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Developing a potentiometric sunset yellow selective electrode and its applications

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Abstract

We developed a potentiometric ion-selective electrode (ISE) for detecting the food dye sunset yellow (SY). Sunset yellow-Methyltrioctylammonium chloride ion pairs were synthesized. The ion pair that was synthesized was employed as ionophorein the configuration of the electrode membrane. PVC membrane ion-selective electrodes in various compositions were produced using the ion pairs that were synthesized and then, potentiometric performance characteristics of these electrodes were investigated. It was determined that the electrode with the composition of 3.0 % sunset yellow-Methyltrioctylammonium (Sunset-MTOA) ion pair, 64.8 % nitrophenyloctyl ether (NPOE), 32.0 % polyvinylchloride (PVC) and 0.2 % potassium tetrakis (4-chlorophenylborate) (KTpClPB) demonstrated the best potentiometric performance properties. The linear range, slope, limit of quantification, pH range, and the response time of the electrode were determined as 1.0×10^{-5}-5.0×10^{-2} M, 23.6 mV,1.0×10^{-5} M,6.4-9.1, and ≈ 5 sec, respectively. The electrode exhibited a highly repeatable potentiometric response.

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1. Introduction

Food dyes are added into foods to provide them with their natural colors that are lost during processing and storing, enhance their pale natural colors, color the originally colorless foods, and obtain appealing and acceptable products provided that they do not hide their low quality [1-3]. Food dyes form an important group in food additives and play a significant role in increasing the appeal of foods[4]. Coloring agents are also used in some foods to remove the color disorders that emerge due to employed food processing techniques. Besides, coloring agents are also used to ensure homogeneous color distribution, make the appearance attractive, and give foods color in novel formulations [5]. Sunset yellow FCF (E110), an azo dye, has long been used as a colorant in many processed foods and food additives. It is used in the production of bakery products, pastry, desserts, snacks, icecreams, beverages and canned fish, instant soups and some drugs in syrup and tablet form. The acceptable daily intake is 2.5 mg/kg over body weight [6].

However, it is worth noting that sun set yellow (SY) is a synthetic dye that exists in widespread food products which may cause allergies, diarrhea, anxiety, and migraine and also it could damage kidneys and livers if it is overconsumed[7-9]. Therefore, it is very important to adjust the daily intake dosage of sunset yellow and control it. There are advanced techniques that necessitate sophisticated equipment and expensive consumables such as the extraction methods [10-12], spectrophotometry [13-15], thin layer chromatography [16] high-performance liquid chromatography [17-19], capillary electrophoresis[20-22] and electrochemical voltammetric methods[23-33] for the quantification of sunset in the literature. The above-mentioned analysis techniques are time-consuming and require sophisticated apparatus, expensive consumables, mostly pretreatment processes and experienced users. However, the potentiometric techniques provide benefits such as short analysis time, low cost, wide concentration range, low limit of quantification, high accuracy, appropriate selectivity, not requiring pretreatment process mostly, sophisticated devices, and experienced users. In this manner, the fabrication of a sunset yellow-selective potentiometric electrode for the quantification of sunset yellow food additive will provide facilities for analytical purposes.

Almost no studies on direct potentiometric determination of sunset yellow by using a selective electrode are available in the literature. We came across only one study [34] that aimed to develop a

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*Corresponding author. *Email address: gsoydan2002@hotmail.com* sunset yellow selective electrode. Therefore, the development of new sunset yellow selective electrodes with better performance characteristics is an important requirement. In this context, in the current study, we attempted to fabricate a potentiometric all-solid-state PVC membrane ion-selective electrode with improved characteristics. For that purpose, initially, sunset yellow-methyltrioctylammonium ion pair type was synthesized. It was employed as the active component in the structure of the PVC membrane ion-selective electrode. All-solid-state PVC membrane sunset yellow selective electrodes were produced using the ion pair that was synthesized. Also, membrane compositions (PVC: plasticizer, ionic component: ion pair) were arranged to obtain the best potentiometric performance properties. The potentiometric performance properties (The limit of quantification, linear range, response time, selectivity, pH range, lifetime, repeatability, sensitivity) were determined for the electrode with excellent characteristics. Finally, an application was performed for quantification of sunset yellow using the produced electrode, the obtained result was compared with the result that was obtained using another method.

