Report of Cooperative Education

Feasibility Study of Self Production of Phosphoric Acid and Monosodium Phosphate from Sodium Phosphate Salt Using Bipolar Membrane Electrodialysis Technology

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รายงานสหกิจศึกษาฉบับสมบูรณ์

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Co-operative Title: Feasibility Study of Self Production of Phosphoric Acid and Monosodium Phosphate From Sodium Phosphate Salt Using Bipolar Electrodialysis Technology

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Abstract

Monosodium phosphate (NaH_2PO_4), sodium hydroxide (NaOH) and phosphoric acid (H_3PO_4) was used in ribonucleotide (Ajitide I+G) production process. Sodium phosphate salt (Na_3PO_4, Na_2HPO_4) was generated after processing, currently used for pH adjustment. Regeneration of phosphoric acid from excess sodium phosphate salt by using bipolar electrodialysis (BPED) was studied to reduce consumption of phosphorus from natural. For this study, the component of chemicals were identified the compositions using High Performance Liquid Chromatography (HPLC) for phosphate group, Atomic Absorption Spectrometry (AAS) for sodium content and measure moisture content by using moisture meter. The effect of important operating parameter (conductivity of chemicals, operating pH and temperature.) with bipolar electrodialysis performance was investigated in this study. The parameters that have direct proportional relation with electric current is feed conductivity and operating temperature while pH are disproportional relation cause of different of dissociation of chemical formula, phosphoric acid and sodium hydroxide conductivity have the suitable value to get low of overall electric resistance at 50 mS/cm and 240 mS/cm, respectively. This feasibility study can produce desired product and reduce sodium in feed stream to generate sodium hydroxide as a by-product.

Keywords: Bipolar electrodialysis, BPED, Sodium phosphate salt, Phosphoric acid, Sodium dihydrogen phosphate

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Table of Contents

Page

Chapter III. Research Methodologies

Chapter IV. Results and Discussion

Chapter V. Conclusions

List of Figures

Page

List of Tables

CHAPTER I

INTRODUCTIONS

1.1 Background

Sodium phosphate salts are undesired products from Ajitide I+G production process of Ajinomoto (Thailand) company limited at Kamphaeng Phet province, currently partially of sodium phosphate salts used for pH adjustment, recovery the raw material that used as phosphate source in phosphorylation reaction to produce Ajitide I+G and produce fertilizer by FD Green (Thailand) company limited.

Ajitide I+G is a ribonucleotide mixture proportion 1 to 1 of inosinate and guanylate in one crystal, being used as flavor enhancer for manufacturing of food for enhance umami taste like MSG. In Ajitide I+G production process used a lot of solvents, especially phosphoric acid and sodium hydroxide for pH adjustment in fermentation, neutralization and crystallization process to control the condition and operating to reach target yield. Nowadays, the company need to supply fresh solvents to use in the Ajitide I+G production process and charge for eliminate some of remained sodium phosphate salt.

This project wants to find the alternative way to recovery or the way to prepare the raw material for reuse in the production process by using undesired products from unit operation that already exists.

1.2 Objective

1.2.1 To study the feasibility of manufacturing phosphoric acid and monosodium phosphate from sodium phosphate salt by using bipolar membrane electrodialysis

1.2.2 To investigate the effect of parameters on electric consumption

1.2.3 To reduce the sodium component in sodium phosphate salt

1.3 Scopes of Work

1.3.1 Study properties and composition of sodium phosphate salt

1.3.2 Research the polyprotic production by using bipolar membrane electrodialysis

1.3.3 Operating bipolar membrane electrodialysis and investigate the experiment

1.3.4 End product results

1.3.5 Study and analyze economic value of bipolar membrane installation and operation

1.4 Expected Output

1.4.1 Produce phosphoric acid and monosodium phosphate from sodium phosphate salt in Ajinomoto plant by using bipolar membrane electrodialysis

1.4.2 Find suitable operating condition to operate bipolar membrane electrodialysis

1.4.3 Reduce operation cost in phosphate source recovery process

1.4.4 Alternative route for bipolar membrane electrodialysis to operate with polyprotic acid

CHAPTER II

THEORY AND LITERATURE REVIEWS

2.1 Sodium phosphate

Sodium phosphate is the general chemical name for a wide variety of salts produced from the reaction of phosphoric acid with sodium hydroxide or sodium carbonate. These salts can be divided into two classes including orthophosphates and complex phosphates.[1]

Chemical analysis predicts three salts resulting from the successive neutralization of the acidic protons of the phosphate group with sodium hydroxide or sodium carbonate. These salts are monosodium dihydrogen phosphate (NaH_2PO_4) , disodium monohydrogen phosphate or sodium orthophosphate (Na_2HPO_4) , and trisodium phosphate (Na_3PO_4) . Each of these salts is produced commercially in the anhydrous state, although disodium monohydrogen phosphate and trisodium phosphate are also produced commercially as hydrates.

The general term " condensed inorganic phosphates" is applied to phosphorus compounds in which various numbers of PO_4 groups are linked together by oxygen bridges. They fall into three classes including cyclic, linear and cross-linked condensed phosphates. In condensed inorganic phosphates, phosphorus is at the highest oxidation state (+5). Linear condensed phosphates, also called polyphosphates have the general elementary composition $[P_nO_{3n+1}]^{(n+2)-}$.[2]

Table 2.1 Sodium phosphates.[2]

Number of P Atoms	**Type**	**Example**	**CAS n°**
1	monophosphates/	Na_3PO_4	7601-54-9
	orthophosphates	Na_2HPO_4	7558-79-4
		NaH_2PO_4	7558-80-7
2	biphosphates/	$Na_4P_2O_7$	7722-88-5
	pyrophosphates	$Na_3HP_2O_7$	14691-80-6
		$Na_2H_2P_2O_7$	7758-16-9
		$NaH_3P_2O_7$	13847-74-0
3	triphosphates	$Na_5P_3O_{10}$	7758-29-4
		$Na_4HP_3O_{10}$	24616-37-3
		$Na_3H_2P_3O_{10}$	-
		…	
4	tetraphosphates	$Na_6P_4O_{13}$	-
		…	
…			

2.2 Phosphoric acid

Orthophosphoric acid (H_3PO_4) , usually referred to as phosphoric acid, is a clear, water-white liquid which is derived from phosphate rock. Phosphoric acid is a stronger acid than the organic acids, however it is not as strong as other mineral acids. Phosphoric acid is a useful synthesis catalyst since it does not react violently with many organic compounds and exhibits catalytic and dehydrating properties. It will react readily with bases to produce alkali phosphates and will become fairly reactive with metals and metal oxides at elevated temperatures. These properties give phosphoric acid a wide range of industrial applications from metal finishing to food additives.[3]

Pure anhydrous phosphoric acid is a white solid which melts at 42.35°C to form a viscous liquid. In aqueous solution, phosphoric acid behaves as a triprotic acid, having three ionizable hydrogen atoms. The hydrogen ions are lost sequentially.[3]

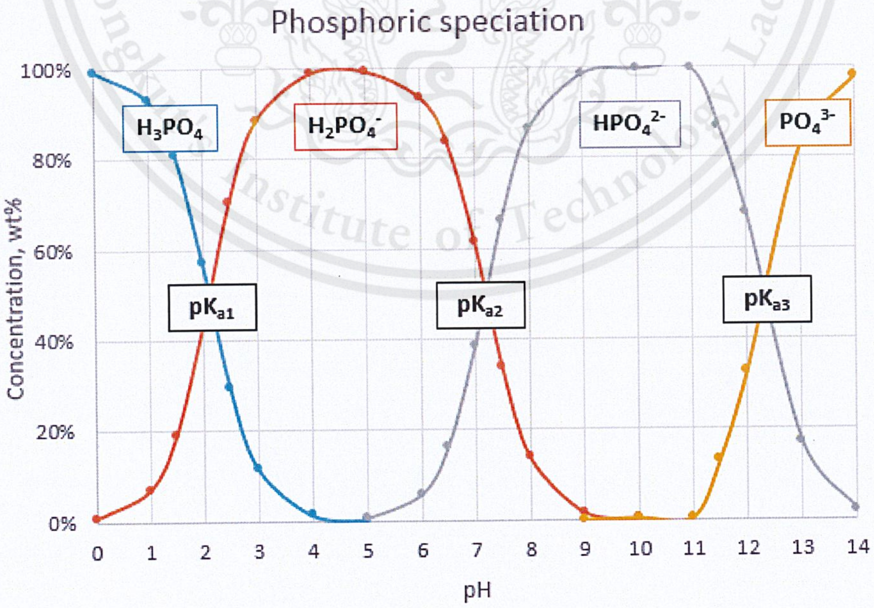

Figure 2.1 Fraction composition diagram for phosphoric acid system.[2]

$$H_3PO_{4(aq)} \longrightarrow H^{+}_{(aq)} + H_2PO^{-}_{4\,(aq)} \qquad K_{a1} = 7.5 \times 10^{3}$$

$$H_2PO^{-}_{4\,(aq)} \longrightarrow H^{+}_{(aq)} + HPO^{2-}_{4\,(aq)} \qquad K_{a2} = 6.2 \times 10^{8}$$

$$HPO^{-}_{4\,(aq)} \longrightarrow H^{+}_{(aq)} + PO^{3-}_{4\,(aq)} \qquad K_{a3} = 1.7 \times 10^{12}$$

Phosphoric acid is not a particularly strong acid as indicated by its first dissociation constant. It is a stronger acid than acetic acid, but weaker than sulfuric acid and hydrochloric acid. Each successive dissociation step occurs with decreasing ease. Thus, the ion $H_2PO_4^-$ is a very weak acid, and HPO_4^{2-} is an extremely weak acid.[4]

Salts of phosphoric acid can be formed by replacing one, two or three of the hydrogen ions. For example, sodium dihydrogen phosphate (NaH_2PO_4). Similarly, disodium hydrogen phosphate (Na_2HPO_4) and trisodium phosphate (Na_3PO_4) could be formed by the reaction of one mole of phosphoric acid with one, two and three moles of sodium hydroxide, respectively.

