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Ionophore-based potentiometric PVC membrane sensors for the determination of moxifloxacin

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Abstract

The construction and development of polyvinyl chloride (PVC) membrane sensors for the determination of moxifloxacin (MOX) were studied. Three different membrane sensors were created by incorporating 4-tert-butylcalix[8]arene (sensor 1), β-cyclodextrin (sensor 2)and γ-cyclodextrin (sensor 3) as ionophores. In a PVC matrix, o-nitrophenyl octyl ether (o-NPOE) was used as a plasticizer and potassium tetrakis (4-chlorophenyl) borate (KTPClPB) as an ion additive. The construction of reaction mechanisms has been facilitated by the formation of supramolecular inclusion complexes between drug and ionophores. The calixarene, β-CD, and γ-CD sensors exhibited a response to moxifloxacin that closely followed the Nernstian behavior within the pH range of 3 to 8. The proposed sensors exhibited a calibration range for MOX from 1×10-2 - 4.3 ×10-6, 1×10-2 - 3.3×10-6 and 1×10-2 - 3.4×10-6, with slope 54, 55 and 56 mV decade−1 and the detection limits were 1.3 ×10-6, 1×10-6, and 1.03×10-6 for sensors 1, 2, and 3, respectively. The interference study of the investigated method was examined, and the low values of the selectivity coefficient indicate that the sensors showed high selectivity for MOX. The developed sensors exhibited favorable relative standard deviation and high recovery for MOX. The sensors were successively used for the evaluation of MOX in a pharmaceutical formulation and spiking urine samples. The results obtained by the sensors are strongly compatible with those of the reported methods. On the other hand, the advanced sensors are used as indicator electrodes for the titration of MOX with NaTPB potentiometrically.

Keywords: Moxifloxacin; Ionophore; Potentiometry; PVC membrane sensors; Quality control

1. Introduction

A new drug called moxifloxacin (MOX), which is an 8-methoxy quinolone derivative of fluoroquinolone, has been shown to kill a lot of different kinds of bacteria (Fig. 1). It has demonstrated encouraging antimycobacterial action and may be able to reduce the amount of time patients must get tuberculosis treatment [1]. Early studies of moxifloxacin's bactericidal activity showed that it can kill persistent bacilli that replicate slowly in tissues. This is regarded as an important characteristic to shorten tuberculosis treatment [2, 3]. In an effort to reduce the length of tuberculosis treatment, controlled clinical trials combining moxifloxacin with first-line anti-tuberculosis medications are being conducted in patients with pulmonary tuberculosis [4]. So, keeping an eye on moxifloxacin levels may help researchers learn more about its pharmacokinetics and how it interacts with other anti-tuberculosis drugs when it is given to them.

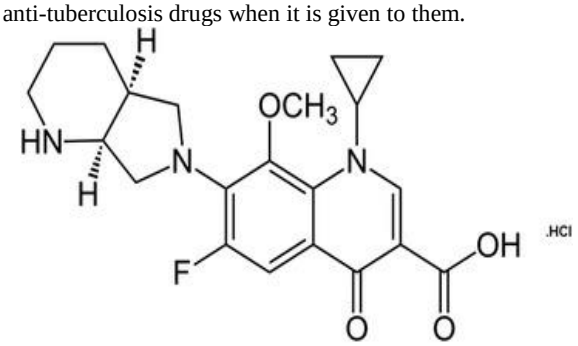

Fig. 1. Chemical structure of moxifloxacin HCl.

Several analytical methods were used for the monitoring and determination of MOX, including spectrophotometry [5], HPLC -mass spectrometry [6], HPLC-fluorescence [7], HPLC-UV [8,9], spectrofluorimetric [10], voltammetry [11], capillary electrophoresis [12], and potentiometry [13]. These technologies have high maintenance and running costs despite their outstanding sensitivity, wide linearity range, and strong selectivity. This is not appropriate for routine analysis of a large number of pharmaceutical samples. As a result, a trustworthy, affordable, rapid, and portable analytical technique is needed.