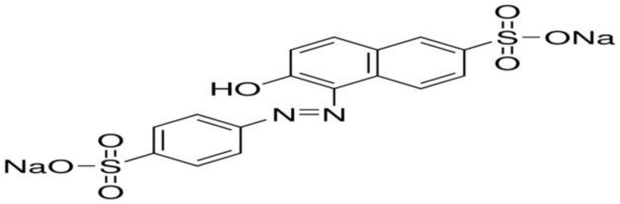

Figure 1. Molecular structure of Sunset Yellow FCF (E110)

2. Materials and Methods

2.1. Chemicals used in the study

Tetrahydrofuran (THF), high molecular weight poly (vinyl chloride) (PVC), o-nitrophenyl octyl ether (NPOE), dibutyl phthalate (DBP), dioctyl phthalate (DOP), potassium tetrakis (4- chlorophenyl) borate (KTpClPB), and graphite powder were procured from Fluka (Bucks, Switzerland). Sunset yellow (SY), Methyl trioctyl ammonium chloride (MTOA), dioctyl sebacate (DOS), Tris, Tris hydrochloride, HCl, H_3PO_4, and NaOH and candies were procured from Sigma-Aldrich (Germany) and markets in Turkey, respectively.

The epoxy (TP3100), used in the preparation of solid-contacts and hardener (Desmodur RFE) were obtained from the Denlaks (Turkey) company and the Bayer (Germany) company, respectively. All salts that were used when preparing the solutions in the study were procured from the Sigma-Aldrich (Germany) Company. All solutions were prepared by 18.3 M Ω deionized pure water.

2.2. Equipment

A computer-controlled potentiometric measurement system was used in performing the potentiometric measurements. In all potential measurements, the Ag/AgCl electrode (Gamry, USA) was employed as reference electrode. A desktop OHAUS measurement device was used for the pH measurements.

2.3. Standard solutions

The standard solutions used in the measurements were prepared using analytical grade substances. Firstly, 5.0×10-2 M standard solution of Isopreterenol was prepared in deionized water. Its solutions at the other concentrations were prepared by diluting its standard solution at the concentration of 5.0×10-2 M with deionized water. After the pH range study was conducted, standard solutions of each type at the concentration of 5.0×10-2 M were prepared at pH = 7.4 Tris buffer. Then, the standard solutions of these solutions at required concentrations in the study were prepared by diluting the initially prepared standard solutions with 5.0×10-2 M concentration using pH = 7.4 Tris buffer.

2.4. Preparation of the buffer solution

The buffer solution was prepared as follows: 1.21 g Tris was dissolved in deionized water, 7.9 g Tris HCl was added, stirred and dissolved in this way, and after the pH was adjusted to 7.4, it was made up to 1 lt with deionized water.

2.5. Synthesis of ion pair that was employed as ionophore in electrodes

Chemical structures of the ion pair forming agents used in the synthesis of ion pairs were shown in Figure 2.

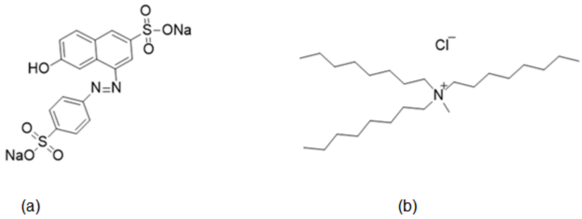

Figure 2. Chemical structures of (a) sunset yellow and (b) methyltrioctylammonium chloride.

To synthesize the Sunset yellow-Methyltrioctylammonium (SY-MTOA) ion pair, employed as ionophore in the structure of electrodes, 10 mL of the 10^{-2} M sunset yellow solution was slowly added into the 10 mL of the solution that contained 10^{-2} M Methyltrioctylammonium chloride and was stirred continuously. The formed precipitate was washed with deionized water 5 times, centrifuged at 4000 rpm for 5 minutes each time and left to dry in dark under room conditions. After it was dried, it was used directly as ionophore.

