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The Coated-Wire Ion-Selective Electrode (CWISE) of Tartrazine Using Chitosan as an Ionophore

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Abstract

Research on the Ion-Selective Electrode (ISE) of coated wire-type tartrazine using chitosan as an ionophore has been developed. The variables used in the manufacture of ISE are membrane composition and immersion time. Meanwhile, the basic characteristics of ISE measured are Nernst value, measurement concentration range, detection limit, and measurement response time. The results showed that ISE tartrazine coated wire type had an optimum membrane composition in a mixture of chitosan: PVC: DOP of 3: 34: 63 (% w/w) and a membrane immersion time 20 minutes. The basic characteristics of ISE produce a Nernst value of 20.976 mV/decade. The measurement concentration range is 1×10^{-7}–1×10^{-2} M with a detection limit of 2.749×10^{-7} M or 0.1469 ppm. The response time ranges from 10-60 seconds, with an average of 40 seconds.

1. Introduction

Food additives are generally used in processed food ingredients during the packaging, processing, and storage processes that are useful for improving appearance, taste, color, texture, preservation, and improving food quality [1]. The most used food additives are dyes, including natural and synthetic dyes. When compared with natural dyes, synthetic dyes over the last four decades have been widely used in the food industry because they have good solubility in water, high stability to oxygen, light and pH changes, low cost, high brightness, a wide color range, and little interference from microbes [2, 3, 4, 5].

Tartrazine is an azo synthetic dye that is bright yellow and is used in food products such as beverages, sweeteners, dairy products, bakery, and fast food products, ink industry, paints, cleaning products, detergents, paper and fabrics, pharmaceuticals, cosmetics [6, 7, 8]. In 2016 the Joint FAO/WHO Expert Committee on Food Additives (JECFA) set the acceptable daily intake (ADI) for tartrazine at 0 to 10.0 mg/kg body weight. Excessive intake of tartrazine can cause allergies, asthma, migraine, eczema, anxiety, diarrhea, hyperactive behavior in children, acute oral toxicity, skin toxicity, chromosomal damage, and thyroid cancer [6, 9, 10]. Therefore, it is necessary to carry out a quantitative analysis of tartrazine in food products.

Several methods are used to determine tartrazine quantitatively, including high performance liquid chromatography [11], spectrophotometry [12, 13, 14], colorimetry [15], fluorescence [16], mass spectroscopy [17] and stop-flow analysis [18]. These methods have the advantage of being very sensitive and accurate. However, these methods require large amounts of reagents, take a longer time because they have to do sample preparation, require experts to operate the instrument because they use sophisticated equipment and are difficult to apply in the field. Therefore, a new simpler, cheaper, selective, sensitive, and fast method is needed.

The electrochemical analysis method is one of the analytical methods that has received much attention because it shows satisfactory measurement results. Several studies of electrochemical determination of tartrazine were carried out by modifying the electrodes,

including the preparation of carbon paste electrodes with cetyl trimethyl ammonium bromide (CTAB) as the ionophore (electrode A) and silver wire coated with CTAB (electrode B) [19], screen-printed carbon electrode (SPCE), modified graphite oxide (ERGO-SPCE) [20], silica modified carbon paste electrode impregnated on cetylpyridinium chloride (CPE/ SG/CPCI) [21], and graphite electrode of pencil modified with graphene/poly (L-phenylalanine) [4]. The weakness of the above studies is that the ionophore used is relatively expensive, so a cheaper alternative is needed. One of the ionophores that can be used is chitosan. Another drawback is that the surface of the paste electrode is easily contaminated, so the paste must be cleaned frequently. This is the reason for using coated wire-type ion-selective electrodes (ISE). The advantages of this ISE are that it has low detection limit characteristics similar to a tube and paste types, has a simple construction because it does not require an internal comparison electrode system, is small and inexpensive, and has mechanical stability so that it is possible to use it once (disposable) which is suitable for field analysis [22, 23, 24].

The choice of chitosan as an ionophore is due to the ability of chitosan to conduct electricity to produce ISE with good conductivity. Chitosan can act as an anion exchanger because of the $-NH_2$ group, which is active and polycationic. The protonation of a pair of electrons from the N atom of the amide group is converted in the form of an amine to RH_3N^+ by adding a weak acid (e.g., acetic acid) capable of binding the anion by electrostatic force [25]. This is used as a reference so that the binding process of the SO_3^{2-}tartrazine group can take place ideally on the active group of chitosan.

