. The internal spacer for each compartment was 3 mm. An adjustable power supply (model PAB, made by Kikusui Electronics Corp.) was used to produce direct current. It could supply voltage and direct current in the range of 0–70 V and 0–1.2 A, respectively.

#### 2.2. Experimental procedure

Model solution was prepared by dissolving raw sugar (low grade sugar, colour: about 300 IU) and NaCl (Pro analysis, Merck) into deionized water in various sugar concentration (10-40% w/w). The EDI process involved three independent streams: feed solution, concentrated solution, and electrode solution streams. Sugar solution with ions concentration was circulated in diluate

#### Table 1 Typical characteris

Typical characteristics of ion exchange membrane.

compartment. The EDI system was operated both batch mode and continuous operation mode (Fig. 1a and b). The flowrate of the feed solution was varied in the range of 1.8–5 L/h. The influences of various operating conditions on EDI performance were assessed.

### 2.3. Analytical method

Ion concentration was analyzed using TDS meter (TDS-3 type from HM digital, unit: ppm). The pH of solution at the cell outlet was measured using a pH-meter (pHep, Hanna Instrument). Colour was analyzed based on absorbance of spectrophotometry (Shimadzu, UV-120-02) at 420 nm. Colour Unit was defined as ICUMSA unit (IU) according to the following equation (Mudoga et al., 2008):

$$U = 1000 \cdot A / (C \cdot b)$$

where *A* is absorbance at 420 nm, b is length of adsorbing path (cm) and *C* is concentration of sample (g sugar/mL).

#### 3. Results and discussion

#### 3.1. Ions removal

Fig. 2 shows ions concentration of diluate stream according to batch operation mode in various sugar concentration. The initial volume, ions concentration, and pH of sugar solution were 500 mL, 52 ppm, and  $7.2 \pm 0.2$ , respectively. As shown in Fig. 3, the increase of sugar concentration, between 10% and 30%, did not significantly reduce ions transfer. There were 4.5% and 6% reduction in total ion removal when the sugar concentration increased from 10% to 20% and 30%, respectively. Meanwhile, the total ion removal decreased significantly when the sugar concentration was increased further. It dropped from 69% to 52.7% when sugar concentration was increased from 10% to 40%.

Mechanism of ions removal in EDI consists of two main steps as reported by Ganzi et al. (1992). The first step is a process of which the ions in the feed are bound by the ion exchange resins, wherein the cations are exchanged with  $H^+$  ions and anions are exchanged with  $OH^-$  ions. The second step is a process of which the ions bound to the resins are transported by the electrical force through the ion exchange membrane surfaces into the concentrate compartment. These two main principles only occur at the early stage. After the process reaches its steady state, ion exchange resins mainly function as ionic bridge that will increase the overall conductivity of the diluate compartment.

In sugar solution, the viscosity of the solution exponentially increases with sugar concentration. The viscosity of 40% sugar solution is 2–3 times greater than 30% and 20% concentrated solution. The reduction in ion removal at higher sugar concentration could be attributed to the decline in solution conductivity. It seems that the increase of viscosity leads to an increase in solution resistance

	Units	Cation, MC-3470	Anion, MA-3475
Thickness	mm	0.38	0.40
Exchange capacity	meq/g	1.4	0.9
Mullen burst test, min	Bar	10.3	10.3
Area resistance	Ohm/cm		
	0.1 N NaCl	25	50
	1.0 N NaCl	10	25
Permselectivity	0.5 N NaCl/1.0 N NaCl	96	99
Water permeability	lmh @ 0.3 bar	0.27	0.27
Temperature stability, max	°C	80	80
Chemical stability, pH		1-10	1–10
Current density, max	Ampere/m <sup>2</sup>	537	537

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