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Ionic Associates of Fuschine Basic Dye as Sensing Probe for Potentiometric Determination of 2,4-Dichlorophenoxy- and 4-Chlorophenoxy Acetic Acids

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Ionic associates of basic fuchsin have been used as an electrochemical sensing probe for the potentiometric determination of 2,4-dichlorophenoxyacetic acid (2,4-D) and 4-chlorophenoxyacetic acid (4-CPA). The influence of the membrane composition on the electroanalytical characteristics of the electrodes has been established. The effect of the modifying additive (N,N,N',N'-tetramethylethylenediamine) on the chemical and analytical characteristics of the electrodes is shown. The sensors can be used for the determination of 2,4-D (4-CPA) in an analytical range of 1×10^{-4} to 3×10^{-2} M with LOD as 10^{-5} M. For 2,4-D ISE, a hyper-Nernstian function was observed. The sensors have been applied for the determination of 4-CPA and 2,4-D in model solutions of pesticide preparations.

Keywords: 2,4-Dichlorophenoxyacetic acid, 4-Chlorophenoxyacetic acid, Potentiometry, Ion-selective electrodes, Modifying additives

INTRODUCTION

2,4-Dichlorophenoxyacetic and 4-chlorophenoxyacetic acids (Fig. 1) possess similar structures and are applied in agriculture as pesticides. 2,4-Dichlorophenoxyacetic acid (2,4-D, $C_8H_6Cl_2O_3$) is one of the most known pesticides, used to protect crops [1]. It is used as an amine salt or as an ester emulsion, while 4-chlorophenoxyacetic acid (4-CPA, $C_8H_7ClO_3$) is a phytohormone, being a plant growth regulator.

Both of these substances possess toxic action on human and mammal organisms [4-6]. Taking into account that they are widely used, and as their action is dose-related, the development of new methods of their determination is highly desirable.

2,4-D is mostly determined by chromatographic analysis. The liquid chromatographic [7-9], HPLC [10-15]
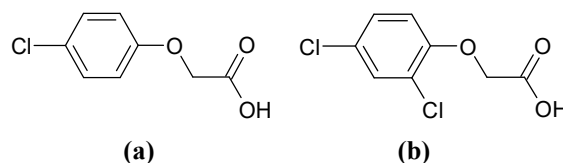

Fig. 1. Structure formulae of 4-CPA (a) and 2,4-D (b) M.P (2,4-D) = 141 °C, B.P. = 160 °C, $pK_a = 2{,}64$, $C_d = 2{,}3 \times 10^{-4}$ [2]. M.P. (4-CPA) = 160 °C, $C_d = 9{,}6 \times 10^{-4}$ [2], $pK_a = 3{,}1$ [3].

and gas chromatographic [7,16-22] methods are sensitive and determine nanomolar quantities of 2,4-D and 4-CPA by pre-concentration. Also, they are very expensive and employ toxic substances as the mobile phase. Also, spectrophotometric methodologies have been developed for both the substances at acidic pH = 1-6 [23]. Nevertheless, in these conditions, the 2,4-D is rapidly decomposed by acidic hydrolysis, which has to be taken into account. Also, the voltammetric [24-27], cryoscopic [28], immunoenzymatic

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[6,30] and Raman spectroscopic [31] methods are used to determine both of the analytes. No potentiometric sensors for 2,4-D and 4-CPA have been reported by far. We have already shown that the ionic associates (IA) of the basic cationic dyes are efficient electroactive substances (EAS) for plastified potentiometric sensor membranes for organic anions´ determination [32-37].

