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PROCEEDINGS

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Utilization of Radish Sprout (*Raphanus sativus* L.) as Biocatalyst for Antioxidant Activity Improvement in The Reduction of Crude Curcuminoid from Turmeric (*Curcuma longa*) 72

Direct Adventitious Shoot Formation from Tomato Hypocotyls and Cotyledons 77

The Potential of Rhizospheric Fungi From Critical Land of Sub-District Cempaka, South Kalimantan as Fe-Absorbent on *Calopogonium Mucunoides* Plant 82

Funneliformis geosporum of Ujung Genteng Coastal Forest Sukabumi: It's Symbiosis and Potency as Biofertilizer 88

Mauritianin: The Main Flavonoids of *Carica papaya* L. Leaf 96

PHARMACY 105

Formulation of Metformin Hydrochloride Floating Tablet With HPMC K4m Cr And Sodium Bicarbonate 106

Differentiation of Leukocyte Sheeps Fed by Fraction Katuk Leaf Ekstract (*Sauropus Androgynus L. Merr*) 112

Potential Analgesic Agents and Action Mechanism of Phytochemicals from Indonesia Natural Products – A Review 117

Optimization of the Drying Method of Red Fruit (*Pandanus conoideus* Lam) by *Détente Instantanée Contrôlée* (DIC) 124

Combination For Stage 1 Hypertension Outpatient in Banjarmasin Ulin Hospital Period of 2013 133

CHEMISTRY 138

The Potential Recycling of Plastic Waste in North Banjarbaru District 139

Utilization of NaOH-activated Papyrus' (*Borassus flabellifer* L) Fiber as a Bio-sorbent for Pb (II) Ions 143

Biogas Quality Improvement Through Tube Purification Engineering by Using the Sodium Hydroxide Adsorbent in Public Farmhouse in Jabung District, Malang 149

Synthesis of Myristicyl Ethanoate from Myristicin Aldehyde 157

The Profile of Volatile Oil on *Blumea balsamifera* L. Calli 161

Isolation and Identification of Terpene From Lampasau (*Diplazium Esculentum* Swartz) Herbs. 166

Study on Precondition Time in Ion Selective Electrode (Ise) of *Methanil Yellow* 170

Adsorption of Methanyl Yellow by Humin 175

Study on Precondition Time in Ion Selective Electrode (Ise) of *Methanil Yellow*

Dewi Umaningrum[1*], Dahlena Ariyani[1], Radna Nurmasari[1]

[1]*Chemistry Department, Faculty of Mathematics and Natural Science, Lambung Mangkurat University Jl. A. Yani km. 35.8 Banjarbaru 70714, South Kalimantan. 70714*

Corresponding author : dewiumaningrum79@gmail.com

ABSTRACT

The sudy on precondition time in ion selective electrode (ISE) of methanil yellow has been done. The first step in this research was optimized of membrane composition by measuring of potensial value of chitosan, PVC and DOP membrane and calculate the Nernst factor. The membrane with Nernst factor nearly the Nernst factor teory 59,2 mV/decade was used to determine precondition time. Precondition time process was measured potensial value of ISE membrane in methanil yellow 1.10^{-1} – 1.10^{-8} M with variation time were 10, 20, 30, 40, 50, 60, 70, 80 minutes and calculated the Nernst factor. The results of this research showed that optimum membrane composition was 20% chitosan : 30% PVC : 50% DOP with Nernst factor 56,82 mV/decade concentration.The precondition time optimum at 50 minutes with Nernst factor 64,25 mV/decade concentration and $R^2 = 0.9992$.

Key words: Ion selective ion (ISE), methanil yellow, precondition time, potensial, nernst factor.