2.3 Sodium hydroxide

Caustic soda (sodium hydroxide or NaOH) is most commonly manufactured by the electrolysis of a sodium chloride (NaCl) solution. Caustic soda is shipped via pipelines, tank trucks, railcars, barges and ships. Terminals are used to maintain stocks of our caustic soda in many principal cities. Distributor stocks are also available in these and many other cities and form a network of supply for the end user's convenience. Sodium hydroxide is one of the most used chemical substances in laboratory and in industrial environment, in the manufacture of paper pulp and of various chemical products: plastics, synthetic textiles, cleaning products for both domestic and industrial use, in the production of petrol and biodiesel, of soaps or even in the aluminum treatment. It is also a food additive.[5]

Pure sodium hydroxide is a white solid. It is translucent and very hygroscopic (great ability to attract and hold water molecules). It reacts easily with the water from the air or from any wet surface (phenomenon of deliquescence). The dissolving of caustic soda in water may be accompanied by heat release. It is sold as: chips, flakes, granules, blocks, cubes or in aqueous solution. In the industrial environment, the most concentrated liquid form of sodium hydroxide is 50%, but then it is very viscous.[6]

2.4 Bipolar electrodialysis (BPED)

BPED is the combination of electrodialysis for salt separation with bipolar membranes for water splitting into its corresponding acid and base. The bipolar membranes are a special type of layered ion exchange membrane where two polymer layers one is only permeable for the anions and the other only for cations to get a reaction in the bipolar junction of the membrane where the anion and the cation permeable layers are in direct contact.[7]

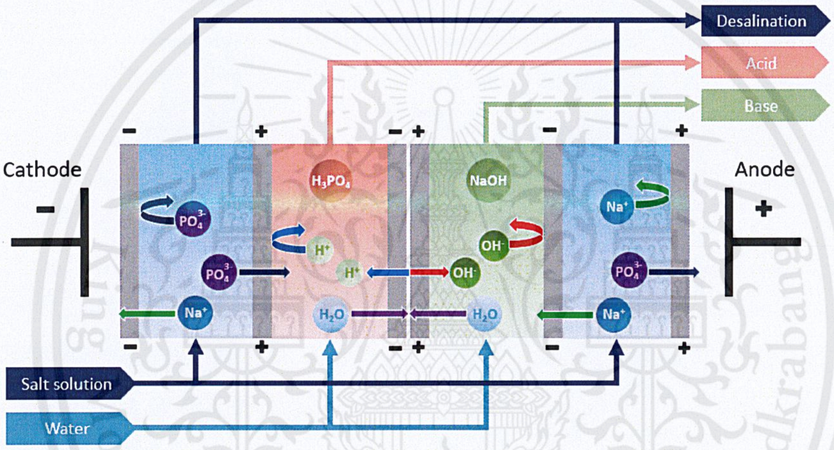

Figure 2.2 Production of acid and base with bipolar membranes.[7]

Figure 2.2 shows cation and anion exchange membranes and a bipolar membrane arranged in parallel between two electrodes to form individual compartments. If a salt solution is introduced between the anion and cation exchange membrane, cations in the salt solution will migrate towards the cathode, permeate the cation exchange membrane and form a base with hydroxyl ions that are formed within the bipolar membrane and are transported towards the anode. On the other side of the bipolar membrane, which is directed towards the cathode, the protons, which are formed simultaneously with the hydroxyl ions, form an acid with the anions migrating from the salt solution through the anion exchange membrane towards the anode. The net result of the process illustrated in figure 2.1 is the production of an acid and a base from the corresponding salt solution in an electrodialysis cell arrangement consisting of three individual compartments. As in conventional electrodialysis, the three compartment units can be stacked between a single pair of electrodes.[7,8]

Xiaolin Wang, et.al was study phosphate recovery from excess sludge by using integrated operation of electrodialysis (CED and BPED) with batch and continuous operation models in enhanced biological phosphorus removal (EBPR) process. The feed sodium phosphate solution was prepared by hydrate sodium phosphate crystal. The phosphate recovery was investigated through the factors consist of initial volume of solution rich in phosphate, operating voltage, feed flow rate, operating current density for BPED. The integrated electrodialysis operation are feasible to recover phosphate from feed solution to phosphoric acid form with energy consumption 29.3 kWh/(kg H_3PO_4).[9]

2.5 Ribonucleotide production process

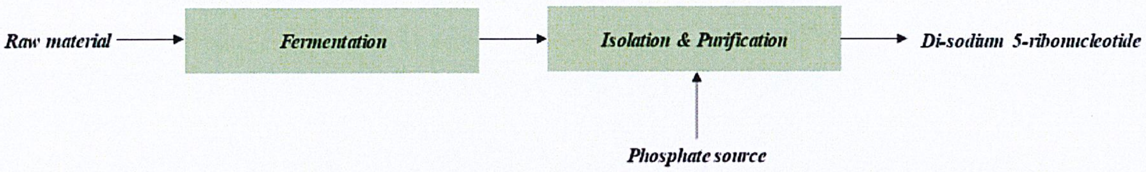

Figure 2.3. Simple block flow of ribonucleotide production process

The production process of ribonucleotide beginning with raw material preparation from polysaccharide as tapioca and molasses, digested and form monosaccharide, glucose is the raw material using in fermentation to produce nucleoside (Inosine, Guanosine and Adenosine) that without phosphate, after make the product from fermentation need to purified by crystallization. To produce ribonucleotide is adding phosphate group into nucleoside and purified the ribonucleotide product by crystallization, the product is in form of solid crystal that can packing.

The sodium phosphate salt in this study are the by-product from the process that occur at purification unit along the ribonucleotide production process, it will be used as the feed solution of BPED to recovery the phosphoric acid, low content sodium of sodium phosphate salt or sodium monophosphate dihydrogen and sodium hydroxide.

2.6 High Performance Liquid Chromatography (HPLC)

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry used to identify, quantify or purify the individual components of the mixture. The technique makes use a separation technique inside the column. The mixture moves through the column at varying velocities and interacts with the sorbent, also known as the stationary phase. The velocity of each component in the mixture depends on its chemical nature, the nature of the column and the composition of the mobile phase. The time at which a specific analyte emerges from the column is termed as its retention time. The retention time is measured under specific conditions and considered as the identifying characteristic of a given analyte. Sorbent particles might be hydrophobic or polar in nature. The commonly used mobile phases include any miscible combination of water and organic solvents. The chart displaying the time-dependent change in signal intensity as a result of the separation is called a chromatogram.[10,11]

2.7 Atomic Absorption Spectroscopy (AAS)

AAS is an analytical technique for measuring quantities of chemical concentrations of elements. The technique makes use of the wavelengths of light specifically absorbed by an element. They correspond to the energies needed to promote electrons from ground state to higher energy level. Atoms of different elements absorb characteristic wavelengths of light. The sample is atomized and a beam of electromagnetic radiation emitted from excited atoms is passed through the vaporized sample. The greater the number of interest atoms there is in the vapor, the more radiation is absorbed. The amount of light absorbed is proportional to the number of interest atoms. A detector measures the wavelengths of light transmitted by the sample and compares them to the wavelengths which originally passed through the sample. A signal processor then integrates the changes in wavelength absorbed, which appear in the readout as peaks of energy absorption at discrete wavelengths. The energy required for an electron to leave an atom is known as ionization energy and is specific to each chemical element. A calibration curve is constructed by running several samples of known concentration under the same conditions as the unknown. The amount the standard absorbs is compared with the calibration curve and this enables the calculation of the interest atom concentration in the unknown sample.[12]

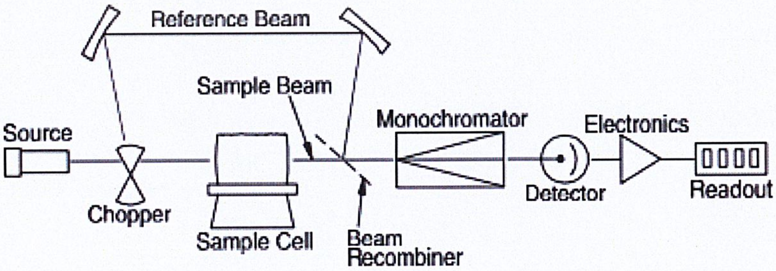

Figure 2.4 Double-beam Atomic Absorption Spectrometer.[12]

2.8 Conductivity

Conductivity is the ability of a material or solution to conduct electric current. The principle by which instruments measure conductivity in solution, two plates are placed in the sample with potentials is applied across the plates and the current that passes through the solution is measured. This parameter can use to analyze in term of electroanalytical method call conductometry.