Potentiometric sensors with PVC membranes have a multitude of benefits, including speed, ease of use, sensitivity, accuracy, precision, affordability, portability, and a broad range of applications pertaining to various analytical areas [14, 15]. On the other hand, ionophore-based ion-selective electrodes have much better selectivity, detection limit, and long life time compared to ion-pair or ion-exchange-based sensors. The proposed method involves utilizing an ionophore as the electroactive material, in contrast to the previously described technique [13], which employed an inclusion complex or host-guest interaction as the underlying mechanism of the reaction. In comparison to the ion-pair approach [13], which relies on solubility, this method exhibits greater selectivity and sensitivity. Calixarenes are cyclic oligomers made up of phenol units connected by alkylidene groups. They have a cavity shape. The formation of host-guest complexes with different host

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compounds occurs through different factors, such as cavity size, conformation, and substituents, which are used in different applications in ion-selective membrane sensors [15, 16].

Cyclodextrins have the ability to establish stable host-guest complexes, known as inclusion complexes, with diverse guests, including both organic and inorganic molecules [17, 18]. Significant families of organic compounds with enormous cavities that are exploited in electrochemical research include cyclodextrins. Developing PVC membrane sensors, for instance, by utilizing a huge compound cavity, a recent area of study for electrochemical sensors, particularly in drug analysis, is cyclodextrin-modified electrodes [17, 18]. Non-covalent interactions, such as electrostatic interactions, van der Waals forces, hydrogen bonds, and dipole-dipole interactions, which may be implicated in host-guest binding, provide the basis for the formation of inclusion complexes between drugs (guest) and CD (host) [19].

The objective of this work was to create new, selective sensors for MOX measurement using β- and γ-modified CD and calixarene. When different ionophores were mixed with a PVC matrix and an ionic additive known as potassium tetrakis (4-chlorophenyl) borate, the three sensors were made. These new MOX sensors, designated Sensors 1, 2, and 3, were constructed with calixarene, β-CD, and γ-CD. The developed sensors were reported for the measurement of MOX in both its dose form and in bulk.

2. Experimental

2.1. Reagents and materials

Each and every reagent was of analytical grade. As needed, double-distilled water was utilized in every experiment. Aldrich (Steinheim, Germany) provided MOX-HCl (Molecular weight 437.9) , dioctyl phthalate (DOP), nitrophenyl octyl ether (NPOE), tetrahydrofuran (THF), and high-molecular-weight PVC powder (all of >99% purity). We acquired β-CD, γ-CD, 4-tert-butylcalix[8]arene, and potassium tetrakis (4-chlorophenyl) borate from BDH (Poole, England). Moxifloxacin, in dosage form of 400 mg (Moxavudex, Shamsomox, and Moxiflox), was obtained from a local pharmacy. A (1×10^{-2} M) MOX standard solution was prepared in water. Five working solutions ($1x10^{-2}$-1×10^{-6} M) were further arranged through successive dilutions.

2.2. Instruments

The Orion pH/mV meter (model 330) was utilized to perform potentiometric measurements. The measurements involved the use of MOX membrane sensors along with an Orion double junction Ag/AgCl reference electrode (model 90-02), which consisted of a 10% (w/v) potassium nitrate solution in its outer compartment. For all pH measurements, a combination glass pH electrode (Orion81–02) was used to adjust the pH.

2.3. Preparation of the MOX-PVC membrane sensors

The ionophore PVC sensors were made using techniques that have been previously documented [20, 21]. To sum up, 2 mg KTPClPB (an ionic additive) was mixed with 190 mg PVC powder and 4 mg of β-CD, γ -CD, or calixarene (ionophores). After adding 350 mg plasticizer DOP, DBS, or NPOE to the mixture and completely mixing it, 5 ml of THF was added to 5 cm-diameter Petri dishes, and all the ingredients were thoroughly mixed once more. To give the sensor membrane time to develop, the combined ingredients were left overnight. After that, sections of the membranes were cut and glued to polyethylene tubes by THF. The electrode bodies were attached to a plastic tube that was adhered to the PVC membrane. The electrodes were filled with a combination of 1×10^{-2} M MOX and KCl [20, 21]. Before being used, the working electrode was conditioned by being submerged for around three hours in an aqueous MOX solution (0.01 M). The studied electrode was maintained in the MOX solution after the experiments were completed.

2.4. Calibration procedure

To set the MOX sensors, the developed membrane sensors and reference electrode were put into an electrochemical cell with containing 9 ml of 0.05 M sodium acetate. Following this, 1 ml of MOX (1×10^{-5}–1×10^{-1} M) was introduced to achieve ultimate MOX concentrations ranging from 1×10^{-2} to 1×10^{-6} M. The solution was continually stirred after each addition, and when the potential remained constant, the electrode's potential (E, mV) was recorded. Plotting the potential values against -log [MOX] resulted in a calibration curve, which was then utilized to measure unknown MOX concentrations.