In this work, novel ion selective electrodes (ISE) with the modified ion-sensitive membranes, based on ionic associates of fuchsine basic (FB) dye has been developed for the determination of 2,4-D and 4-CPA. Influence of the modifying lyophylic additive N,N,N´,N´-tetramethylenediamine on the basic sensor characteristics has also been studied. The sensors have been applied for the determination of 4-CPA and 2,4-D in model solutions in pesticide preparations.
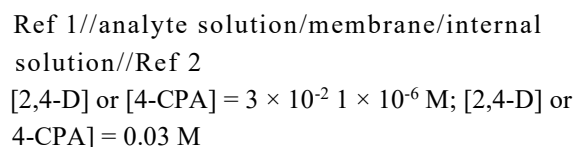
EXPERIMENTAL

The standard (0.01 M) solutions of 2,4-D and 4-CPA were prepared by dissolving each compound separately in 5 ml of 0.2 M NaOH solution in distilled water. The pH of the solution was regulated by a universal buffer acid mixture till pH = 8. The fuchsine basic (FB) solution (0.01 M) was prepared by dissolving an exact mass of the dye salt in a small quantity of ethanol, diluting it to the mark in distilled water.

The ionic associates (IA) were obtained by the slow stirring of solutions of 2,4-D/4-CPA (0.02 M) and the FB dye in a 1:1 ratio. The mixture was kept for 8-10 h at room temperature. The insoluble salt was collected on filter paper, rinsed by cold distilled water and dried at room temperature. The plastified PVC membranes were prepared according to the recommendations [27]. The exact quantities of the IA were mixed with PVC (0.075 g). A mixture of 0.5 ml of THF solvent and 0,15 ml of the each of the plasticizers (dibutylphthalate (DBP), dibutylsebacynate (DBS), dinonylphthalate (DNP), dioctylphthalate (DOP) and tricresylphthalate (TCP)) was stirred vigorously in separate containers to obtain homogenous mass with each plasticizer. The mixture was put onto a glass plate (of a diameter of 1.5-2 cm) and dried in air for 3-5 h. From the films, a disk with the diameter of 0.5-0.6 cm was cut and attached to the end of the PVD tube. After complete drying of the film, the electrode was filled ¾ with standard 2,4-D or 4-CPA solution with a copper wire therein.

The potentiometric measurements were carried out on the ionometer AI-123 (MLost Instruments, Ukraine), the measurements deviations is not more than ± 0.2 mV/pC. The reference electrodes (Ref 1 and Ref 2) were Ag/AgCl, Cl$^-$ electrodes EVL-1M3 or ESR-10103 at RT. The solution pH was controlled using an ionometer AI-123 or I-160 M with the glass electrode. The medium acidity was maintained by buffer mixtures (containing 0.04 M CH_3COOH, H_3BO_3, H_3PO_4 and/or 0.2 M NaOH solutions). The measurements were carried out by the classical electrochemical cell scheme:

Ref 1//analyte solution/membrane/internal solution//Ref 2
[2,4-D] or [4-CPA] = 3×10^{-2} 1×10^{-6} M; [2,4-D] or 4-CPA] = 0.03 M

The IR spectra were taken on IR spectrometer Nicolet iS10 with a continuum microscope in the wavelength range of 4000-650 cm^{-1}.

RESULTS AND DISCUSSION

Schematically, the ion associates' formation scheme may be depicted as:

$$An^- + Ct^+ \leftrightarrow An^-Ct^+\downarrow$$
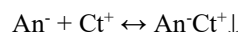
Herein, An$^-$ stands for the organic anion of 2,4-D or 4-CPA, Ct$^+$ stands for FB dye cation.

The IA formation of FB with the analyte acids has been detected by IR-spectroscopy methods (Fig. 2). IR spectra of the associates manifest the presence of the characteristic absorbance bands of the phenoxyacetic acids at 1736 cm^{-1}, caused by the carbonyl double bond oscillations. Yet, the band at 756 cm^{-1} confirms the presence of the chlorine atoms in the IA. The IA spectra also show the bands at 3320-3200 cm^{-1} and 1638-1546 cm^{-1}, characteristic for ν and δ (N-H) correspondently, like also the absorbance peak at 1609 cm^{-1}, corresponding to the Schiff base bond of the FB dye. The IR spectra of IA show the changes in the shape of the peak, like also a slight shift of the absorbance bands'
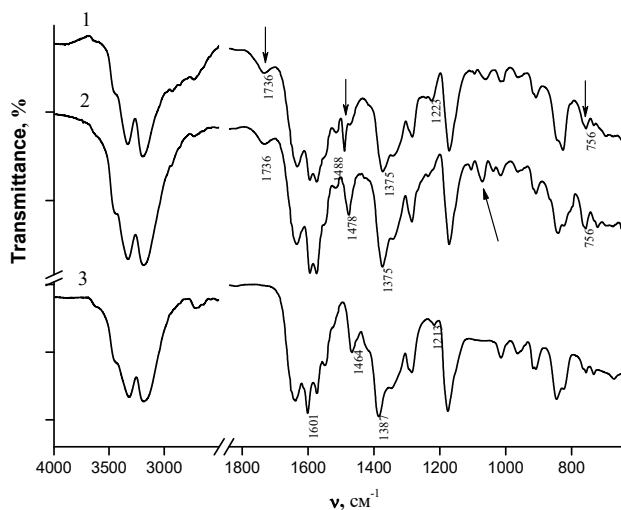
Fig. 2. The IR spectra for IA of 4-CPA (1), 2,4-D (2) with FB (3).