The conductivity of chemicals solution is the specific property of solution. The conductivity of a solution depends on the number of charge carriers, the mobility of the charge carriers and their charge. Theoretically, conductivity should increase in direct proportion to concentration. In practice, this does not hold true. The concentration and mobility of the ions are not independent properties. As the concentration of an ion increases, its mobility decreases. As a consequence, the conductivity increases linearly with respect to the square root of concentration instead of in direct proportion as shown in Figure 2.5[13]

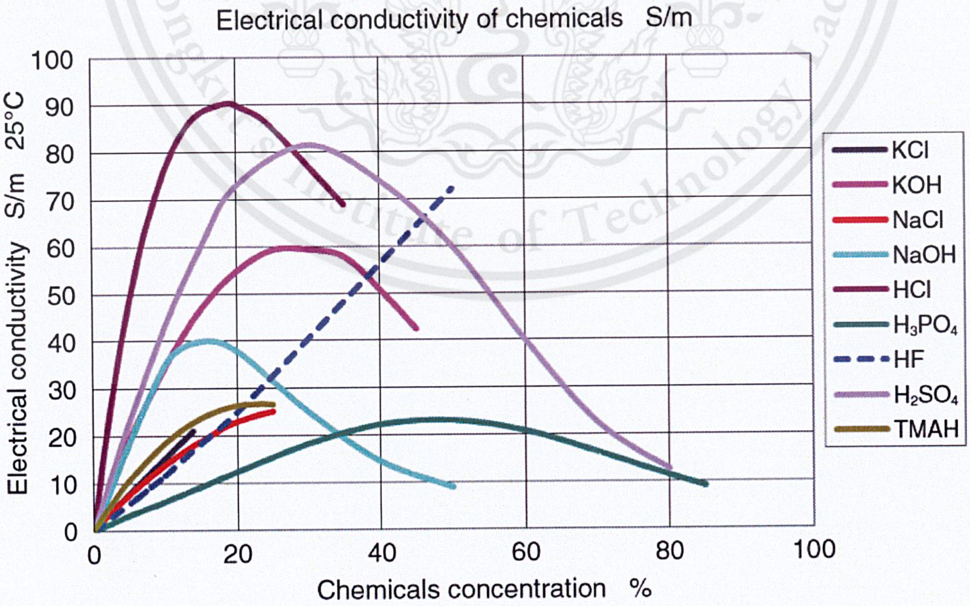

Figure 2.5 Graph show relation between electric conductivity and concentration of chemicals.[13]

The temperature is the state that have high effect with conductivity, high temperature will get the molecules more kinetic energy, the conductivity will be increasing due to mobility of an ion increases.[14]

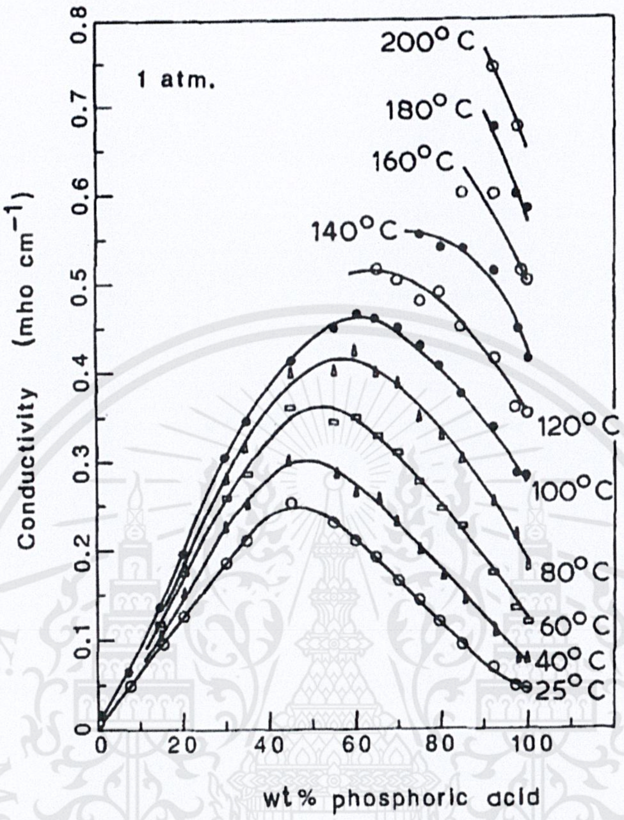

Figure 2.6 Specific conductivity vs concentration of phosphoric acid in the temperature range 25-200 ^{0}C.[14]

Der-Tau Chin and Haward H. Chang research the relation between concentration of phosphoric acid and electric conductivity at different temperature, in dilute phosphoric acid solution, the major contribution to electric conductivity is from the Stokesian ion transport mechanism, whereas in more concentrated phosphoric acid solutions, the major contribution to conductivity comes from the Grotthus proton switching conduction mechanism. The proton switching mechanism was initially proposed to explain the high mobility values of hydrogen and hydroxyl ions. In this mechanism, the electric conduction occurs by the hopping of protons from one solvated acid or water molecule to the other without actual transport of hydrogen ions in the solution.[14]

CHAPTER III

RESEARCH METHODOLOGIES

3.1 Chemicals and equipment

1) Sodium hydroxide
2) Phosphoric acid
3) TSP x'al
4) DSP x'al
5) Distilled water
6) Hydrochloric acid
7) Barium chloride
8) Pipette
9) Burette
10) Basket centrifugal separator
11) Filter cloth
12) Spray bottle
13) Vacuum pump
14) Buchner funnel
15) Buchner flask
16) Circular qualitative filter paper
17) Filter aid celatom
18) Agitator motor
19) Clamp
20) Volumetric flask
21) Beaker
22) Magnetic stirrer
23) Dropper
24) Moisture meter
25) Halogen moisture analyzer glass fiber disc
26) Stainless steel moisture pan
27) Atomic absorption spectroscopy (AAS)
28) Falcon tube
29) Milli-Q water
30) Circular qualitative filter paper
31) High pressure liquid chromatography (HPCL)
32) Syringe
33) Syringe filter
34) Vial
35) Laboratory weight scale
36) Peristaltic pump

37) Heater

38) Propeller

39) Micro filter membrane

40) Bipolar membrane electrodialysis tool set (BPED)

3.1.1 Bipolar membrane electrodialysis tool set

1 Components list

Table 3.1 BPED component list.

(1)	Electrodialyzer	1st stage	(38)	1st stage Pressure guage	salt
(2)		2nd stage	(39)		acid
(3)		3rd stage	(40)		base
(4)	Electrode connector (Cathode)		(41)		electrode
(5)			(42)	2nd stage Pressure guage	salt
(6)			(43)		acid
(7)	Stack Voltage tap		(44)		base
(8)			(45)		electrode
(9)			(46)	3rd stage Pressure guage	salt
(10)	1st stage pump	salt	(47)		acid
(11)		acid	(48)		base
(12)		base	(49)		electrode
(13)	2nd stage pump	salt	(50)	Blower	
(14)		acid	(51)	1st stage tank	salt
(15)		base	(52)		acid
(16)	3rd stage pump	salt	(53)		base
(17)		acid	(54)	2nd stage tank	salt
(18)		base	(55)		acid
(19)	pump; electrode rinse solution		(56)		base
(20)	1st stage Flow Control valve	salt	(57)	3rd stage tank	salt
(21)		acid	(58)		acid
(22)		base	(59)		base
(23)		electrode	(60)	tank; electrode rinse solution	
(24)	2nd stage Flow Control valve	salt	(61)	Exhaust gas outlet line	
(25)		acid	(62)	Feed solution pump	Feed
(26)		base	(63)		Pure water
(27)		electrode	(64)		Pure water
(28)	3rd stage Flow Control valve	salt	(65)	1st stage drain valve	salt
(29)		acid	(66)		acid
(30)		base	(67)		base
(31)		electrode	(68)	2nd stage drain valve	salt
(32)	1st stage Flow rate	salt	(69)		acid
(33)		electrode	(70)		base
(34)	2nd stage Flow rate	salt	(71)	3rd stage drain valve	salt
(35)		electrode	(72)		acid
(36)	3rd stage Flow rate	salt	(73)		base
(37)		electrode	(74)	drain valve; electrode rinse solution	
(75)	Emergency stop		(76)	Conductivity meter	salt
			(77)		acid
			(78)		base

2 Bipolar membrane electrodialysis operation procedure

1) Setting place

Set the electrodialyzer at ventilated place.

2) Preparation

2.1) Check the tanks, piping and valves

2.1.1) Check the tank and the piping for clean condition. If there are soiled with dust or dirt, they prevent normal operation such as will clogging up of the electrodialyzer.

2.1.2) Use this unit in ventilated room, a small amount of mixture of oxygen and hydrogen gases generated at electrode compartment of ED will blow off.

2.1.3) Ascertain that the drain valves are closed.

2.1.4) Slightly open the Flow Control valves.