2.6. Preparation of the electrodes

In the study, the SY-MTOA ion pair was used as ionophore at various rates and SY-selective electrode was formed. Electrode production takes place in two stages in general. The first stage consists of the preparation of the solid contacts that form the surface on which the membrane will be coated, and the second stage consists of the preparation of the membrane cocktails that contained SY-MTOA ion pairs and coating the solid contact surfaces with it. The solid contacts were prepared by submerging of one of the open ends of a copper wire into a mixture that contained 50% (a/a) graphite, 35% epoxy (a/a), and 15% (a/a) hardener and was homogenized, and then its drying under room conditions for a period of 24 hours. All the PVC membranes constantly contain 32% PVC. The other components of the PVC membranes comprised of plasticizers at different types, ion pairs, and sometimes ionizer component. The membranes were prepared by solving of total membrane mass of 100 mg in 2 mL THF. The solid contact surfaces were coated by being submerged into the prepared PVC membrane cocktails for a few times and were left to dry under room conditions for at least 24 hours. After the electrode membranes were dried, the electrodes were submerged into the 20 mL 10^{-2} M sunset yellow solution in the measurements taken in the aqueous solution and 10^{-2} M sunset yellow containing 20 mL standard solution, which was prepared in pH=7.4 Tris buffer, in the measurements to be taken in buffer, for 12 hours and they were conditioned and made ready for measurement. The electrodes were kept in dark under laboratory conditions when not in use. Before starting each measurement, the electrodes were kept in the conditioning solution for half an hour.

3. Results and Discussion

3.1. Investigation of the optimum membrane composition

To detect the electrode that displayed the best potentiometric performance characteristics, different membrane compositions were prepared by changing the rates of SY-MTOA ion pair and the other parameters. The potentiometric performance characteristics (slope, the limit of quantification, linear range, and R^2 value for the calibration curve) of the electrodes that were prepared using these membranes were investigated. The compositions of 27 electrode membranes prepared using the SY-MTOA ion-pair were given in Table-1.

Table 1. The compositions of the electrode membranes that were prepared using Rac-TPB ion pair as ionophore

	Composition (mg %)					
Electro	Ionophore (SYMTOA),%	PVC,%	NPOE,%	DBP,%	DOS,%	KTPClPB
A1	1	32	-	-	66	1
A2	1	32	-	-	67	-
A3	1	32	-	66	-	1
A4	1	32	-	67	-	-
A5	1	32	67	-	-	-
A6	1	32	66	-	-	1
A7	3	32	65	-	-	-
A8	3	32	64	-	-	1
A9	3	32	-	65	-	-
A10	3	32	-	64	-	1
A11	3	32	-	-	65	-
A12	3	32	-	-	64	1

A13	5	32	62	-	-	1
A14	5	32	63	-	-	-
A15	5	32	-	-	63	-
A16	5	32	-	-	62	1
A17	5	32	-	63	-	-
A18	5	32	-	62	-	1
A19	3	32	64.9	-	-	0.1
A20	3	32	64.8	-	-	0.2
A21	3	32	64.7	-	-	0.3
A22	3	32	64.6	-	-	0.4
A23	3	32	64.5	-	-	0.5
A24	3	32	64.4	-	-	0.6
A25	3	32	64.3	-	-	0.7
A26	3	32	64.2	-	-	0.8
A27	3	32	64.1	-	-	0.9

3.2. Potentiometric performance properties of the sy-selective electrode

3.2.1. Determination of the slope, limit of quantification and linear range of the sunset-yellow-selective electrode

For detecting the potentiometric performance characteristics of the developed electrodes, 5.0×10^{-2} M – 1.0×10^{-6} M sunset yellow solutions were prepared in deionized water. Slope, linear range, and R^2 values of each electrode from the calibration graphs obtained from the series of the Sunset yellow solution were calculated. The results were given in Table 2.