frequency, while compared with the spectra of the proper analytes and FB dye.

The IA, obtained by the salt deposition, was used as an electroactive substance (EAS) in ion-selective electrodes (ISE) composition. The dependence of the electroanalytical ISE properties on membrane composition was investigated by the performance of the uniform membranes with the constant compositions of nearly all the components, except for the investigated one.

It is known that the electrode response to the corresponding ion may be influenced by the membrane composition, like the addition of the different ionogenic additives [38-42]. In order to investigate the effect of IA and lipophilic additive tetramethylethylenediamine (TMEDA) on the electrochemical electrode parameters, the membranes with IA content (by mass) of 3%, 5%, 7%, 9%, 11% and 4N-TMEDA content 0.04, 0.06, 0.08 and 0.12 ml have been conducted. The results show that the TMEDA addition in the 4-CPA-selective membrane lowers the detection limit with the enhancement of the slope of the electrode function (Fig. 3). In the case of the 2,4-D-selective electrode, the addition of the lipophilic TMEDFA strongly augments the electroanalytical membrane properties.

It is known that the electrode response is highly influenced by membrane plasticizer nature [32-42]. The commonly used plasticizers are DBP, DOP, DBP, DBS and
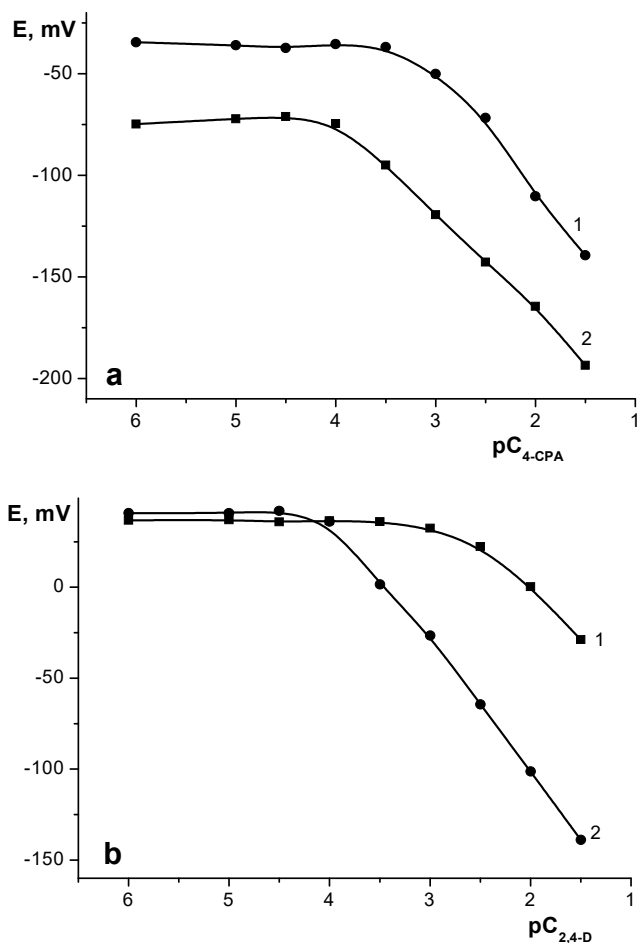

Fig. 3. The membrane composition dependence on the (a) 4-CPA and (b) 2,4-D ISE (electroanalytical properties; 1-classical membrane; 2-TMEDA-containing membrane).