2.2) Preparation of solutions

2.2.1) Prepare salt solution, acid solution, base solution and electrode rinse solution into S, A, B and E tank respectively.

Table 3.2 Recommended standard solution.

Salt solution (1st, 2nd)	3.0 liters / each tank	Feed solution
Acid solution (1st, 2nd)	1.5 liters / each tank	2nd stage ; 2.0eq/L-HCl 3rd stage ; 1.0eq/L-HCl
Base solution (1st, 2nd)	1.5 liters / each tank	2nd stage ; 2.5eq/L-NaOH 3rd stage ; 1.2eq/L-NaOH
Electrode rinse solution	15.0 liters	1.0eq/L-NaOH

2.2.2) Supply the cooling water to heat exchanger line, only salt solution and electrode rinse solution are applied, heating will be exchanged through the membranes in the electrodialyzer.

3) Operation

3.1) Set 5 parameters consist of Salt conductivity, Acid conductivity, Base conductivity, 2nd stack voltage and 3rd stack voltage (The recommended stack voltage value is 25V or less per 10 cells).

3.2) Set the operation mode of REC-3 to “SLIDE” mode

3.3) Turn on, all solutions will be fed automatically. After all solutions are fed, all circulation pumps will start. And all solutions will be fed again by follow PLC system.

4) Adjustment of flow rate

Table 3.3 Adjust the flow rate of each solution to specified flow rate.

Salt solution	2.5 L/min
Acid solution	Set the same circulation pressure as Salt solution
Base solution	Set the same circulation pressure as Salt solution
Electrode solution	Set the same circulation pressure as Salt solution

3 Programmable Logic Controller for bipolar membrane electrodialysis operation

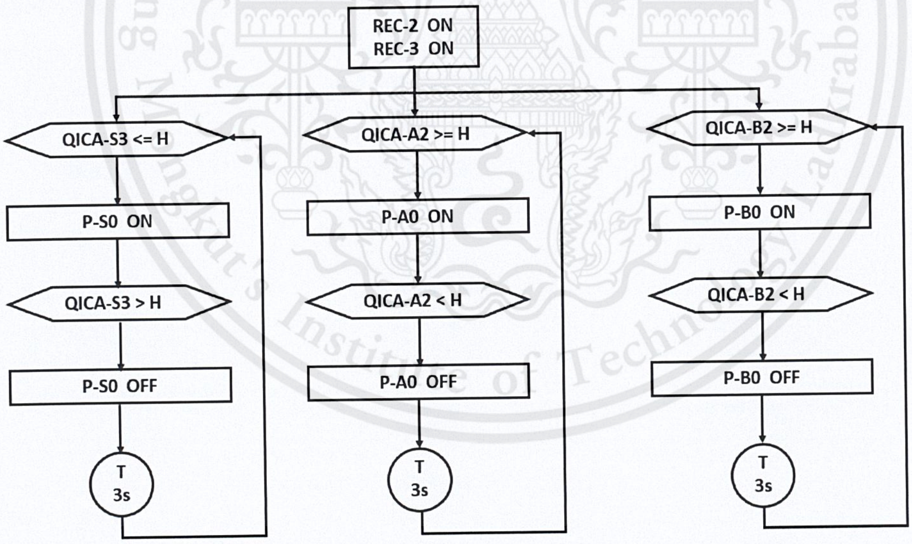

Figure 3.1 Programmable Logic Controller for bipolar membrane electrodialysis operation.

When QICA-S3 is salt conductivity of 3rd stage of salt tank

QICA-A2 is acid conductivity of 2nd stage of acid tank

QICA-B2 is base conductivity of 2nd stage of base tank

P-S0 is feed pump

P-A0, P-B0 are diluted pumps

From figure 3.1 shows mainly of operating sequence of BPED after key conductivity set point of 3 chemicals consist of salt, acid and base and stack voltage of REC-2 and REC-3. For the 3rd stage of salt tank during operation the conductivity of salt solution will be decrease cause of salt concentration is used for generate acid and base until less than set point (H) of QICA-S3, P-S0 will be on to feed salt solution that have higher concentration than 3rd stage of salt tank to increase QICA-S3 cause of concentration of salt inside the 3rd stage of salt tank is increased until QICA-S3 more than H, P-S0 will be stop to fill high concentration salt solution. For the products, acid and base. The conductivity of the product solution will be increase during operation cause of the concentration that is generated by salt solution until QICA-A2 or QICA-B2 more than H, P-A0 or P-B0 will be on to fill process water for decrease the conductivity of the products by dilution until QICA-A2 or QICA-B2 less than H, P-A0 or P-B0 will be off. This operation will follow this PLC sequence chart for automatically operation.

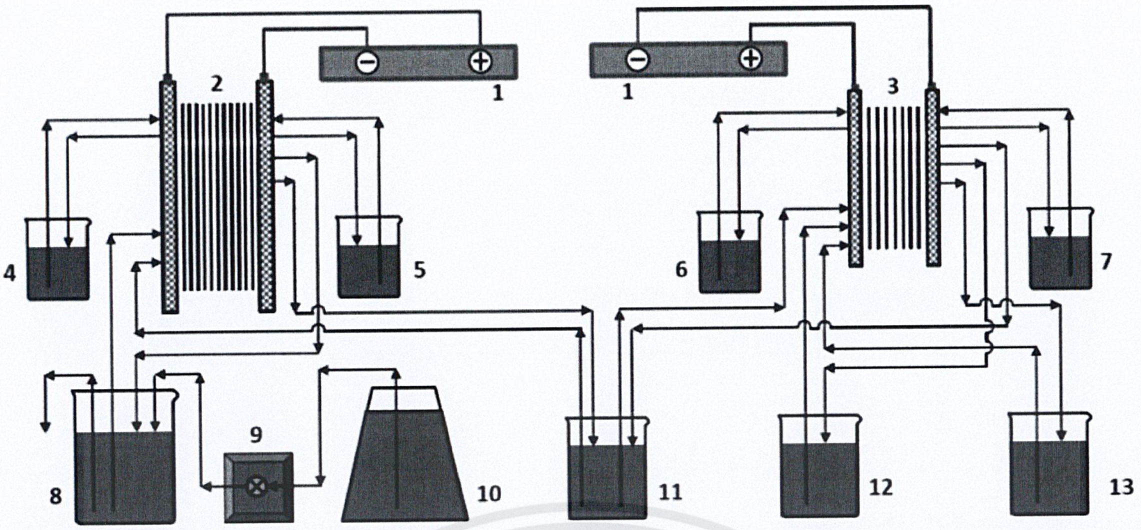

Figure 3.2 Flow chart of the BPED. (1) Direct current power supply. (2) BPED 2nd stack. (3) BPED 3rd stack. (4,5,6,7) Electrode rinse solution tank. (8) Diluted solution tank. (9) Peristaltic pump. (10) Feed tank. (11) Buffer tank (concentrated solution tank for BPED). (12) acid tank. (13) alkali tank.[9]

3.1.2 Raw materials

1 DSP x’al

DSP is the inorganic compound in crystal form. DSP was generated during phosphorylation process and separated after purification process. The major component of DSP is disodium phosphate with the formula Na_2HPO_4. Nowadays, Ajinomoto (Thailand) company limited uses for recovery phosphate source raw material, sodium tripolyphosphate (STPP) in STPP recovery process with H_3PO_4.

Table 3.4 Component and important physical properties of DSP x’al.

Component	$Na_2HPO_4 \cdot 12H_2O$ 80.6 wt% $Na_4P_2O_7 \cdot 10H_2O$ 11.0 wt%
Solubility in water (25^0C)	Approx. 12 g/ 100 mL
pH (at saturated point)	Aprrox. 10.0

2 TSP x'al

TSP is one of sodium phosphates compound as DSP, TSP was generated during phosphorylation process and separated after purification process before DSP. The major component of TSP is trisodium phosphate and tetrasodium pyrophosphate with the formula Na_3PO_4 and $Na_4P_2O_7$, respectively. Currently, TSP uses as pH adjustment in purification process to increase pH of sodium phosphates solution.

Table 3.5 Component and important physical properties of TSP x'al.

Component	$Na_3PO_4 \cdot 12H_2O$ 63.5 wt% $Na_4P_2O_7 \cdot 10H_2O$ 20.5 wt%
Solubility in water (25^{0}C)	Approx. 13 g/ 100 mL
pH (at saturated point)	Aprrox. 12.3

3.2 Procedure

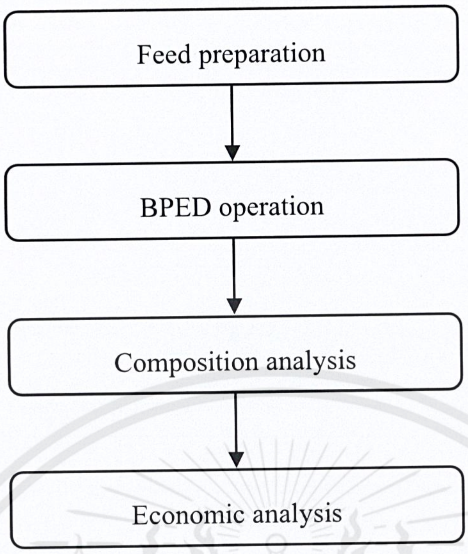

Figure 3.3 Flow chart of experiment procedure.