In this research, coated wire-type ISE was made using chitosan as an ionophore. The effect of membrane composition and ion-selective electrode characterization, including immersion time, Nernst value, linear concentration range, detection limit, and response time, were also studied.

2. Methodology

2.1. Tools

The tools used in this study included a digital multimeter SANWA CD800A, glass tube Ag/AgCl Koslow electrode as a comparison electrode, Pt wire (10 cm long and 0.5 mm in diameter), polyethylene plastic, RG-58 coaxial cable, Memmert oven, O'haus analytical balance, Thunder PS10A magnetic stirrer, and common laboratory glassware such as 1000 mL, 100 mL, and 50 mL Duran beakers, dropper pipettes, 1 mL precicolor HBG and 2 mL volume pipettes Iwaki, 5 mL and 10 mL Iwaki measuring cups, spatula, porcelain cup, 10 mL Iwaki flask, and Thermo Scientific pH-meter.

2.2. Materials

The materials used in this research include chitosan DD 85%, tartrazine T0388-100G (Sigma Aldrich), CH_3COOH 99% (Merck), concentrated HNO_3 (Merck), alcohol 96% (Merck), dioctyl phthalate (DOP) D201154-500 mL (Sigma Aldrich), polyvinyl chloride (PVC) 81387-250G (Sigma Aldrich), tetrahydrofuran (THF) (Merck) and distilled water.

2.3. Research procedure

Platinum wire (length = 10 cm and diameter = 0.5 mm) was used as the electrode body was immersed in concentrated HNO_3 for 5 minutes, then rinsed using distilled water and dried with 96% alcohol (the lower end of the wire). The bottom end of the clean wire was then coated with a membrane mixture consisting of 1 gram of chitosan, polyvinyl chloride (PVC), and dioctyl phthalate (DOP), and 3 mL of tetrahydrofuran (THF) solution was added. After the coating was completed, the electrodes were dried for 30 minutes and heated in an oven at 50°C for 12 hours. The tartrazine ISE that had been made was then assembled and used Ag/AgCl electrodes as comparison electrodes. The two electrodes were connected to a digital multimeter.

In optimizing the composition, the membrane constituent materials were made with several variations (Table 1). Each composition was used to measure the potential value in the 1×10^{-1}–1×10^{-8} M tartrazine solution series. The measurement data obtained were graphed the relationship between E (mV) and log[tartrazine]. The curve obtained is a straight line in a specific concentration range with a slope of -2,303 RT/nF, the Nernst value.

Table 1. Variations in membrane composition

Membrane Composition	Material composition (%)		
	Chitosan	PVC	DOP
A	3	35	62
B	3	34	63
C	4	35	61
D	4	34	62
E	5	34	61
F	5	35	60
G	5	33	62

In optimizing the immersion time, tartrazine ISE was made by coating a membrane with the optimum composition and immersed in a 0.5 M tartrazine solution with an immersion variation of 10-80 minutes with an interval of 10 minutes and measuring the potential value on the tartrazine solution series 1×10^{-1}–1×10^{-8} M. The measurement data obtained were graphed the relationship between E (mV) and log[tartrazine].

In determining the basic characteristics of ISE tartrazine, the Nernst value was determined by measuring the potential value of ISE tartrazine 1×10^{-1}–1×10^{-8} M. The measured data were processed and extrapolated into a graph of the relationship between the log [tartrazine] and the measured potential value (mV) so that Nernst value, linear concentration range and detection limit were obtained. The response time was determined by measuring the potential value of the tartrazine solution with an interval of 10-180 seconds.

3. Results and Discussion

The first step in the manufacture of ISE tartrazine is the manufacture of membranes. The membrane used was a mixture of chitosan: PVC: DOP with a total weight of 1 g with a ratio of % by weight of chitosan: PVC: DOP, i.e., 3: 34: 63 dissolved in 3 mL of THF. The membrane mixture was coated on the Pt wire. Pt wire is used because Pt is inert, not physically and chemically affected, and resistant to membranes. Pt wire coated with a membrane thickness of 0.35 mm, then dried in the open air for several minutes and then heated in an oven at 50°C for 12 hours (Figure 1).