INTRODUCTION

The food actually plays an important role in nutrient and energy intake for children of school age. However, the level of food safety is very alarming. Whereas food safety is a key condition that must be possessed by the food manufacturers. Many of misuse of dangerous chemicals by food producers of snacks, such as using a preservatives like formalin, borax, dyes like rhodamine b (red), methanil yellow (yellow), malachite green (green), tartrazine and an artificial sweeteners such as like saccharin and cyclamate. These chemicals were a source of major risk to public health, especially the food were not controlled so that its existence was not observed. Therefore, to protect consumers, it needs to make a methanil yellow sensor based on potential solution measurement (potentiometric ISE).

Potentiometric as an inexpensive technique used in clinical laboratories, environmental and toxicology, and also can be used in the fields of pathophysiology, biotechnology and food testing technology (Atikah, 2011) . The advantages of Selective Ion (ISE) are having a high selectivity and sensitivity so that generally do not require the separation process. ISE is an electrochemical sensor which sensitive toward ion solution activity and characterized by potential changes (Bailey, 1976). Resulting an ideal ISE with Nernstian potential response, low detection limit, selective and has a long life time, it's necessary to election the active ingredient and the membrane compositio (Ardakani, 2004). This character can be achieved by mikroporus a membrane, quite hydrophobic, flexible so it has a large enough conductivity. Guibal, 2004 stated that chitosan have characteristics to conduct electricity so that the ISE membrane will have good conductivity. Chitosan also has an edge on the side $-NH_2$ which makes an active and polycationic so can act as an nanion exchanger. The ability of chitosan as an anion exchanger can be used when the process of protonation a pair of electrons from the N atom in the amide group converted into RH_3N^+ amine formation with the addition of a weak acid occurs at an acidic pH so it can bind anions by electrostatic bonding. It can be used as a reference so that the binding process functional group SO_3^- of methanil yellow can take place perfectly in the active group of chitosan.

Using a support materials were to improve the mechanical stability and homogeneity of the membrane. Some of supported polymeric materials that can be used in the membrane are

polyvinyl chloride (PVC), polyvinyl alcohol (PVA), polyamide, polyether, N-methylol nylon 6, polisiklosan and PMMA (Brouillet et al., 2009). PVC is a support material that is widely used in the ISE membrane because of the small pores (Lewis, 1997), stable in acidic or alkaline conditions (Souza, et al., 2006) and also hydrophobic so did not experience swelling (Sombatsri, 2008).

The addition of other materials with a lower molecular weight (plasticizer) will change the charateristic of rigid PVC into a flexible PVC (Wilkes, 2005). Plasticizer were used should be insoluble in water, not volatile and can descending the membrane Tg so can produce a flexible membrane. One type of plasticizer that is widely used as a plasticizer of PVC is the DOP. Dioktilftalat (DOP) which is an organic compound with a high viscosity (81.4 cp) at a temperature of 20°C, DOP plastic containing approximately 1% - 40% and has a large molecular weight (390.56 g / mol) and insoluble in water (Tehrani, 2010). In addition, making of the membrane is also required THF as a solvent (Efendy, 2001). THF tetrahydrofuran is the most polar eter forming, this solution is commonly used as a polar solvent. Using THF in the ISE membrane aims to homogenize the composition of the ISE membrane. Making of electrode membrane with evaporating the THF solvent from the mixture of PVC and electroactive materials which is added with a plasticizer. THF has a being polarity and dissolve a wide range of nonpolar and polar compounds (Lazo, 2000).

MATERIALS AND METHODS

Tools and materials

The equipment required in this research are a potentiometer Fisher Accumet models 955, reference electrode, analytical balance, an electric heater, spectrophotometer UV - Vis, FT - IR (SHIMADZU), magnetic stirrer, rod magnets (stirrer), stative, appliance glass/plastic commonly used in chemical laboratories.

The materials used in this research are methanil yellow, acetic acid 3 % (v/v), NaOH, H_3PO_4, CH_3COONa, NaH_2PO_4, saline, distilled water, the polymer polyvinylchloride (PVC), plasticizers Diocthylftalat (DOP), solvent Tetrahydrofuran (THF).