Table 2. Potentiometric performance characteristics of the electrodes prepared using SY-MTOA ion pair as ionophore in the sunset yellow solutions prepared in deionized water

Electrod	Slope, mV/decade concentration change	Linear range, M	R^2	Limit of quantification, M
A1	13.5	5.0×10^{-2}-1.0×10^{-4}	0.8998	1.0×10^{-4}
A2	22.9	5.0×10^{-2}-1.0×10^{-5}	0.9840	1.0×10^{-5}
A3	10.8	5.0×10^{-2}-1.0×10^{-6}	0.9623	1.0×10^{-6}
A4	18.2	5.0×10^{-2}-1.0×10^{-4}	0.9880	1.0×10^{-4}
A5	29.6	5.0×10^{-2}-1.0×10^{-4}	0.9725	1.0×10^{-4}
A6	29.1	1.0×10^{-2}-1.0×10^{-4}	0.9639	1.0×10^{-4}
A7	26.9	1.0×10^{-2}-1.0×10^{-5}	0.9465	1.0×10^{-5}
A8	30.8	5.0×10^{-2}-1.0×10^{-4}	0.9912	1.0×10^{-4}
A9	25.1	1.0×10^{-2}-1.0×10^{-4}	0.9766	1.0×10^{-4}
A10	21.2	1.0×10^{-2}-1.0×10^{-5}	0.9401	1.0×10^{-5}
A11	27.7	1.0×10^{-2}-1.0×10^{-4}	0.9696	1.0×10^{-4}
A12	21.2	1.0×10^{-2}-1.0×10^{-4}	0.9274	5.0×10^{-5}
A13	23.4	5.0×10^{-2}-1.0×10^{-4}	0.9522	5.0×10^{-5}
A14	22.9	5.0×10^{-2}-1.0×10^{-5}	0.9903	5.0×10^{-5}
A15	31.6	1.0×10^{-2}-1.0×10^{-4}	0.9756	1.0×10^{-4}
A16	30.3	1.0×10^{-2}-1.0×10^{-4}	0.9831	1.0×10^{-4}
A17	31.9	1.0×10^{-2}-1.0×10^{-5}	0.9832	1.0×10^{-5}
A18	33.3	1.0×10^{-2}-1.0×10^{-4}	0.9707	1.0×10^{-4}
A19	27.4	1.0×10^{-2}-1.0×10^{-5}	0.9870	1.0×10^{-5}
A20	28.5	5.0×10^{-2}-1.0×10^{-5}	0.9945	1.0×10^{-5}
A21	31.6	5.0×10^{-2}-1.0×10^{-5}	0.9862	1.0×10^{-5}

A22	29.0	5.0×10^{-2}-1.0×10^{-5}	0.9632	1.0×10^{-5}
A23	47.7	5.0×10^{-2}-1.0×10^{-5}	0.9974	1.0×10^{-5}
A24	26.6	1.0×10^{-2}-1.0×10^{-5}	0.9819	1.0×10^{-5}
A25	32.5	1.0×10^{-2}-1.0×10^{-5}	0.9624	1.0×10^{-5}
A26	22.5	5.0×10^{-2}-1.0×10^{-5}	0.9882	1.0×10^{-5}
A27	28.0	1.0×10^{-2}-1.0×10^{-5}	0.9919	1.0×10^{-5}

As can be seen from the Table 2, the electrodes A20, A21, and A22 displayed the best potentiometric performance characteristics regarding slope, linear range, limit of quantification and R^2 values.

3.2.2. Potentiometric performance of the sunset yellow selective electrode in tris buffer

Using the sunset yellow selective electrodes A20, A21, and A22, performing better analytical characteristics, the potentiometric performance characteristics were reevaluated in the 5.0×10^{-2}-1.0×10^{-6} M Sunset yellow solutions, which were prepared in pH = 7.4 Tris / Tris HCl buffer. The obtained potentiometric performance characteristics were given in Table 3 in follow.

Table 3. Potentiometric performance characteristics of the sunset yellow selective electrodes A20, A21, and A22.

Electrode no.	Slope, mV	Linear Range, M	R^2	Limit of quantification, M
A20	23.6	5.0×10^{-2}-1.0×10^{-5}	0.9938	1.0×10^{-5}
A21	22.1	5.0×10^{-2}-1.0×10^{-5}	0.9864	1.0×10^{-5}
A22	22.3	5.0×10^{-2}-1.0×10^{-5}	0.9886	1.0×10^{-5}

When the obtained data given in Table 3 are evaluated it is seen that A20 electrode has the best potentiometric performances characteristics in the sunset yellow solutions, which were prepared in Tris buffer.However, in the buffered sun set yellow solutions,it was seen that the slope of the electrode response became narrover compared to the that of the prepared aqueous solutions.