TCP. As shown in Fig. 4, the TCP-plasticized membranes possess better electroanalytical properties with an electrode function slope of 71.3 ± 0.1 мВ/pC and DBP-plasticized membranes with the electrode function slope of 47.7 ± 0.1 мВ/pC for 2,4-D and 4-CPA, correspondently.

Table 1 contains the electroanalytical properties of IA-based membranes with $(FB^+)(2,4-D^-)$ and $(FB^+)(4-CPA^-)$, with different plasticizer contents. All the membranes possess the electrode function slope, characteristic of the one-charged ions with a low detection limit (nearly 10^{-4}, 10^{-5} M) of analytes. Yet for 2,4-D ISE, a hyper-Nernstian function is observed.

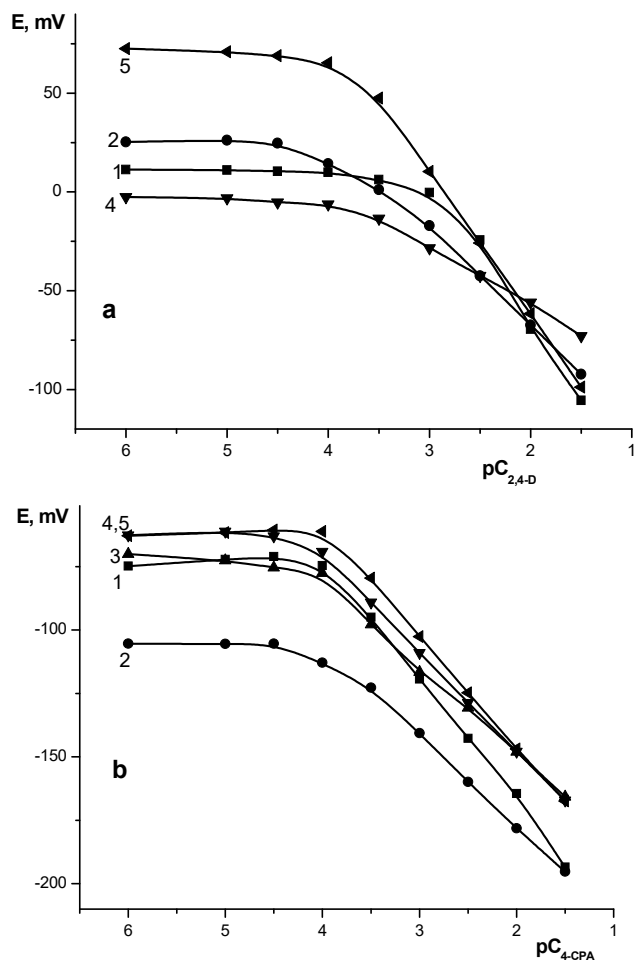2,4-D ISE works in pH interval of $6 < pH < 10$ while the 4-CPA ISE works in the pH range $5 < pH < 12$. The stable

Fig. 4. The plastifier nature influence on the ISE response. Plasticizer: 1-DBP, 2-DBS, 3-DOP, 4-DNP, 5-TCP. a) IA (FB$^+$) (2,4-D$^-$): 5% IA, 65% plasticizer, 0.06 ml TMEDA, pH 8,0; b) IA (FB$^+$) (4-CPA$^-$): 5% IA, 65% plasticizer, 0.06 ml TMEDA, pH 8,0.

potential values are established by 30-40 s and 20-25 s for 2,4-D and 4-CPA, correspondently. Performances of the synthetsized membranes are stable for up to 4 months. The selectivity of the interfering ions is an important property of the selective electrodes. The selectivity of the electrode to an interfering ion is shown by the selectivity coefficient, calculated by the Nikolski-Eisenmann equation [42]:

$$\Delta\varphi_{g1} = \Delta\varphi_{g1}' - \frac{RT}{z_m F}\ln\left(a_m + \sum_{a_s} K_{m-s} a_s^{z_m/z_s}\right)$$