Procedures

1. Preparation of the electrode body.

Eelectrode body (construction) were made from Pt wire with a length of ± 10 cm and a diameter of ± 0.5 mm. At both ends ± 1.5 cm was left open and the other part is covered with plastic polyethylene, and then washed for 5 minutes to remove mechanical impurities and fat . Subsequently, it was rinsed with distilled water and dried with 96 % alcohol.

2. Preparation of the membrane.

Membranes were made by using variations of chitosan, DOP and PVC with the composition (in% w / w). The total weight of the composition of the mixture membrane material are 1 gram. The membrane mixture were dissolved in THF to weight ratio: volume = 1: 3 and stirred with a magnetic stirrer for 3 hours to obtain a homogeneous solution

3. Preparation of ESI methanil yellow coated wire type.

A forming of membranes solution coating on the Pt wire were done by plunging the Pt wire into a membrane solution for a while until a solution of the membrane attached to the Pt wire, followed by a drying wire that has been coated with a membrane solution in the open air for 30 minutes and heating in an oven at 50 ° C for 12 hours and cooled.

4. The timing of the preconditions

ISE were preconditioned in a solution of 1 M methanil yellow with time during ranging from 10-80 minutes with an interval 10 minutes. From the measurement results will be generated curves Nernst value on the y axis and precondition times on the x axis. The potential value were determined Nernst factors approaching the theoretical Nernst price, ie 59.2 mV / deck.

RESULTS

1. Preparation of ESI methanil yellow .

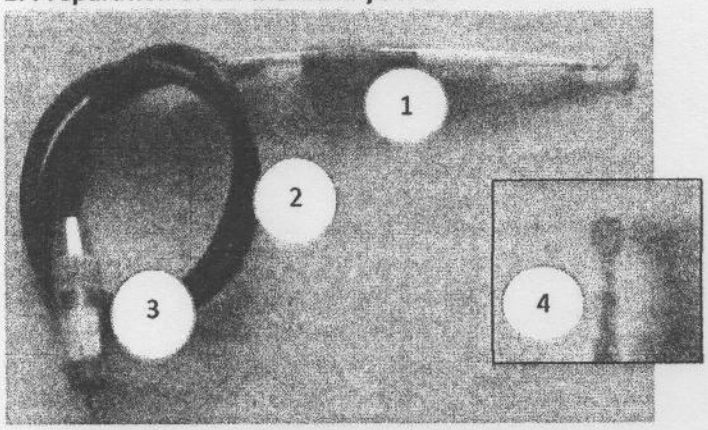

Figure 1. Construction of ISE Methanil Yellow

2. Optimization of precondition times.

Table 1. Precondition times for membran ISE

Concentration	Potential value at minutes -							
	10	20	30	40	50	60	70	80
1.10^{-1}	182,3	132,2	184,1	164,2	144,1	114,9	1	246
1.10^{-2}	298	288,2	272,4	202,3	212,4	222,8	122	261
1.10^{-3}	325,67	320,3	331,8	251,2	276,9	267,9	142	285
1.10^{-4}	350,7	328,2	366,1	346,1	336,9	316,9	175	306
1.10^{-5}	455,6	239,2	339,9	239,1	212,2	292	174	368
1.10^{-6}	444,67	201,6	321,2	262,2	279	239	218	290
1.10^{-7}	536	316,9	326,9	285,3	215,9	295,9	184	250
1.10^{-8}	517	292,8	241,8	221,3	192,2	199,2	184	159
Nernst factor	53,287	62,01	60,54	59,46	64,29	65,11	24,5	20,4