The potential values obtained for the 5.0×10^{-2} M, 1.0×10^{-2} M, 5.0×10^{-3} M, 1.0×10^{-3} M, 5.0×10^{-4} M, 1.0×10^{-4} M, 5.0×10^{-5} M, 1.0×10^{-5} M sunset yellow solutions in the pH = 7.4 Tris / Tris HCl buffer using the sunset yellow selective electrode formed with the A20 membrane composition and the relevant calibration graph were presented in Figure 3 and Figure 4, respectively.

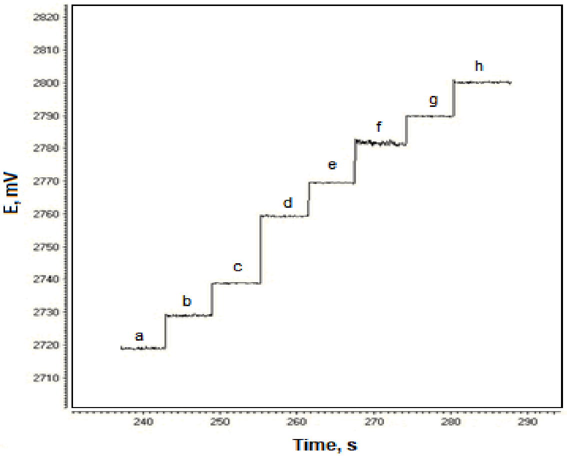

Figure 3. Potentiometric behavior of the sunset yellow selective electrode in (a) 5.0×10^{-2} M, (b)1.0×10^{-2} M, (c)5.0×10^{-3} M, (d) 1.0×10^{-3} M, (e) 5.0×10^{-4} M, (f) 1.0×10^{-4} M, (g) 5.0×10^{-5} M, (h) 1.0×10^{-5} M sunset yellow solutions in pH 7.4 Tris buffer.

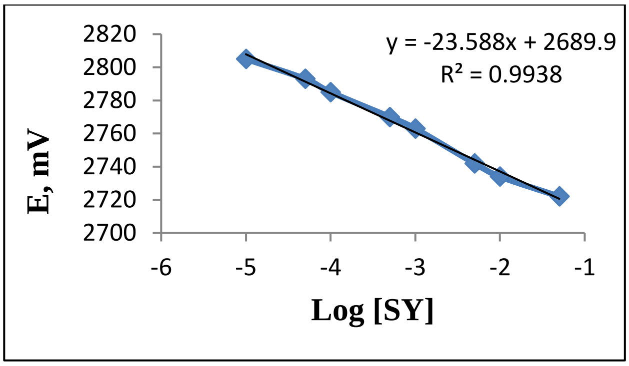

Figure 4. Calibration curve of the sunset yellow selective electrode in 5.0×10^{-2} M, 1.0×10^{-2} M, 1.0×10^{-3} M, 1.0×10^{-4} M and 1.0×10^{-5} M sunset yellow solutions in pH 7.4 Tris buffer.

3.2.3. Detecting the response time of the sunset yellow selective electrode

For determining the response time of the electrode, the sunset yellow electrode A20 was used in the sunset yellow solutions, which were prepared in 10^{-3} M and 10^{-4} M pH 7.4 Tris buffer, and while the solution was being mixed at a fixed speed, the times needed for the potential to become stable were written down (t_{95}). The mean time during which the potentials became stable was decided as the response time of the electrode. The equilibration time of the electrode in the 10^{-4} M and 10^{-3} M sunset yellow solutions is seen in Figure 5. The mean response time of the electrode was found to be about 5 sec.

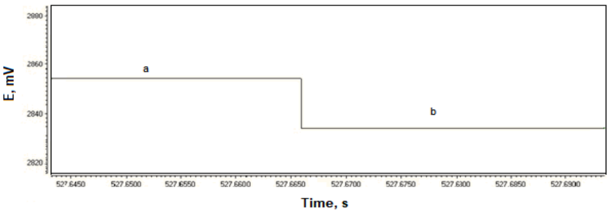

Figure 5. Response time graph of the A-20 sunset yellow-selective electrode for (a)10^{-4} M and (b) 10^{-3} M sunset yellow solutions.