3.2.1 Feed preparation

1) Loading DSP x'al or TSP x'al from the production process.

2) Scrub TSP x'al by distilled water at 10^0C in basket centrifugal separator with filter cloth (amount of distilled water is 1:1 ratio of TSP x'al mass per distilled water mass) to eliminate polyphosphate group.

3) Rest TSP x'al for 1 day for dehumidification.

4) Check moisture content of TSP x'al (after wash) or DSP x'al by moisture meter with stainless steel moisture pan. Record moisture content of crystals.

5) Dissolve TSP x'al or DSP x'al with process water at saturated point of their solubility (at saturated point of both solution have pH specifically, for this experiment was fix pH of feed solution at 12.30 and 10.00 for TSP solution and DSP solution, respectively).

6) Filtrate TSP or DSP solution in micro membrane filter to prevent plugging in BPED and eliminate remained activated carbon inside TSP or DSP solution from production process.

7) Fill TSP or DSP solution in feed tank for BPED operation.

3.2.2 BPED operation

BPED operation required mainly 5 parameters (QICA-S3, QICA-A2, QICA-B2, 2nd stack voltage and 3rd stack voltage) for run PLC system. For this study was operating with 2 different raw materials consist of TSP x'al and DSP x'al for BPED feed with variance operating conditions and control method.

The objective of the first experiment will study the parameters effect on BPED performance by considering electric current on both membrane stack consist of QICA-S3, QICA-A2, QICA-B2, operating pH and operating temperature in salt solution tank and the feasibility of production in each case by vary single variable to investigate the effect of each parameters.

The objective of the second experiment to calculate electric consumption by estimate the conditions from the first experiment to find the optimum condition.

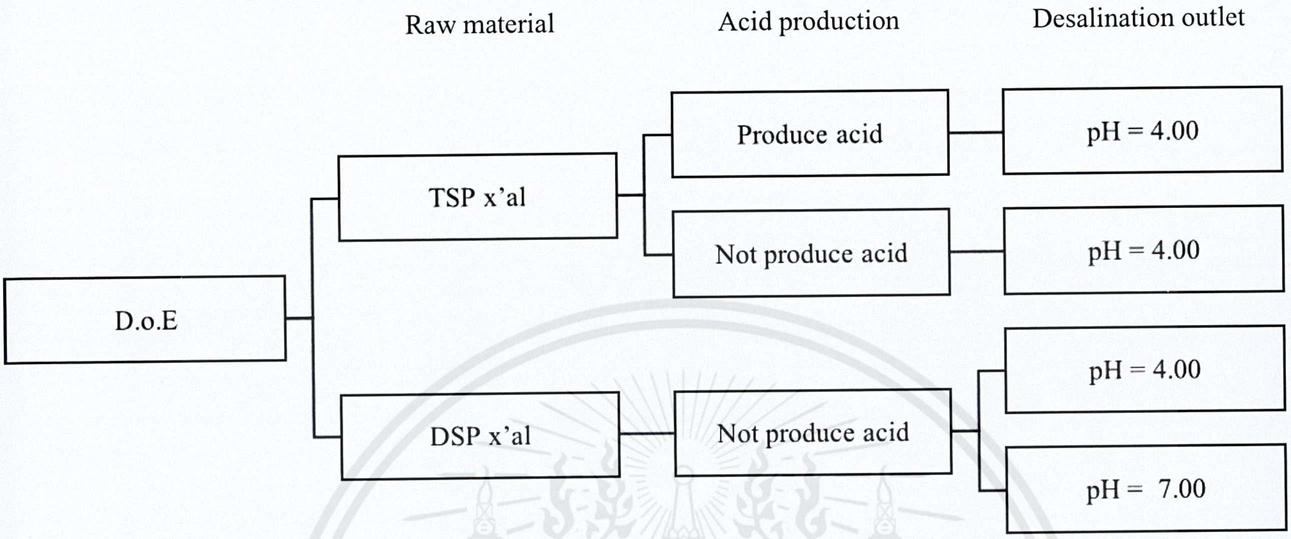

Figure 3.4 Design of experiment of BPED operating conditions.

From figure 3.7 shows the conditions for 4 experiments consist of the first condition, feed TSP solution to BPED and operate phosphoric acid and desalination at pH of 4 and other conditions was eliminated phosphoric acid stream outlet, the last condition produces desalination at pH of 7. These 4 conditions were operating to study the electric consumption on production rate.

3.3 Analysis

Material composition analysis

1) Phosphate group analysis of raw material, desalt product and phosphoric acid

    1.1) Dilute the sample solution with Milli-Q water in range of calibration

    1.2) Analyze the phosphate group of sample solution with High Performance Liquid Chromatography (HPCL)

2) Sodium content analysis of raw material and desalt product

    2.1) Dilute the sample solution with Milli-Q water in range of calibration

    2.2) Analyze the sodium content of sample with Atomic Absorption Spectroscopy (AAS)

3) Sodium hydroxide concentration analysis of base product

    Analyze the concentration of sodium hydroxide with titration method

4) Total solid analysis of sample solution

    Analyze the moisture content of sample solution with moisture meter

CHAPTER IV

RESULTS AND DISCUSSIONS

4.1 Performance of BPED operation with different condition

4.1.1 Phosphoric acid conductivity (QICA-A2) effect

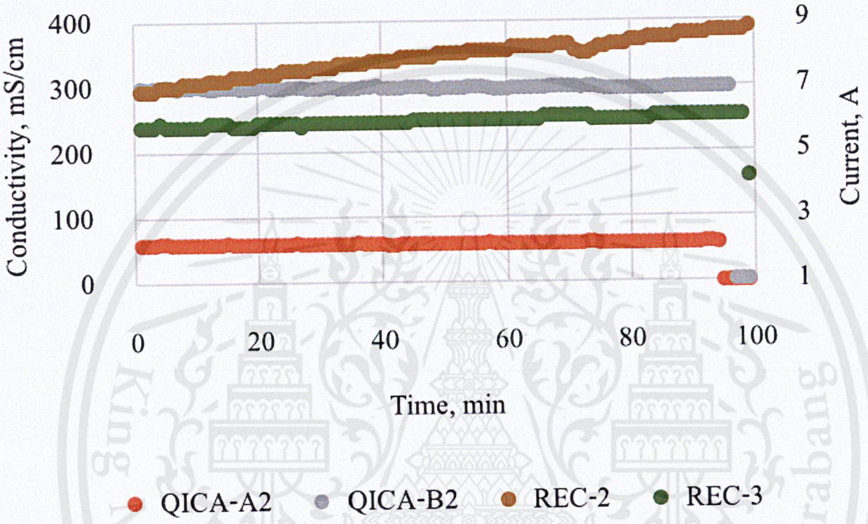

Figure 4.1 Graph show relation between phosphoric acid conductivity (orange) at QICA-A2 of 60.0 mS/cm with electric current on 2nd stack (brown) and 3rd stack (green).

From figure 4.1 show the BPED operating condition, QICA-A2 and QICA-B2 were set at 60.0 and 300.0 mS/cm to monitoring electric current REC-2 and REC-3 real-time. For REC-3 (low concentration stack) have overall conductivity of chemicals inside membrane stack lower than REC-2 (high concentration stack), so the total resistance between electrode plates of REC-2 is lower than REC-3. The average of REC-2 change between 80 min to 10 min is around 0.05 A/min, while the average of REC-3 change at the same interval of time is almost constant. The electric current can raise beyond overall conductivity of REC-3 in REC-2 condition and reach 9 A when operate for 100 min.

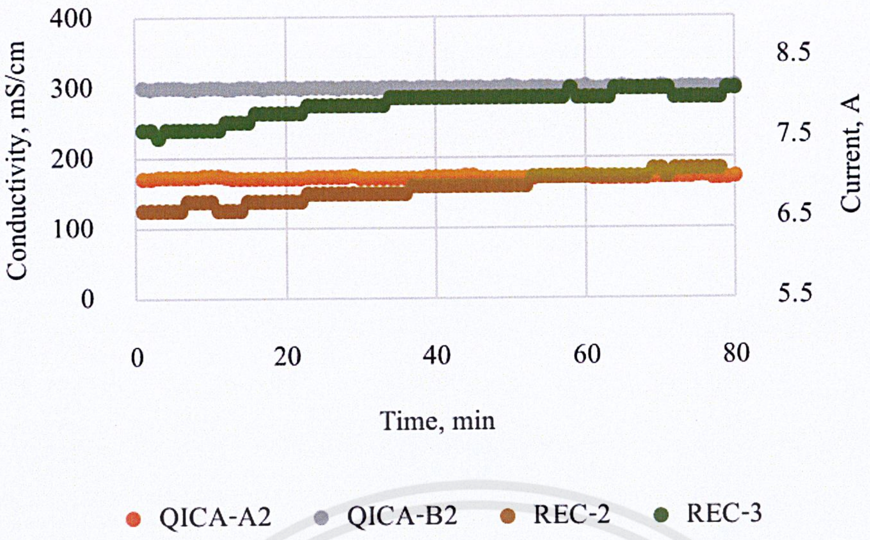

Figure 4.2 Graph show relation between phosphoric acid conductivity (orange) at QICA-A2 of 173.0 mS/cm with electric current on 2nd stack (brown) and 3rd stack (green).