By testing the 2,4-D- and 4-CPA-ISE for selectivity coefficients determination, we used the method of the separate solutions [42]. In order to do this, we measured the dependence of the electrochemical system potential on the interfering ion concentration and compared the values of the detected (a_A) and interfering (a_B) anion concentrations, which compose the main system potential:

$$K_{A/B} = \frac{a_A}{a_B}$$

The selectivity coefficients for the classical membrane and membrane with lipophilic additive are presented in Table 2. In order to check whether the proposed ISE is ready for the real sample analysis, the quantitative content of 2,4-D in plant protective systems and of 4-CPA in model preparations was found by the method of "added and found".

Liquid 2,4-D - sensitive sensors based on tetrazolium ionic associates in nitrobenzene have been described in [43]. Table 3 shows the comparative characteristics described in

Table 1. Membrane Characteristics of 2,4-D and 4-CPA Based ISE

Plasticizer content (%) (by mass%)	2,4-D-ISE, TCP, TMEDA			4-CPA-ISE, DBP, TMEDA		
	S (мV/pC)	a (M)	C_{min} (M)	S (мV/pC)	a (M)	C_{min} (M)
35	-	-	-	33.0	1×10^{-4}-3×10^{-2}	1.0×10^{-4}
45	80.2	3×10^{-4}-3×10^{-2}	2.5×10^{-4}	33.5	1×10^{-3}-3×10^{-2}	6.2×10^{-4}
55	97.0	1×10^{-4}-3×10^{-2}	1.0×10^{-4}	40.2	1×10^{-3}-3×10^{-2}	5.9×10^{-4}
65	71.3	1×10^{-4}-3×10^{-2}	9.5×10^{-5}	47.7	1×10^{-4}-3×10^{-2}	8.0×10^{-5}
75	75.4	1×10^{-3}-3×10^{-2}	7.0×10^{-4}	38.4	3×10^{-4}-3×10^{-2}	1.7×10^{-4}

C_{min}-detection limit; S-electrode function slope; a-linear measurement interval.

Table 2. Selectivity Coefficients for the Membranes of 2,4-D and 4-CPA ISE

Interfering ion	Classical membranes		Membranes with lipophilic additive	
	2,4-D	4-CPA	2,4-D	4-CPA
I^-	>5	1.2	1.4	2.7
$B_4O_7^{2-}$	>5	1.4	4.0	2.0
F^-	>5	>5	>5	>5
Br^-	>5	1.4	3.0	>5
Cl^-	>5	>5	>5	>5
$C_6H_5COO^-$	>5	1.8	3.8	1.7
SCN^-	1.1	0.8	0.8	1.1
2,4-D	-	0.8	-	>5
4-CPA	1.4	-	1.2	-
1-ONA[a]	1.1	0.8	1.1	4.0
IAC[b]	0.9	0.9	1.0	1.1
PO_4^{3-}	>5	2.0	2.3	>5
NO_3^-	2.4	1.1	1.2	1.6

[a]1-ONA-1-oxynaphthoate. [b]IAC-3-indolyleacetate.

Table 3. The Comparative Characteristics of 2,4-D-Sensitive Sensors

Characteristic	This work	[43]
Ease of use	+	-
Potential stability after contact with a foreign ion solution	+	-
S (мV/pC)	71.3	57
a (M)	1×10^{-4}-3×10^{-2}	1×10^{-4}-1×10^{-2}
C_{min} (M)	9.5×10^{-5}	-
pH	6-10	6-10
	Selectivity coefficients	
I^-	>5	-
$B_4O_7^{2-}$	>5	-
F^-	>5	-
Br^-	>5	-
Cl^-	>5	3
$C_6H_5COO^-$	>5	~2
SCN^-	1.1	-
4-CPA	1.4	-
1-ONA[a]	1.1	-
IAC[b]	0.9	~2
PO_4^{3-}	>5	-
NO_3^-	2.4	-

LOD-detection limit; *S*-electrode function slope; *a*-linear measurement interval; [a]1-ONA-1-oxynaphthoate; [b]IAC-3-indolyleacetate.