3.2.4. Detecting the response time of the sunset yellow selective electrode

Measurements of A-20 electrode were taken consecutively in 10^{-4} M, 10^{-3} M, and 10^{-2} M sunset yellow solutions, which were prepared in pH 7.4 Tris buffer, to calculate the repeatability of sunset yellow-selective electrode. Potential measurements and repeatability graph for the electrode A-20 were presented in Figure 6 and Table 4, respectively.

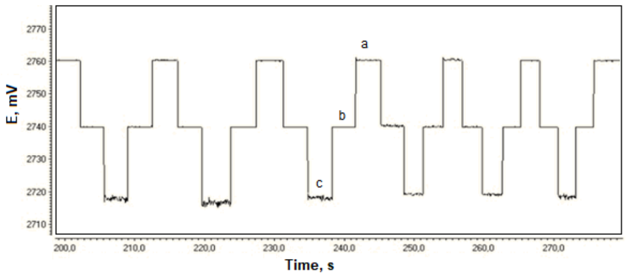

Figure 6. Repeatability of the A-20 SY-selective electrode in (a) 10^{-4} M, (b) 10^{-3} M and(c) 10^{-2} M SYsolutions.

Table 4. Repeatability results of the A-20 electrode inA-20 electrode in 10^{-2} M, 10^{-3} M and 10^{-4} M sunset yellow solutions

Sunset yellow Concentration (M)	Potential, mV					$X^a \pm S^b$
10^{-2}	2732	2732	2733	2733	2733	2732± 0.5
10^{-3}	2752	2752	2752	2752	2752	2752 ± 0
10^{-4}	2773	2773	2773	2773	2773	2773 ± 0

[a] Mean potential values for 5 measurements.
[b] Standard deviations

3.2.5. Detecting the selectivity of the sy-selective electrode

To find out the effects of ions that can interfere on the response of the sunset yellow-selective electrode, the separate solution method was employed to calculate the selectivity coefficients for these types(E_A=E_B). The genaral potantiometric responses of the electrode to sunset yellow and the studied foreign ions were given in Figure 7. In calculating selectivity coefficients, sunset yellow concentrations corresponding to the potential values measured in 1.0×10^{-2} M solutions of interfering ions were detected by using calibration curve. Concentration value, which corresponded to the potential value of the interfering ion for 1.0×10^{-2} M concentration in the calibration curve that was obtained for sunset yellow, was placed in the selectivity coefficient equation and the selectivity coefficients of the electrode were determined for each interfering anion. When the selectivity coefficients were evaluated, it was seen that the electrode was very selective for the measured types. The result was given in Table 5.

Table 5. Selectivity coefficients of the sunset yellow-selective electrode for some anions

Ion	-log $K_{SY,B}$
ClO_3^-	2.95
I^-	3.47
CO_3^{2-}	1.91
SCN^-	5.22
PO_4^{3-}	0.81

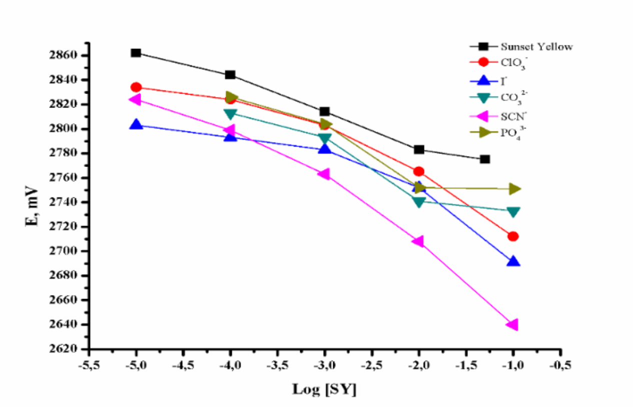

Figure 7. Potentiometric behavior of the sunset yellow-selective electrode for sunset yellow and some interfering types.

