THE DEACIDIFICATION OF PINEAPPLE JUICE USING ELECTRODIALYSIS WITH MONOPOLAR ION EXCHANGE MEMBRANES

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Specially dedicated to my beloved mak, abah, Achik, Abang Dzul, Jojo, Angah, Abang Chik, Alang, Along and my fiancé, Hadi for all the encouragement and help during the hard time…

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ABSTRACT

The objective of this research was to study the performance of the electrodialysis using monopolar ion exchange membranes for the deacidification of pineapple juice. Based on preliminary experimental data using citric acid solution, AFN monopolar anion exchange membrane was selected for the subsequent study of the deacidification of pineapple juice. Electrodialysis of pineapple juice resulted in the reduction of the conductivity and the total acidity in the diluted stream. The deacidification value was 27.53 %. Deacidification process unexpectedly decreased the pH value from 4.01 to 3.75. However the Brix values were essentially unchanged (from 10.50 to 10.17 % Brix). A slight fluctuation in sugar compositions during electrodialysis was observed after 80 minutes of the processing. The sugar analysis was carried out using High Performance Liquid Chromatography (HPLC). HPLC analysis also showed that citric acid and malic acid compositions were always higher in the concentrated stream as compared to the diluted stream. The elemental analysis of the pineapple juice indicated that the composition of magnesium, phosphorus and potassium were reduced in the diluted stream but increased in the concentrated stream. The results from sensory evaluation test showed a higher overall acceptance for the electrodialysed pineapple juice compared to the fresh juice. The values of the overall acceptance were 66.0 % and 56.0 %, respectively. The findings showed that electrodialysis offer a better processing route and produce a more acceptable product for the deacidification of pineapple juice.

ABSTRAK

Objektif penyelidikan ini adalah mengkaji prestasi elektrodialisis menggunakan membran pertukaran ion monopolar bagi penyahasidan jus nanas. Berdasarkan data eksperimen permulaan menggunakan larutan asid sitrik, membran pertukaran anion monopolar, AFN dipilih untuk kajian penyahasidan jus nanas. Elektrodialisis jus nanas menyebabkan pengurangan nilai konduktiviti dan asid keseluruhan di dalam aliran pencairan. Nilai penyahasidan adalah 27.53 %. Nilai pH berkurang tanpa dijangka daripada 4.01 kepada 3.75. Walaubagaimanapun, nilai Brix tidak banyak berubah (daripada 10.50 kepada 10.17 % Brix). Komposisi gula semasa elektrodialisis menunjukkan sedikit turun naik selepas minit ke-80 pemprosesan. Analisis gula dijalankan menggunakan Kromatografi Cecair Prestasi Tinggi (*HPLC*). Analisis *HPLC* turut menunjukkan kandungan asid sitrik dan asid malik di dalam aliran pemekatan sentiasa lebih tinggi daripada aliran pencairan. Analisis elemen jus nanas menunjukkan komposisi magnesium, fosforus dan kalium berkurang di dalam aliran pencairan tetapi bertambah di dalam aliran pemekatan. Keputusan ujian penilaian sensori menunjukkan penerimaan keseluruhan yang lebih tinggi bagi jus nanas yang diproses menggunakan elektrodialisis berbanding jus segar, di mana nilai masing-masing adalah 66.0 % dan 56.0 %. Penemuan ini menunjukkan elektrodialisis menyediakan aliran pemprosesan yang lebih baik dan menghasilkan produk yang lebih diterima bagi penyahasidan jus nanas.

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LIST OF SYMBOLS

A	-	Ion transport rate constant (M/(A.s))
A, AEM	-	Anion exchange membrane
B	-	Ion transport rate constant (1/(A.s))
BMED	-	Bipolar-membrane electrodialysis
C, CEM	-	Cation exchange membrane
CD	-	Current density
CD_{lim}	-	Limiting current density
CE	-	Current efficiency
CP	-	Concentration polarization
CS	-	Concentrating stream
D	-	Diffusion coefficient
DC	-	Direct current
DS	-	Diluting stream
ED	-	Electrodialysis
ERS	-	Electrode rinse solution
F	-	Faraday constant (96500 $C.mol^{-1}$)
HPLC	-	High Performance Liquid Chromatography
i	-	Current density
I	-	Current, Current intensity
ICP-MS	-	Inductively Coupled Plasma Mass Spectrometer
IEM	-	Ion exchange membrane
I-V curve	-	Current-voltage curve
m, n	-	Empirical constant
n	-	Average current efficiency
pK	-	Dissociation constant
R	-	Gas constant

S	-	Effective membrane area
SS	-	Soluble solid
t	-	Time
T	-	Temperature
TA	-	Titratable acidity
v	-	Velocity of convection transport, Feed solution velocity
V	-	Voltage
x	-	Direction of coordinate
z	-	Electrochemical valence
α	-	Phenomenological coefficient (ml/s)
β	-	Phenomenological coefficient ($ml.cm^2/(A.s)$)
ϕ	-	Electrical potential
H^+	-	Hydrogen ion
OH^-	-	Hydroxide ion
C_0	-	Feed concentration
J_k	-	Flux of component k
Q_F	-	Amount of electrical charges expressed in Faraday
$\frac{dV}{dt}$	-	Water transport rate
C_c^0	-	Concentration at time = 0 in the concentrate stream
C_c^t	-	Concentration at time = t in the concentrate stream
C_d^0	-	Concentration at time = 0 in the diluate stream
$n_{citric}^0(0)$	-	Number of moles of citric acid in the concentrating stream at initial stage
$n_{citric}^c(t)$	-	Number of moles of citric acid in the concentrating stream at time t
V_c^0	-	Initial volume of the concentrating stream
V_c^t	-	Volume of the concentrating stream at time t
Δn	-	The difference in the number of moles of citric acid in the concentrating stream at time t and at initial stage
ΔV_c	-	Volume of water transported from diluting stream to concentrating stream

+	-	Anode
-	-	Cathode
(% w/v)	-	Percent by weight

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CHAPTER 1

INTRODUCTION

1.1 Electrodialysis and Ion Exchange Membrane

Electrodialysis is an electrochemical membrane separation process in which electrically charged membranes and an electrical potential difference used to separate ionic species from aqueous solution and other uncharged components (Strathmann, 1992; Bazinet *et al*., 1998). The separation is not based on the size of the particles, but by their electrical charge (Bazinet *et al*., 1998).