From figure 4.2, the operating condition is 173.0 and 300.0 mS/cm for QICA-A2 and QICA-B2, respectively. The electric current of both of stacks are almost constant after BPED operate for 50 min. REC-3 is higher than REC-2 while overall conductivity of chemicals inside 2nd stage is higher than 3rd stage. This phenomenon occurs cause of the compartment that adjacent with bipolar membrane have rich electric charges and form ion film layer that resulting in greater resistance between electrode plates, this phenomenon called concentration polarization or shock electrodialysis. The condition of REC-3 (low concentration stack) is suitable than REC-2 (high concentration stack) to get high electric current but lower yield.

4.1.2 Sodium hydroxide conductivity (QICA-B2) effect

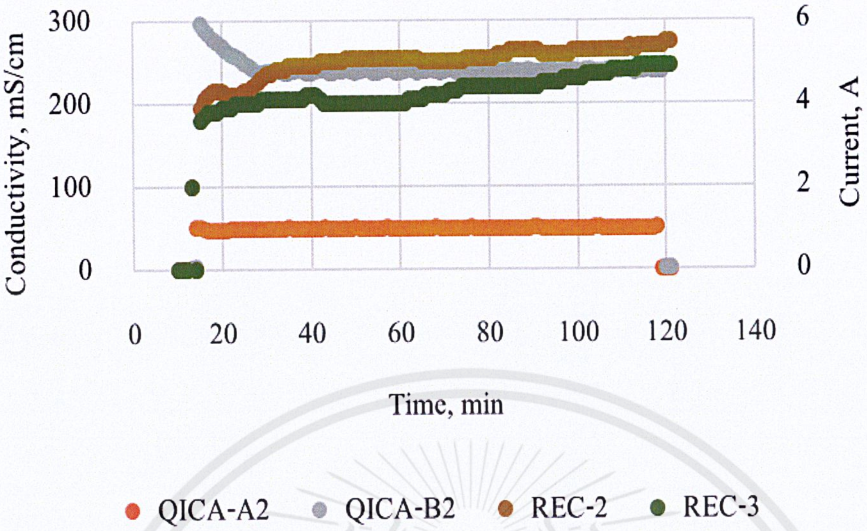

Figure 4.3 Graph show relation between phosphoric acid conductivity (orange) at QICA-B2 of 240.0 mS/cm with electric current on 2nd stack (brown) and 3rd stack (green).

From figure 4.3, the operating condition is QICA-A2 set at 50.0 mS/cm, while QICA-B2 are start from 300.0 mS/cm and decrease to 240.0 mS/cm. In 15 min to 30 min interval, the electric current increase rapidly at 0.07 A/min for REC-2 and 0.03 A/min and decrease after QICA-B2 is constant at 240.0 mS/cm. This show the limiting of base conductivity at QICA-A2 is 50.0 mS/cm, if set QICA-B2 more than 300.0 mS/cm while QICA-A2 is 50.0 mS/cm or less than the electric current will be drop down cause of concentration polarization. After the first period the current is raise up for both stage cause of rate of ion transfer is increasing compare with first period (high QICA-B2).

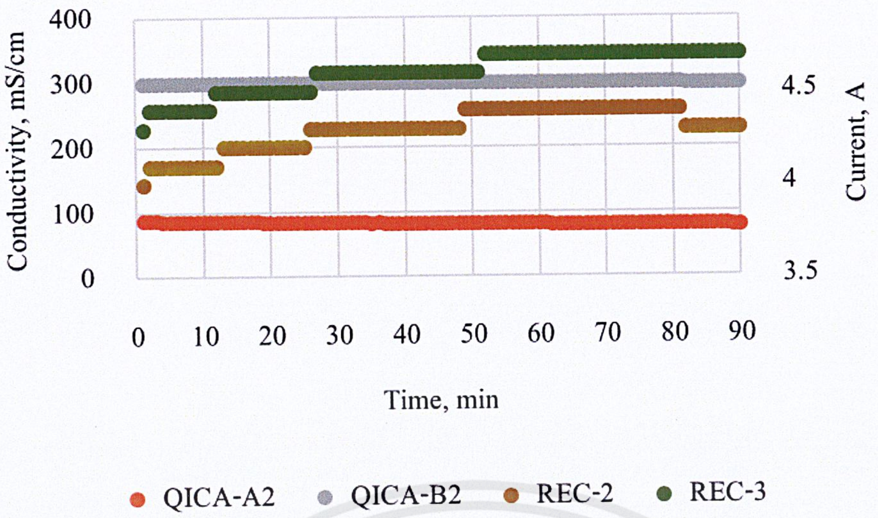

Figure 4.4 Graph show relation between phosphoric acid conductivity (orange) at QICA-B2 of 300.0 mS/cm with electric current on 2nd stack (brown) and 3rd stack (green).

From figure 4.4, the operating condition is QICA-B2 set at 300.0 mS/cm without QICA-A2 control (H of QICA-A2 set higher than 200.0 mS/cm, P-B0 off). The QICA-A2 is decrease cause of rate of water recombine and generation inside acid compartment higher than phosphoric formation rate. This system has more stability (almost constant of electric current) but use highly electric consumption to operate with shock electrodialysis and high rate of water formation.

4.1.3 Sodium phosphate salt solution conductivity (QICA-S3) effect

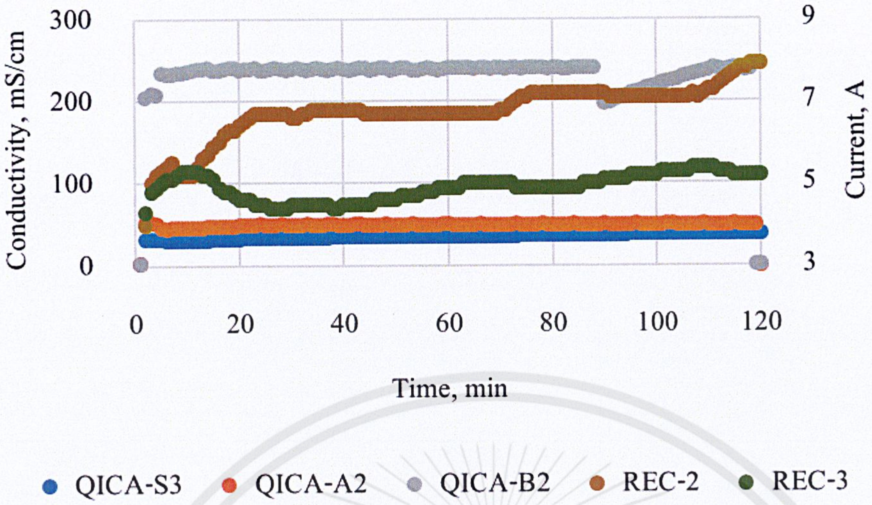

Figure 4.5 Graph show relation between phosphoric acid conductivity (orange) at QICA-S3 of 34.0 mS/cm with electric current on 2nd stack (brown) and 3rd stack (green).

From figure 4.5, the operating condition is QICA-S3 set at 34.0 mS/cm, QICA-A2 set at 50.0 mS/cm and QICA-B2 set at 240.0 mS/cm. The electric current of both stages have highly fluctuation, the maximum current around 7.0 A and 5.2 A for REC-2 and REC-3, respectively. After disturbed the system by pulse the QICA-B2 by adjust the sodium hydroxide concentration the electric current is raise up rapidly and reach 8.0 A for REC-2.

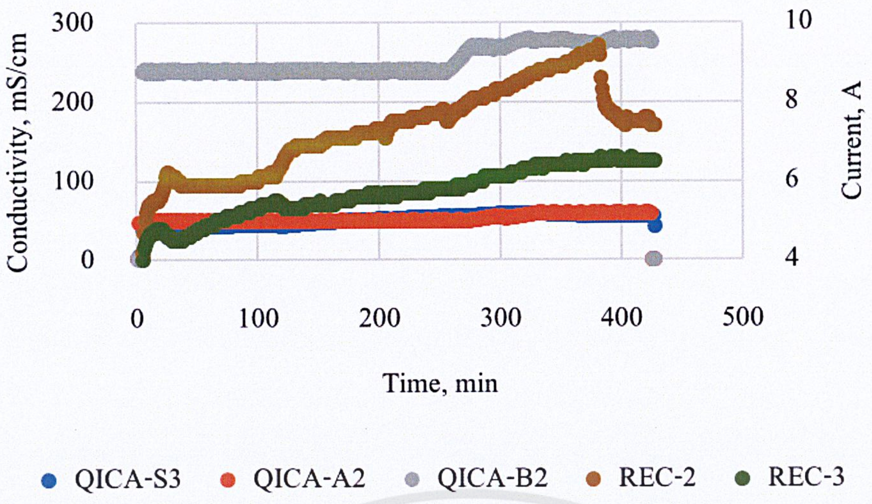

Figure 4.6 Graph show relation between phosphoric acid conductivity (orange) at QICA-S3 of 55.0 mS/cm with electric current on 2nd stack (brown) and 3rd stack (green).