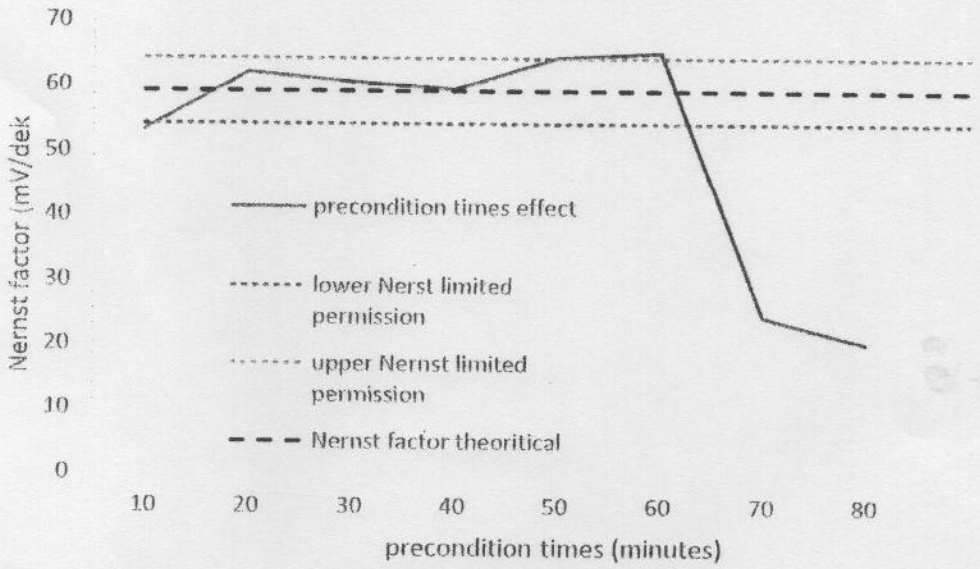

Figure 2. Optimization of immersion time

DISCUSSION

1. Preparation of ISE methanil yellow.

ISE methanil yellow design were made by coating the Pt wire with appropriate membrane specific composition. The ISE methanil yellow design can be seen in Figure 1. ISE methanil yellow design included : 1. Electrodes body made from plastic, 2. Cable RG 58.3 as a connector connecting the potentiometer, and 4. The shape of the membrane was coated on the Pt wire. ISE methanil yellow design has a simple construction and more efficient so it is suitable for the quantitative analysis and routine in the field when compared to other commonly used methods, such as thin-layer chromatography and visible spectrophotometry. The analysis process can be done more efficiently because it requires only a relatively small sample so it is portable in a way, easy to use and inexpensive.

2. Optimization of membrane composition.

The composition membrane of chitosan : PVC: DOP = 20%: 30%: 50% is the optimum composition of several variations that has been done, because it gives Nernst factor f 56.82 mV / deck nearly the theoretical Nernst factor is 59.2 mV / deck for monovalent anion.

3. Optimization of precondition time.

The aim of the precondition time to reduce the resistance of the membrane that is characterized by an increase of the membrane conductivity. Precondition time to saturate the membrane with the sensored ion. Precondition time is one of the factors that influence the quality of the ISE shown by Nernst factor. One of the most widely used method is to soak the membrane in the analyte with high concentrations. This method has the effectiveness due to the precondition process, the membrane will be saturated with the ions to be censored, causing the conductivity of the membrane becomes uniform and thorough so that would be obtained reproducibility and a good life time of the membrane ISE generated.

Based on the above reasons , the research were done by soaking an ISE in a methanil yellow solution with concentration range $1.10^{-1} - 1.10^{-8}$ M with a variation of a certain time. Effect of precondition times to Nernst factors is presented in Figure 2 and Table 1. Based on Figure 1 and Table 1 can be seen that there are some times at permitted Nernst theoritis ranges from 54.2 to 64.2 mV / deck (Atikah, 2005) , but at 60-80 minutes, it describe the amount of water included in the excessive membrane causing the membrane pores become enlarged , then swelling of the

membrane and the membrane sensitivity is reduced and difficult in ion exchange because it was blocked by the presence of water . This is why the Nernst factor less than the theoretical value. Precondition times which is allowed the Nernst factor criteria at 10 minutes and a maximum at 60 minutes , but the precondition times closest to the theoretical Nernst factor at the 50 minutes with the resulting Nernst factor is 64.29 (mV / deck) with R^2 value of 0.9992 .

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CERTIFICATE OF PARTICIPATION

This certificate is awarded to:

Dewi Umaningrum

In recognition of his/her contribution as a

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