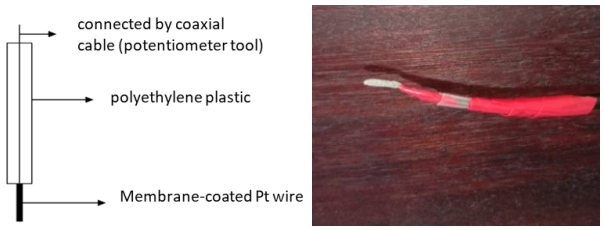

Figure 1. Membrane-coated Pt wire electrode

The ISE obtained was then immersed in a 0.5 M tartrazine solution for 20 minutes. The immersion process is carried out to saturate the membrane with ions trapped in the sensor and fill the amount of water on the membrane to experience good ion dissociation [26]. Next, the ISE was rinsed with distilled water and dried before being used for measurement. The mechanism of ion exchange that occurs at the chitosan membrane interface with tartrazine solution at the membrane-solution interface is proposed in Figure 2.

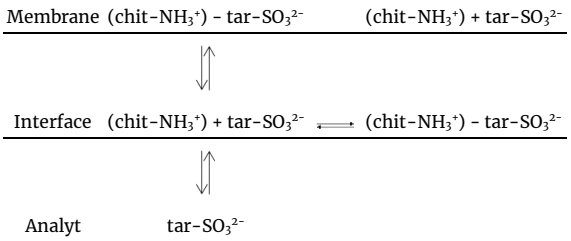

Figure 2. The membrane-solution interface ion-exchange mechanism

The outside of the membrane is in direct contact with the analyte solution when measurements are made so that the membrane active ingredient, namely chitosan, dissociates into free ions at the interface of the membrane with the solution, the active ingredient chitosan, in which $(R–NH_3)^+SO_3^{2-}$ dissociates into cations $(R–NH_3)^+$ and the anion SO_3^{2-}. The ion exchange reaction of the analyte with free ions on the active site of the membrane occurs when the anions present in the solution can reach the interface boundary of the membrane with an immiscible solution until an electrochemical equilibrium is reached. Furthermore, an ion-exchange reaction occurs between SO_3^{2-} ions in the analyte solution with free ions on the active site of the membrane (membrane-solution interface reaction) and forms a salt association $(R–NH_3)^+SO_3^{2-}$ which separates into the membrane. This process continues until equilibrium is reached, which is indicated by the constant potential value. The reaction mechanism of chitosan with tartrazine is shown in Figure 3.

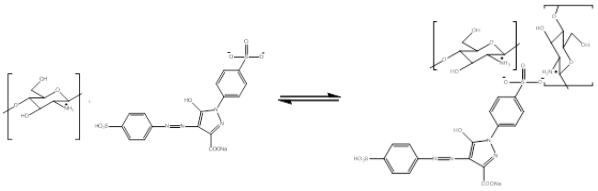

Figure 3. The reaction mechanism of chitosan and tartrazine

Based on Figure 3, the active group of chitosan $–NH_3^+$ can interact strongly on the surface of a solution that has a negative charge, in this case, the sulfite group on tartrazine. When soaked in 0.5 M tartrazine solution, the ammonium ion in the chitosan binds to the sulfite ion. The ion is trapped in the membrane, which acts as a comparison solution when measuring the potential value. The process of exchanging sulfite ions at the membrane interface with the analyte solution continues until it reaches equilibrium which is indicated by a constant potential value. This can also occur due to differences in analyte concentrations on the internal and external sides of the membrane.

3.1. Optimization of Membrane Composition and Membrane Immersion Time

Membrane composition is an important parameter to determine the quality of an ISE. The appropriate amount of PVC, chitosan, and DOP affects the sensitivity and selectivity of ISE. In this study, seven membranes were made with several compositions (% w/w) consisting of the active ingredient chitosan, PVC support material, and DOP as a plasticizer, and THF as a solvent. The measurement of the potential value and Nernst value for each composition are presented in Table 2.