From figure 4.6, the operating condition is the same with figure 4.5 except QICA-S3 is set at 55.0 mS/cm. The electric current is rapidly increasing, at 200 min to 300 min, the electric current rate of change around 0.01 A/min at 7.3 A to 8.4 A for REC-2 after set QICA-B2 to 280.0 mS/cm, the concentration polarization is occur, resulting in REC-2 drop to 7.5 A while REC-3 almost stable at final period.

4.1.4 pH effect

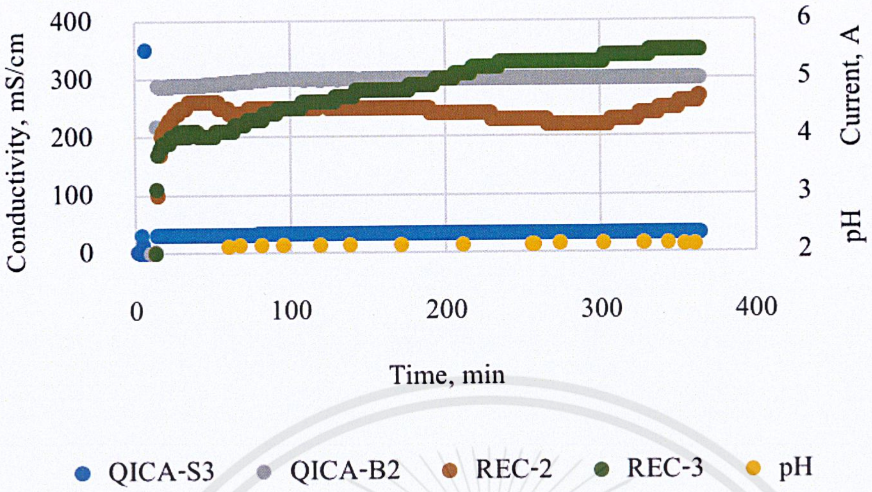

Figure 4.7 Graph show relation between pH (yellow) at pH of 2 with electric current on 2nd stack (brown) and 3rd stack (green).

From figure 4.7, the control system for study pH effect on electric current was change from feed control by P-S0 to P-S4 and P-S5 (no required QICA-S3) while QICA-B2 is 300.0 mS/cm and QICA-A2 is set over 200.0 mS/cm to eliminate acid stream outlet. Because of the composition of electrolyte of triprotic system depends on pH (rate of protonation). This condition is fixed pH at salt solution tank at 2 to maintain ratio of sodium and phosphate at 1 per 2, respectively.

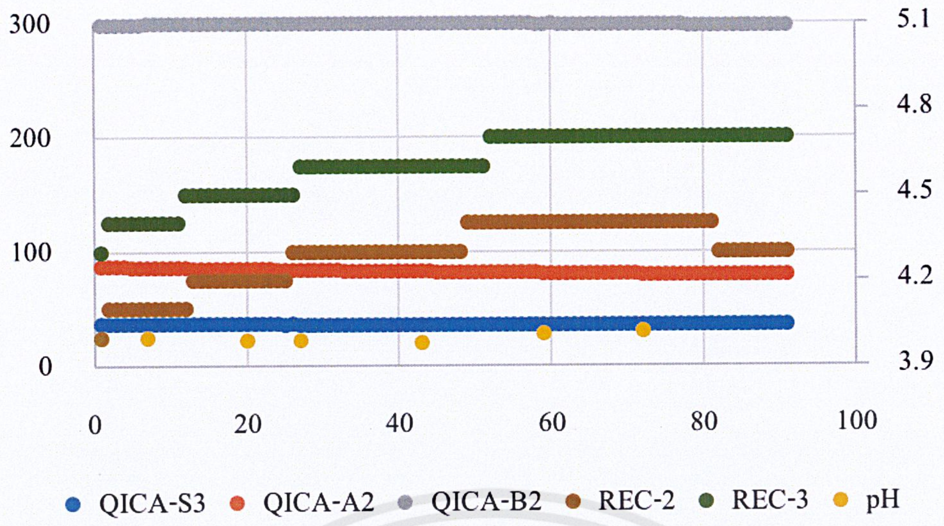

Figure 4.8 Graph show relation between pH (yellow) at pH of 4 with electric current on 2nd stack (brown) and 3rd stack (green).

From figure 4.8, the control system is used as same as condition figure 4.7 at operating pH of 4. For this pH, the almost all of sodium phosphate salt is form at monosodium phosphate inside desalination compartment, when compare with condition that was operating at pH 2, the electric current is lower cause of the rate of dissociation of monosodium phosphate is less than phosphoric acid. The overall chemical conductivity is similar but rate of products formation are lower than lower pH condition.

4.1.5 Temperature effect

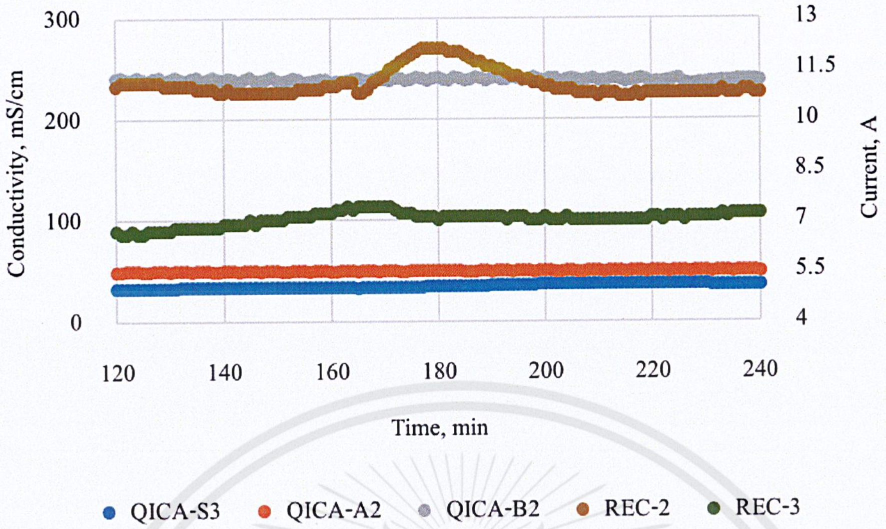

Figure 4.9 Graph show effect of temperature of salt solution is control at 39.0°C at 170 min.

From figure 4.9, the normal operating temperature is around 31.0°C. After 165 min, the cooling water is turn off to heating system until 39.0°C and cool it down at 175 min to avoid damage with membrane. The overall of conductivity system is increase, resulting in resistance between electrode plates is decrease.

The results from performance of BPED operating with different condition. The conductivity of chemicals related with concentration of the chemicals, for the electric conductivity property of the chemicals, the electric conductivity is proportional with concentration at the first period and decreasing.

To get products have high conductivity that refer to high concentration that mean to get high electric resistance to lower electric resistance, respectively. The current will be increasing with conductivity at initial (for low concentration or almost water) and rapid decreasing after conductivity increase because the chemicals that used are dissociate easier compare with water.

For this production is use the conductivity of chemicals for more than 5 wt% of chemicals concentration so the relation between conductivity of products and current are disproportional.

The desalination pH effect was test for below pH of 7 and approximately 0. 8 at least. The condition that get high current is the low pH condition because of the sodium content in phosphate group. At low pH the sodium content in salt get lower and form sodium hydroxide and phosphoric acid to get acid and alkaline compartment have higher concentration or lower resistance.

The desalination temperature effect is related directly with conductivity of chemicals, at high temperature, electric conductivity will be high and get the rate of acid, base and salt dissociation dominant than water recombination.

4.2 Product with electric consumption

Table 4.1 Properties of feed and product with electric consumption to produce.

Condition*	Feed		Desalt		Electric consumption (kWh/kg feed)
	Concentration (wt%)	pH	Concentration (wt%)	pH	
1	9.81	12.30	7.24	4.00	0.1729
2	10.17	12.30	7.95	4.00	0.1406
3	8.36	10.00	7.24	4.00	0.2072
4	11.33	10.00	11.21	7.00	0.0586

* Condition 1	acid product of 20.86 wt%, base product of 8.07 wt%, membrane arrangement: BAC
Condition 2	no acid product, base product of 8.07 wt%, membrane arrangement: BAC
Condition 3	no acid product, base product of 8.07 wt%, membrane arrangement: BC
Condition 3	no acid and base product, membrane arrangement: BC

The results from data analysis of BPED performance, the condition number 1 use the highest electric consumption for feed pH of 12.30 that have high sodium and phosphate ratio as a raw material to purified phosphoric acid, the condition number 2 is produce similar condition with condition number 1 except no acid product, this condition use low electric consumption than condition number 1, the condition number 3 and 4 are change the pattern of membrane arrangement to BC pattern and blind the acid compartment to produce just monosodium phosphate, the condition number 3 use the highest electric consumption because the efficiency of membrane getting low due to swelling and fouling, after recovery the membrane, the condition number 4 get the lowest electric consumption to produce monosodium dihydrogen phosphate at pH 7.

4.3 Sodium reduction

Table 4.2 Sodium content before and after pass BPED.