2.5. Moxifloxacin determination in its pharmaceutical dosage forms

Ten tablets containing 400 mg of moxifloxacin (loxavudex, shamsomox, or roxiflox) were carefully weighed, crushed, and combined in a mortar. Each moxifloxacin powder sample (containing about 400 mg MOX) was weighed, then transferred to a 100-ml beaker and dissolved in double-distilled water. The mixture was then sonicated for approximately 15 minutes. The solutions were completed to the mark with double-distilled water. A 5.0-ml aliquot of these solutions was transferred to a 50-ml standard flask, the pH was adjusted using a 0.05 M sodium acetate solution, and the reaction was completed with water. Using an Orion Ag/AgCl double junction reference electrode and MOX sensors, the potential of the solution was determined. The concentration was estimated from the previously constructed calibration curve (as in the procedure section).

3. Results and Discussion

3.1. Nature of sensing element

The sensors that were studied used ionophores in a PVC membrane matrix and were controlled by how the host molecules (calixarene, β-CD, and γ -CD) and guests (MOX) recognized each other. The hydrophobic contacts between the hydrophobic cavity of the CD receptors and the MOX were primarily responsible for facilitating the interactions between the host and guest [18, 22]. The formation of inclusion complexes between MOX and calixarene, β-CD , and γ -CD sensors, respectively, occurs through the hydrophobic cavity [18]. Numerous pharmaceutical compounds can form inclusion complexes with calixarene and CDs [18, 22] through inclusion complexes. The basis for the inclusion complexes is a variety of interactions, such as dipole-dipole interactions, van der Waals forces, and hydrogen bonding [23, 24]. The selection of the ion exchanger or lipophilic ion, which effectively neutralizes the charge between the host and guest, was based on the

specific type of analyte being considered [25, 26]. To enhance the selectivity for moxifloxacin and minimize anionic interferences, we employed the lipophilic ion KTPClPB, which effectively neutralized the charge produced between the MOX and host [25, 26]. The addition of the ionic additive KTPClPB to the membrane composition at a ratio of 0.5:1 (additive: ionophore) resulted in an enhancement of both the selectivity and sensitivity of the sensors.

3.2. Effect of the plasticizer

We looked into how well the plasticizer worked on the membrane by testing the MOX sensors using the researched ionophore and three different plasticizers: DOP, DBS, and NPOE. The structure of PVC-based sensors primarily necessitates the employment of a plasticizer, which is a fluidizer that allows uniform dissolution and diffusion mobility of the ionophores. It is widely accepted that plasticizers are significant components of PVC membrane sensors. Membrane sensors benefit from these effects by reducing the detection limit and enhancing both selectivity and sensitivity. The solubility of the membrane sensors for the three plasticizers was approximately the same. On the other hand, o-NPOE shows high polarity (ε= 24) and provides a good potentiometric response compared with the low-polarity DOP (ε = 7) and DBS (ε=5). The electrode response (sensor 1) was 54 mV compared with 52 mV and 50 mV. Sensor 2 shows a response of 55 mV compared with 54 and 52 mV while sensor 3 shows a response of 56 mV compared with 55 mV and 53 mV for o-NPOE compared with DOP and DBS, respectively. Consequently, o-NPOE was employed for all the subsequent studies, as indicated in Table 1.

Table 1. Effect of plasticizer on the developed sensors.

Plasticizer	**DOP**	**DBS**	**o-NPOE**
Sensor 1	**Calixarene**		
Slope	52	50	54
Response time , sec	20	25	20
Calibration range	4.8×10^{-6}-1×10^{-2}	5×10^{-6}-1×10^{-2}	4.3×10^{-6}-1×10^{-2}
Sensor 2	**Beta**		
Slope	54	52	55
Response time , sec	20	25	20
Calibration range	3.8×10^{-6}-1×10^{-2}	4.2×10^{-6}-1×10^{-2}	3.3×10^{-6} -1×10^{-2}
Sensor 3	**gamma**		
Slope	55	53	56
Response time	20	30	20
Calibration range	4×10^{-6}-10^{-3}	6×10^{-6}-10^{-3}	3.4×10^{-6} -1×10^{-2}