Table 2 shows that membrane B with the composition of chitosan: PVC: DOP of 3:34:63 (%w/w) is the optimum tartrazine ISE membrane because it has a Nernst value of 20.61 mV/decade with an R^2 value of 0.943, close to Nernst. The theoretical value of tartrazine is 17.6-19.4 mV/decade [19], where the concentration range is linear.

Table 2. Membrane composition of ISE tartrazine

Membrane composition	Material composition (%)			The potential value of tartrazine (mV) at a concentration (M)								Nernst value	R^2
	Chitosan	PVC	DOP	10^{-1}	10^{-2}	10^{-3}	10^{-4}	10^{-5}	10^{-6}	10^{-7}	10^{-8}		
A	3	35	62	71.2	68.0	57.4	56.3	45.2	20.8	19.0	11.7	10.46	0.924
B	3	34	63	161.1	123.6	77.4	65.4	43.5	20.2	18.2	13.0	20.61	0.943
C	4	35	61	119.0	46.8	7.4	37.0	30.7	8.3	27.5	11.5	2.89	0.118
D	4	34	62	85.1	7.0	45.8	77.4	69.8	58.0	40.8	20.3	6.6	0.242
E	5	34	61	83.0	89.4	65.4	44.5	39.0	36.5	30.0	28.6	11.12	0.859

F	5	35	60	93.5	35.4	48.9	42.2	28.8	23.9	16.9	0.4	5.21	0.666
G	5	33	62	332.4	11.1	28.0	16.3	14.4	9.5	3.0	17.4	2.79	0.369

The value of R^2 in membrane composition B is higher than the others. This shows a linear correlation between the resulting cell potential value and the concentration of SO_3^{2-} anion so that this ISE can be used to determine the concentration of tartrazine. In membrane compositions C to G, more chitosan causes swelling of the membrane, thereby increasing the movement of molecular chains. The sulfite ion in the membrane-bound tartrazine undergoes diffusion so that the membrane is filled with other ions present in the solution or water molecules. This causes the measured potential value to decrease so that the Nernst value is also small. The addition of more PVC also affects the Nernst value. This is because the membrane's mechanical properties become more robust so that the movement of ions in the molecular chain is limited. The structure of chitosan in the membrane becomes denser and stiffer so that fewer ions are exchanged, and this causes the potential value to decrease. The Nernst value to be smaller than the theoretical Nernst value.

Immersion is one part of the conditioning process that saturates the ISE with objections to the membrane. Immersion accelerates the response time and causes the electrostatic interactions in the anion exchange to be specific and produce a response potential that can be Nerstian [27]. Membrane immersion was carried out in 0.5 M tartrazine solution for 10-80 minutes with 10-minute intervals.

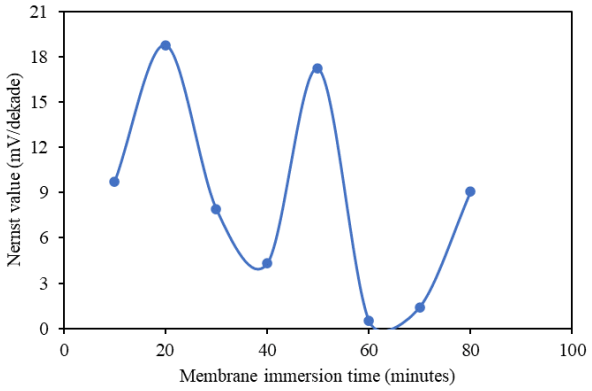

Figure 4. Optimization of membrane immersion time

Figure 4 shows that the optimum immersion time is 20 minutes with a Nernst value of 18.79 mV/decade of concentration. This is because the need for water in the membrane for the dissociation of sulfite ions has been met, causing the electrostatic interactions on anion exchange to be specific and produce a potential response that can be Nerstian. At 10 minutes, it is estimated that the number of sulfite ions on the membrane surface is still too small and cannot interact ionically. At the time of immersion above 20 minutes, there was a decrease in the Nernst value because the membrane was in contact with the tartrazine solution for too long, resulting in swelling. The membrane's pores become large, so ion exchange is difficult because it is blocked by water. However, there was a deviation at 50 minutes. The Nernst value increased, which was probably caused when the membrane began to swell. The sulfite ions in the membrane increased, causing the response potential to increase.