The interest in electrodialysis as a membrane separation process due to the developments in new membrane materials that offer better stability and performance, thus facilitating for new applications (such as bipolar membrane) (Bazinet *et al*., 1998). The applications of electrodialysis include the desalination process, production of table salt from seawater, demineralization of cane sugar, desalination of cheese whey, deacidification of fruit juices, wines stabilization, production of acids and bases from a salt stream and to the recovery of concentrated acids (Strathmann, 1992; Alvarez *et al*., 1997; Bazinet *et al*., 1998; Ling, 2002).

The ion exchange membranes are the most important component in the electrodialysis equipment. Ion exchange membranes are made of polymer matrix material with fixed charged groups covalently bonded to the polymer matrix (Strathmann, 1992). The ion exchange membranes are subdivided into two basic categories, which are the monopolar membranes and the bipolar membranes. The type of fixed charged groups attached to the polymer matrix, determines the classification of ion exchange membrane (Ling, 2002). Monopolar membranes can be further classified into anion exchange membrane and cation exchange membrane. A membrane containing fixed negative charges is called a cation exchange membrane, while a membrane with fixed positive charges is referred as anion exchange membrane (Strathmann, 1981).

1.2 Deacidification of Pineapple Juice by Electrodialysis

Pineapple or *Ananas comosus* is a popular tropical fruit due to its unique aroma and pleasant flavour. A ripe pineapple is juicy, succulent and has a tangy sweet-tart flavour. Pineapple juice is obtained by squeezing pineapple fruit. Pineapple juice contains 12 % - 15 % (w/v) sugars of which two-third is in the form of sucrose and the rest are glucose and fructose. 0.6 % - 1.2 % (w/v) of pineapple is acid of which 87 % is citric acid and 13 % is malic acid (Samson, 1986; Adhikary *et al*., 1987). The pH of pineapple juice is in the acidic region, which is of around 3.71 and the acidity percentage is 53.5 %. The composition of the juice varies with geographical, seasonal harvesting and processing.

Several methods have been attempted to reduce the sour taste of pineapple juice, due to its high acidity. The methods include adding sweetening agents, simple neutralization by alkali and partially removal of the acid by ion-exchange techniques (Bhatia *et al*., 1979; Adhikary *et al*., 1983; Lue and Chiang, 1989).

At present, there is an increase demand for fruit juices with authentic characteristics of fresh fruits and free from chemical additives. As a result, the industry is searching for new technology that could maintain the sensorial and nutritional quality of the fruit juices (Carneiro *et al*., 2002). The conventional thermal processing method reduced the quality of the pineapple juice. An alternative pineapple juice processing is by using electrodialysis. The advantages of electrodialysis in relation to the thermal processing are the use of mild temperature and pressure conditions, which maintain the nutritional quality and the sensorial attributes of the products. Hence a study is warranted to develop pineapple juice with acceptable acidity level using laboratory scale electrodialyzer equipment.

1.3 Objective of the Study

The objective of this research was to study the performance of monopolar ion exchange membranes during electrodialysis for the deacidification of pineapple juice.

1.4 Scope of the Study

In order to achieve the objective, the research is divided into three phases. The first phase involved the study on the effect of operating parameters on the performance of electrodialysis equipment using citric acid solution. The operating parameters studied were the current density, feed concentration and anion exchange membranes type. The study on limiting current density, water transport and ion transport were also conducted.

The second phase was to study the reliability of the developed limiting current density, water transport and ion transport equation to describe the performance of electrodialysis process using citric acid solution.

The third phase was to study the deacidification of pineapple juice using electrodialysis. The effect of electrodialysis on the physico-chemical and chemical characteristics of pineapple juice was investigated. The sensory analysis was also conducted to determine the acceptability of the processed pineapple juice using electrodialysis.

1.5 Organization of Thesis

The research work in this thesis deals with the application of electrodialysis with ion exchange membranes for deacidification of pineapple juice. The thesis is divided into six chapters. Chapter 1 presents the background and overview of the overall study. An introduction to electrodialysis, ion exchange membrane and its application in the process of pineapple juice deacidification are presented. The research objective and scope are also outlined.

The Chapter 2 of this thesis is the comprehensive literature review, including membrane separation technology, the basic principles of electrodialysis and ion exchange membranes, the applications of electrodialysis, limiting current density in electrodialysis process and ion transport in ion exchange membranes.

In Chapter 3, the background of pineapple, the pineapple and pineapple juice composition and pineapple industry in Malaysia are presented. Besides that, membrane processing of fruit juices and the conventional methods for fruit juice deacidification are presented. A review of the previous study on the application of electrodialysis with ion exchange membrane for deacidification of fruit juices is also cited.

Chapter 4 listed out the materials and equipments used in this study. The methods for the experimental work were also described. All the experimental results and discussion of the performance of electrodialysis to deacidify citric acid solution and pineapple juice are outlined in Chapter 5. This chapter also includes the analysis of limiting current density and water and ion transport. Finally, Chapter 6 concludes the findings from this research and with recommendations for future work.

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