Condition*	**Feed**		**Desalt**		**Residue sodium content in desalt (%)**
	pH	**Na (wt%)**	**pH**	**Na (wt%)**	
1	**12.30**	**3.93**	**4.00**	**1.48**	**30.61**
2	**12.30**	**3.67**	**4.00**	**1.82**	**42.54**
3	**10.00**	**3.74**	**4.00**	**2.41**	**45.70**
4	**10.00**	**3.77**	**7.00**	**3.32**	**75.04**

* Condition 1 acid product of 20.86 wt%, base product of 8.07 wt%, membrane arrangement: BAC

Condition 2 no acid product, base product of 8.07 wt%, membrane arrangement: BAC

Condition 3 no acid product, base product of 8.07 wt%, membrane arrangement: BC

Condition 4 no acid and base product, membrane arrangement: BC

The results show the reduction of sodium content compare with sodium content in feed, the pH is proportional with sodium and phosphate ratio, the condition number 1 that have acid and base product can reduce sodium more than another condition and condition number 2 that acid compartment still installed can reduce more than condition that does not connect with acid compartment, that mean, the sodium ion can migrate pass through the anion exchange membrane.

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The objective of this project is to study the feasibility of production of phosphoric acid and monosodium phosphate from sodium phosphate salts (TSP x'al and DSP x'al) by using bipolar membrane electrodialysis technology. Can be summarized as follows:

5.1.1 The phosphoric acid can produce by use sodium phosphate salts (TSP x'al and DSP x'al) using bipolar membrane electrodialysis technology.

5.1.2 The main parameters that affect with electric consumption are electric conductivity of chemicals (salt, acid and base), pH of desalination and operating temperature.

Table 5.1 The effect of parameters on electric consumption.

Parameter	Too low	Too high	Optimum
QICA-A2	High resistance	Concentration Polarization	50 mS/cm
QICA-B2	High resistance		240 mS/cm
QICA-S3	High resistance	Low resistance	70 mS/cm
Temperature	High resistance	Low resistance	40 ^{0}C
pH	High dissociation rate	Low dissociation rate	1-2

5.1.3 Sodium content was reduced to 30-75 wt% of feed inlet stream.

5.2 Recommendations

5.2.1 This production can produce by using electrodialysis without bipolar membrane.

5.2.2 The fouling is form at anion layer on bipolar membrane for this system. The assumption if from piping system corrosive from alkaline corrosion and form a glue film layer with sodium phosphate stain on anion layer.

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APPENDIX A

Conductivity curve data

Table A.1 Phosphoric acid conductivity.

#	H3PO4 85%wt g	H3PO4 conc. %wt	Conductivity mS/cm
1	0.00	0.00%	0.002
2	2.52	2.09%	15.77
3	5.12	4.14%	26.80
4	7.77	6.13%	37.50
5	10.59	8.14%	48.20
6	13.51	10.12%	58.90
7	16.66	12.14%	70.00
8	21.70	15.16%	87.10
9	31.07	20.15%	115.90
10		21.08%	127.30
11	54.90	30.13%	176.20
12	89.13	40.06%	213.00
13		42.14%	218.00
14		85.00%	94.00

Table A.2 Sodium hydroxide conductivity.

#	NaOH conc. %wt	Conductivity mS/cm
1	0.0001%	0.0062
2	0.0003%	0.0184
3	0.001%	0.0611
4	0.003%	0.182
5	0.01%	0.603
6	0.03%	1.78
7	0.10%	5.82
8	0.30%	16.2
9	1.00%	53.2
10	3.00%	144
11	5.00%	223
12	10.00%	358
13	20.00%	414
14	30.00%	292
15	40.00%	191
16	50.00%	150

Table A.3 TSP solution conductivity.

#	TSP x'al g	TSP conc. %wt	Conductivity mS/cm	Temperature °C	TSP conc. %wt. of TS
1	0.00	0.00%	0.0018	26.7	0.00%
2	3.65	1.20%	8.88	25.8	0.67%
3	6.13	2.00%	12.93	25.5	1.11%
4	9.27	3.00%	17.36	25.2	1.66%
5	12.51	4.00%	21.40	24.9	2.22%
6	26.09	8.00%	34.7	23.9	4.44%
7	40.94	12.01%	45.3	25.4	6.66%
8	57.17	16.01%	53.6	24.6	8.88%
9	75.57	20.12%	60.5	24.0	11.16%
10	94.74	24.00%	65.2	25.7	13.31%
11	94.75	24.00%	66.5	26.6	13.31%
12	753.89	23%	67.5	27.7	14.16%
13	1034.12	23%	67.8	27.9	14.16%
14			84.3	29.3	15.96%
15			86.6	29.6	16.50%

Table A.4 Phosphoric acid consumption.

pH	consumption (g.H3PO4/g.TSPx'al)	consumption (g.H3PO4/g.TS)	H3PO4
12.31	0.00	0	0
9.97	0.03	0.2	1.13 g
8.00	0.03	0.3	1.43 g
6.00	0.08	0.6	3.36 g
3.93	0.15	1.1	6.10 g

APPENDIX B

Table B.1 Component of raw material.

Condition	Feed (wt%)				
	R/M	Na	P1 (as PO4)	P2 (as PO4)	MC
1	TSP x'al	3.93%	3.73%	1.97%	90.19%
2	TSP x'al	3.67%	5.87%	0.00%	89.83%
3	TSP x'al	3.67%	5.90%	0.00%	89.83%
4	TSP x'al	4.57%	4.61%	2.03%	88.62%
5	DSP x’al	3.01%	5.47%	0.43%	89.83%
6	DSP x’al	3.01%	5.47%	0.43%	91.64%
7	DSP x’al	3.74%	1.38%	1.38%	-
8	DSP x’al	3.77%	6.59%	0.81%	-

Table B.2 Feed component.

Sample	condition	Na		P1 as PO4	P2 as PO4	Moisture content percentage		
		ppm	(%)			%MC		
TSP x'al	existing	0.0527	12.10%	21.79%	4.80%	34.00%	36.99%	36.24%
	washing 100%	0.1837	16.79%	21.95%	4.21%			
	washing 150%	0.1799	16.70%	22.70%	3.19%			

Table B.3 Desalt component.

Condition	Base (wt%)
	NaOH
1	8.07%
2	8.86%
3	8.86%
4	9.26%
5	9.26%
6	9.26%
7	7.96%
8	8.07%

Table B.4 Sodium hydroxide titration analysis.

Condition	Desalination component (wt%)			
	Na	P1 (as PO4)	P2 (as PO4)	MC
1	1.48%	4.00%	2.05%	92.76%
2	1.40%	5.99%	0.00%	93.41%
3	1.82%	7.02%	0.00%	92.05%
4	1.30%	7.15%	2.04%	89.16%
5	2.09%	6.45%	1.74%	90.29%
6	1.50%	5.80%	1.26%	-
7	2.41%	8.92%	1.14%	-
8	3.32%	8.57%	0.55%	-

Table B.5 Phosphoric acid analysis.

Condition	Acid (%wt)	
	P1 (as PO4)	MC
1	20.22%	79.70%
2	-	-
3	-	-
4	-	-
5	-	-
6	-	-
7	-	-
8	-	-

APPENDIX C

Table C.1 Water quality required for electrodialyzer operation.

Temperature	15-40^{o}C
pH	5-8
Turbidity	1 ppm or less
BOD	5 ppm or less
COD	5 ppm or less
SiO_2	5 ppm or less
Multivalent Cations	10 ppb or less
HCO3	20 ppm or less
Oxidizing	Not to be detected.
Surface active agent	Not to be detected.

Table C.2 Specification table.

Code	Name	Specification
BP-2 **BP-3**	**Bipolar Membrane Electrodialyzer**	**Material PVC (electrode chamber)** **Membrane area 2dm^2/sheet** **Number of membrane** **Cation exchange membrane; 10 sheets** **Anion exchange membrane; 10 sheets** **Bipolar exchange membrane; 10 sheets** **Electrode diaphragm; 2 sheets** **Anode Nickel plate** **Cathode Nickel plate**
T-S2 **T-S3**	**Salt solution tank**	**Vertical square tank** **Capacity ; 3.0L, Material PVC**
T-A2 **T-A3**	**Acid solution tank**	**Vertical square tank** **Capacity ; 2.0L, Material PVC**
T-B2 **T-B3**	**Base solution tank**	**Vertical square tank** **Capacity ; 2.0L, Material PVC**
T-E1	**Electrode solution tank**	**Vertical square tank** **Capacity ; 3.0L, Material PVC**
P-S2 **P-S3**	**Salt solution pump**	**Magnet pump** **Capacity ; 3.0L/min×3m-H_2O**

		Material ; GFRPP, 20kW
P-A2 **P-A3**	**Acid solution pump**	**Ditto**
P-B2 **P-B3**	**Base solution pump**	**Ditto**
P-E1	**Electrode solution pump**	**Ditto**
P-S0	**Feed solution pump**	**Metering pump** **Capacity ; 0.1L/min × 0.2MPa**
P-A0	**Dilution water pump (for acid solution)**	**Ditto**
P-B0	**Dilution water pump (for base solution)**	**Ditto**
REC-2 **REC-3**	**Rectifier**	**DC ; 20A×35V**
	Frame	**Material SS + coating** **Remark caster provided**
	Valve, piping	**Material PVC soft and hard pipes**

BIOGRAHPY

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- June 2019 – July 2019 : Siam Cement Public Company Limited (SCG),
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