3.2. Basic Characteristics of ISE Tartrazine

An ISE is said to be good if it has a Nernst value close to theoretical, has a low detection limit, and has a wide concentration range. Nernst value greatly determines the feasibility of ISE as a measuring tool in analysis. Deviations from the theoretical value can cause an ISE not suitable for analysis in a sample. The Nernst value was determined by measuring the Potential value of the tartrazine solution from a concentration of 1×10^{-1} M to 1×10^{-8} M using the prepared ISE.

Table 3. The potential value of tartrazine solution

[Tartrazine] (M)	Log[tartrazine]	The potential value of ISE Tartrazine (mV)			
		1	2	3	average
1×10^{-1}	-1	165.7	160.1	159.3	161,7
1×10^{-2}	-2	123.8	123.9	123.2	123,6
1×10^{-3}	-3	77.5	77.3	77.5	77.4
1×10^{-4}	-4	65.5	65.3	65.4	65.4
1×10^{-5}	-5	43.8	43.6	43.1	43.5
1×10^{-6}	-6	20.2	20.3	20.2	20,2
1×10^{-7}	-7	18.5	18.0	17.7	18.1
1×10^{-8}	-8	13.0	13.0	13.1	13.0
Nernst value (mV/decade)		21.298	20.856	20.775	20.976
Concentration Range		1×10^{-7}–1×10^{-2} M			
Detection Limit		2.749×10^{-7} M or 0.1469 ppm			

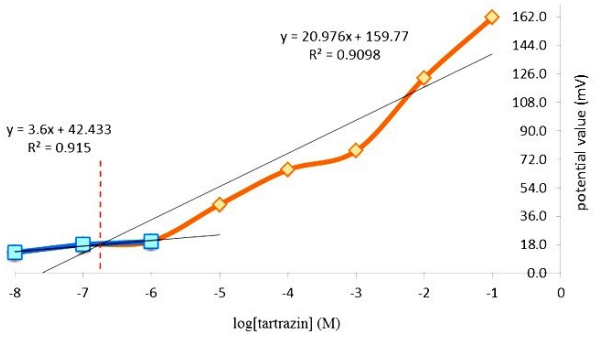

Figure 5. Relationship of log[tartrazine] with Potential value (mV)

Based on Table 3 and Figure 5, the Nernst value obtained is 20.976 mV/decade with an R2 value of 0.9098. The value of R2 shows a linear correlation between the Potential value as measured by the concentration of tartrazine so that this ISE can be used to accurately determine the concentration of tartrazine. The concentration range of the ISE measurements of tartrazine made is shown from the linear line on the graph of the relationship between Potential value vs. log[tartrazine] (Figure 5), which is in the concentration range of 1×10^{-7}–1×10^{-2} M.

Based on Figure 5, the linear area has a value of y = 20.976x + 159.77 while the non-linear area has a value of y = 3.6x + 42.433. The intersection of these two lines becomes a reference for the detection limit or the minimum limit for tartrazine that can be measured by the tartrazine ISE made. The detection limit of this measurement using the ISE of tartrazine reaches 2.749×10^{-7} M or 0.1469 ppm. Based on this, the ISE of tartrazine can be used as a measuring tool for sample analysis. The comparison of tartrazine ISE characteristics of this study with ISE made by Abu Shawish *et al.* [19] is presented in Table 4.

Table 4. Comparison of ISE characteristics of tartrazine

Characteristics of ISE Tartrazine	ISE tartrazine type chitosan membrane coated wire: PVC: DOP	ISE Tartrazine type modified carbon paste CTAB as an active ingredient [19]	ISE CTAB-coated and modified silver tartrazine as an active ingredient [19]
Nernst value (mV/decade)	20,976 mV/decade	17,9 mV/decade	19,4 mV/decade
Concentration range (M)	1×10^{-7}–1×10^{-2} M	4.3×10^{-7}–1.0×10^{-2} M	1.1×10^{-7}–1.0×10^{-2} M
The detection limit (M)	2.749×10^{-7} M	3.2×10^{-7} M	5.5×10^{-8} M

Based on Table 4, it can be seen that the ISE of tartrazine type coated wire has results that are close to the results of previous studies conducted by Abu Shawish *et al.* [19], with the Nernst value obtained ranging from 17.9-19.4 mV/ decade. In comparison, the Nernst value of this study is 20.976 mV/decade.