3.2.6. Detecting the ph range of the sunset yellow-selective electrode

To determine the pH range of the Sunset yellow-selective electrode, 10^{-3} M sunset yellow solution, which was prepared in 50 mL deionized water, was stirred at a fixed speed. The reference electrode, A-20 sunset yellow-selective electrode, and pH electrode were simultaneously submerged into the solution. 10^{-2} M HCl and 10^{-2} M NaOH were slowly added into the sunset yellow solution and each time acid and base were added into the solution, the pH value of the solution and its potential values that were measured were plotted on the graph and the obtained graph was given in Figure 8. As one can see in the graph, the potential values that were taken from the ion-selective electrode system did not vary significantly in the range of pH=6.4-9.1. This indicates that the hydronium ion did not impact the potentiometric response of the electrode in the range of pH=6.4-9.1, but the electrode potentials start to decline rapidly in the increasing pH values.

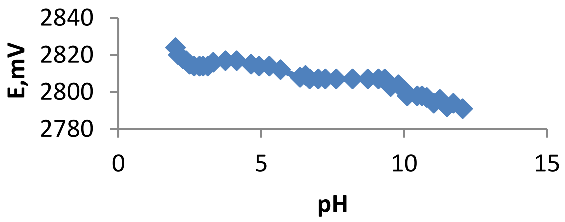

Figure 8. pH range of the SY-selective electrode.

3.2.7. Detecting the lifetime of the sunset yellow selective electrode

To calculate the lifetime of the SY-selective electrode, measurements were performed with the sunset yellow-selective electrode every day in the concentration range of 1.0×10^{-5}-5.0×10^{-2} M, which is the linear range of the electrode. The electrode was conditioned in the sunset yellow solution, which was prepared in1.0×10^{-2} M pH 7.4 Tris buffer, before the measurements for half an hour each time. It was stored under room conditions and in a dark and closed medium when not in use. The slope values obtained from the calibration plots and the correspondence values were plotted against week of the measurements (Figure 9). The slope values of the electrode depending on weeks remained nearly unchanged until the ninth week. After this time the slope values started to decrease. Therefore the lifetime of the electrode can be accepted as the period of nine weeks.

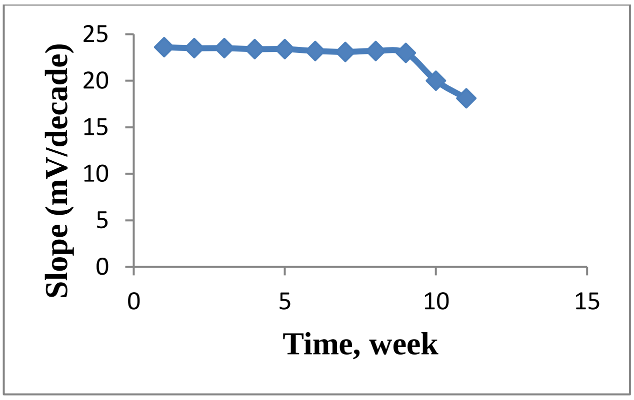

Figure 9. The graph depicting the lifetime of the sunset yellow-selective electrode

3.3. Electroanalytical application of the sunset yellow selective electrode

5 candies, purchased from a market, were weighed and crushed. Then, 2.348 g, which is the mean weight, were taken, stirred and solved in 10 mL pH 7.4 Tris buffer, and its sample analysis was carried out by UV spectroscopic method at 482 nm and Potentiometric method. The result was given in Table 6.

Table 6. Sample analysis result of the sunset yellow-selective electrode by UV and Potentiometric method (n=5)

Sample	UV (mg/mL)	Potentiometry (mg/mL)	P
Candy	0.01312±0.00008	0.01316±0.00010	0.545

The results were given in mean±standard deviation. The difference between the mean values of the methods was found to be statistically insignificant as the obtained 0.545 value, the two-sided t statistical value with a degree of freedom of 8, is greater than 0.05, the P-value accepted to be within 95% confidence limits (P>0.05).

4. Conclusions

The developed HPLC method is simple, fast, reliable and validated for simultaneous determination of SCP and TMP at first time. This method has a well resolution between SCP and TMP moreover the analysis time is very short. The proposed chromatographic methods accuracy, precision and the kimit of detection values are particularly satisfactory and comparable with more other analytic protocols. Thus developed method could be suggested for quality control analysis of and determination of veterinary formulation which are containing SCP and TMP.

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Conflicts of interest

The authors state that did not have conflict of interests.

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