3.3. Effect of pH and response time

The response of the MOX sensors was analyzed in various pH environments to study their behavioral patterns. Figure 2 illustrates the graphical representation of the impact of pH. HCl or NaOH, which are extremely diluted solutions, were used to regulate the pH level of the MOX solution. The recorded electrode response of the test solution was measured in terms of its electromotive voltage (E, mV) and plotted against the corresponding changes in pH. In Figure 2 (a, b, and c), it can be observed that the slope (E, mV) of the sensors under investigation remained consistent for sensors 1, 2, and 3, respectively. The constant slopes were measured as 54, 55, and 56 mV per 10-fold concentration change for calixarene, β-CD, and γ -CD, respectively, within the pH range of 3–8. The decrease in potential in an alkaline medium with a pH greater than 8 (pKa 9.2) [27] can be attributed to the higher concentration of MOX species that are not protonated.

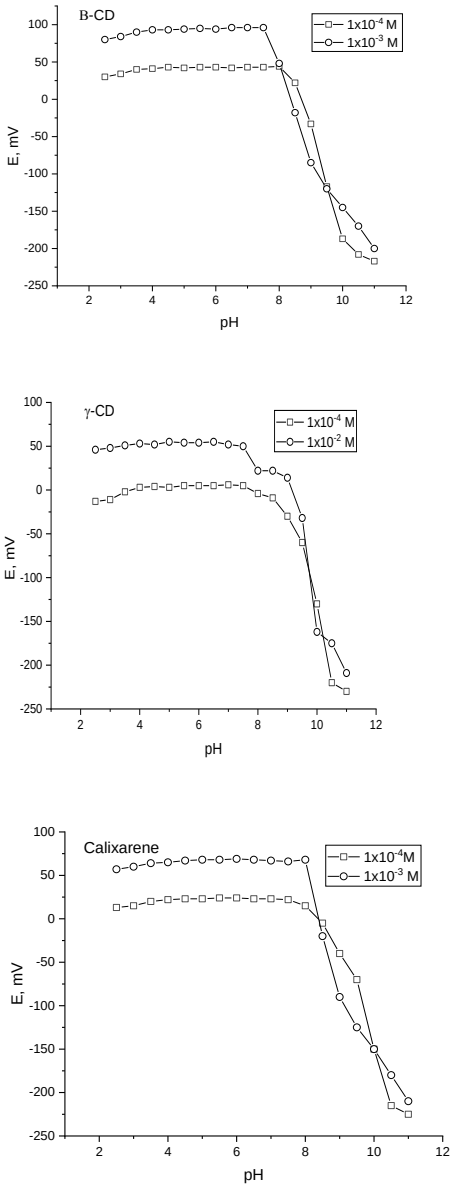

Fig. 2. Effect of pH on the electrode response of moxifloxacin sensors.

An essential component of electrode characterization is response time [28], or the time needed for the electrode to attain a steady reading. Upon immersing the electrode in varying concentrations of MOX (tenfold), the response time may either lengthen or shorten. In comparison to low concentrations, the reaction time was shorter at higher MOX concentrations. We noted that the MOX sensors exhibited an average response time of 20 seconds, (Figure 3). The period of time from the electrode's fabrication until a distinctive

response parameter is altered is known as the membrane's lifetime. The sensors had a life limit of more than 50 days; during this time, the electrodes' analytical characterization remained constant.

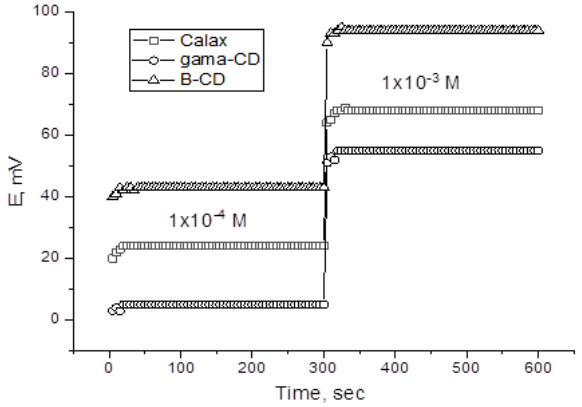

Figure 3. Dynamic response time of different MOX sensors

3.4 Effect of interference

Using separate or mixed solution procedures, the selectivity coefficients were determined in accordance with IUPAC recommendations [28, 29]. To investigate the selectivity of the sensors, several chemical species and inorganic ions were investigated as interfering substances. Based on a separate solution method The selectivity coefficients were calculated via the next equation:

$$log\ K_{A,B}^{pot} = \frac{E_B - E_A}{S} + \left[1 - \frac{Z_A}{Z_B}\right] log\ a_A$$

where E_A and E_B are the sensors' potentials when they are put in a solution of MOX and the interfering species (at the same concentration), and $K_{A,B}^{pot}$ is the selectivity coefficient. Whereas a_A stands for the activity of MOX, Z_A and Z_B denote the charges of MOX and the interfering species, respectively.