The range of concentrations obtained for ISE tartrazine in this study is also close to the theoretical results of the research conducted by Abu Shawish *et al.* [19]. The concentration range obtained is quite broad. The ion exchange capacity of the membrane affects the width of the linear concentration range of an ISE. The detection limit obtained is also close to the value of the results of previous studies. Based on these data, the wire type ISE coated with tartrazine with chitosan as the ionophore fulfills one of the requirements of the ideal ISE characteristics.

Response time is the time required to achieve equilibrium between sulfite ions in solution and ammonium ions in the membrane at each measurement of tartrazine solution until each shows a fixed cell potential value. If the response time obtained is faster, the ISE performance is getting better and more stable.

Table 5. Response time for each concentration of tartrazine solution

Log[tartrazine]	Response time (s)	The potential value of ISE tartrazine (mV)			
		1	2	3	average
-1	60 seconds	119.1	119.0	118.8	119.0
-2	50 seconds	63.7	63.4	64.3	63.8
-3	60 seconds	28.0	28.5	29.1	28.5
-4	60 seconds	33.1	33.2	33.7	33.3
-5	60 seconds	19.0	19.2	19.5	19.2
-6	10 seconds	20.0	20.1	20.2	20.1
-7	10 seconds	8.4	8.0	8.0	8.1
-8	10 seconds	19.4	19.5	19.3	19.4
Average		40 seconds			

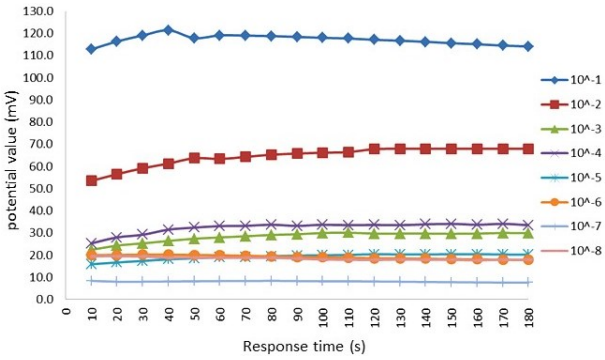

Figure 6. Correlation of potential value of tartrazine solution vs. response time

Based on Table 5 and Figure 6, the response time for a tartrazine solution with a concentration of $1\times10-2$ M is 50 seconds, for a concentration of 1×10^{-3} M to 1×10^{-5} M is 60 seconds, and for a concentration of 1×10^{-6} M and 1×10^{-7} M is 10 seconds. If averaged, the response time for the tartrazine ion-selective electrode with the optimum composition 3: 34: 63 ranges from 10 to 60 seconds. Determination of response time is influenced by analyte concentration. The time obtained will be fast if the analyte concentration is high or concentrated, and vice versa [26]. However, if seen from Table 3, there are slight deviations at the concentrations of 1×10^{-6} M and 1×10^{-7} M. This is due to the influence of the measured analyte concentration. If the measurement is carried out from low concentration to high concentration, i.e., from 1×10^{-8} M to 1×10^{-1} M, the response time obtained will be better when compared to measurements made on the contrary. However, the results of measuring the potential value obtained are still relatively fast. This means that the ISE used is of good value because it can reach equilibrium and ion association quickly. Based on Table 5 and Figure 6, the ISE of tartrazine made has fulfilled one of the characteristics of the ideal ISE, where the response time can be achieved for ±1 minute in ±5 minutes of measurement [19].

4. Conclusion

Based on the study results, it can be concluded that the coated wire type ISE tartrazine has an optimum mixed membrane composition of chitosan: PVC: DOP of 3:34:63 (%w/w) and a membrane immersion time of 20 minutes. The basic characteristics of the ISE produced resulted in a Nernst value of 20.976 mV/decade. The measurement concentration range was 1×10^{-7}–1×10^{-2} M with a detection limit of 2.749×10^{-7} M or 0.1469 ppm, the response time ranged from 10- 60 seconds with an average of 40 seconds.

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