[43], and we developed 2,4-D-sensitive sensors. As you can see, the developed sensor has better performance and better selectivity. The advantages of the developed sensor are obvious. The scheme of formation of the electrode potential of the electrodes described in [43] and developed by us have both similar and different factors. In liquid electrodes, the electrode potential is determined by the extraction behavior of the ionophore at the phase boundary and its solubility and diffusion in the organic phase. Instead, plasticized membrane electrodes are quasi-liquids. They are characterized by better permselectivity. The characteristic of the surface at the solution-membrane boundary and the properties of the ionophore are decisive for the emergence of the electrode potential. The permselectivity of membranes is determined by parameters such as charge density, degree of delocalization of the electron density and the polarizing ability. In tetrazoles (ionophore proposed in [43]) the positive charge is concentrated in the tetrazolium cycle. They have a relatively high charge density and low

polarizability. In contrast, the cations of basic dyes (this work) are characterized by significant delocalization of the positive charge [44-45] and polarizability. This may explain the better selectivity of the developed sensors.

Methodology for 2,4-D and 4-CPA Determination

Solutions of the pesticide formulations for analysis were prepared by the following manner: 1.0 ml of the herbicide is put into a 100 ml beaker and diluted in distilled water to the mark, stabilizing the pH with universal buffer at 8.0 ± 0.5. Then, 1 ml of the ester (plasticizer) is hydrolyzed by 10 M NaOH till the complete dissolution. Then, standard actions (see experimental part) are realized. The solution is put into a container glass, electrodes are immersed and the potential is recorded. The quantity of the analyte is found by the calibration curve, calculated in the analogous conditions. The results of the 2,4-D in the herbicide formulations and of the 4-CPA in model solutions are shown in Tables 4, 5. As shown in the Tables 3 and 4, the membranes modified

Table 4. Determination of the 2,4-D Content in Herbicide Preparations ($n = 5$, $P = 0.95$) Using the Proposed Method

Preparation (Country)	The regulated content 2,4-D (g l^{-1})	Found 2,4-D (g l^{-1})	RSD (S$_r$, %)
«Khlebodar», (China), emulsion	395.5	382.8 ± 4.6	0.55
	395.5[a]	392.6 ± 3.5	0.22
«Dialen Super» (Switzerland), solution	319.4	322.7 ± 4.0	0.49
	319.4[a]	315.1 ± 3.2	0.30
«Prima» (Austria), emulsion	199.1	194.2 ± 4.2	0.89
	199.1[a]	196.7 ± 3.5	0.54

[a]-TMEDA-containing membranes.

Table 5. Determination of the 4-CPA Content in Herbicide Preparations ($n = 5$, $P = 0.95$) Using the Proposed Method

Type of the ISE-membrane	Added 4-CPA (mg)	Found 4-CPA (mg)	RSD (%)
1	50.0	48.6 ± 1.2	1.0
2	50.0	50.2 ± 1.0	0.9
1	75.0	73.4 ± 3.3	1.8
2	75.0	74.8 ± 1.1	0.6
1	100.0	100.2 ± 2.3	1.2
2	100.0	100.1 ± 0.9	0.4

1-Membranes without TMEDA; 2-TMEDA-containing membranes.

with lipophilic additives show better reproducible results. Therefore, they are more efficient.

CONCLUSIONS

From the experimental details and real sample testing of the ion-selective 2,4-dichlorophenoxy- and 4-chlorophenoxyacetic acid electrode it was possible to conclude that:

- The associate ion formation of Fuchsine Basic Dye is realized in an efficient manner. The spectral data confirm the presence of both dye and analyte acid ion in the ion-associate forms, as seen from the shift of absorption bands of the imine group in the range 3300 cm^{-1} in the complexed receptor.

- The electrode composition was optimized for better efficiency with the lypophylic basic additive (4N of TMDA) is used in the range of 0.04 ml to 0.12 ml and the IA content is varied in the range 4% - 11% (by mass) for electrode production. Its addition has significant importance for electrode response.

- The ion-selective electrode may be efficiently used in either model or real samples, providing a low detection limit of 10^{-5} M.

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