Alternatively, the selectivity coefficient determined using the mixed-solution method can be derived from the following equation:

$$K_{A,B}^{pot} = \frac{(a`_A - a_A)}{a_B}$$

where $a`_A$ is the known concentration of MOX added to an unknown concentration a_A. The variation in potential (ΔE) was recorded. Another test experiment used a solution with a known concentration of interfering ion (A_B) added to a fixed concentration of MOX until the same potential is reached. The researched method is free from interference, as indicated by the low values of the selectivity coefficient. Results were listed in Table 2.

Table 2. Potentiometric selectivity coefficients of some interfering ions, using MOX sensors.

Interferent, J*	$K_{MOX,B}^{Pot}$		
	Sensor 1	**Sensor 2**	***Sensor 3***
Na^+	1.04×10^{-3}	1.02×10^{-3}	1.11×10^{-3}
K^+	1.29×10^{-3}	1.25×10^{-3}	1.18×10^{-3}
Ca^{2+}	1.1×10^{-3}	1.09×10^{-3}	1.12×10^{-3}
Fe^{2+}	8.41×10^{-4}	8.8×10^{-4}	8.21×10^{-4}
Fe^{3+}	9.18×10^{-4}	9.08×10^{-4}	9.0×10^{-4}
Mg^{2+}	1.29×10^{-3}	1.23×10^{-3}	1.25×10^{-3}
Ni^{2+}	6.82×10^{-4}	6.59×10^{-4}	6.77×10^{-4}
Cu^{2+}	6.82×10^{-4}	6.83×10^{-4}	6.79×10^{-4}
Zn^{2+}	8.07×10^{-4}	8.11×10^{-4}	7.83×10^{-4}
Chloride	1.0×10^{-3}	1.13×10^{-3}	1.11×10^{-3}
Acetate	6.21×10^{-4}	6.08×10^{-4}	6.11×10^{-4}
Phosphate	6.82×10^{-4}	6.79×10^{-4}	7.29×10^{-4}
Glucose*	7.74×10^{-4}	7.9×10^{-4}	7.45×10^{-4}
Lactose monohydrate*	7.74×10^{-4}	7.9×10^{-4}	7.45×10^{-4}
Starch*	7.74×10^{-4}	7.9×10^{-4}	7.45×10^{-4}
Microcrystalline cellulose*	7.74×10^{-4}	7.9×10^{-4}	7.45×10^{-4}
Urea*	9.1×10^{-4}	8.92×10^{-4}	8.92×10^{-4}
Thiourea*	1.59×10^{-3}	1.43×10^{-3}	1.43×10^{-3}
Urea*	9.18×10^{-4}	9.01×10^{-4}	9.01×10^{-4}
Glycin*	8.11×10^{-4}	8.13×10^{-4}	8.13×10^{-4}

* match potential method

3.5 Sensors characteristics

Based on whether they used calixarene, γ-CD, or β-CD as sensing materials, PVC as the matrix, and o-NPOE as a plasticizer [28], the IUPAC guidelines were used to evaluate the analytical categorization of MOX-PVC sensors. The analytical characteristics of the proposed methods are presented in Table 3. The standardizing curves' least squares equations were displayed as follows:

$$E(mV) = S\ log[PR] + Intercept$$

where S is the slope (53 ± 0.5, 54.5 ± 0.5, and 55.5± 0.5 mV/decade) and the intercept (270.1 ± 0.5, 257.5

± 0.5 and 264.5 ± 0.5) for calixarene, β-CD, or γ -CD, respectively. E is the potential of the sensor (mV).

3.6 Validity of the MOX-Sensors

3.6.1 The detection (LOD) and limit of quantification (LOQ)

Plotting the electrode potential (n = 5) vs. MOX concentration was done. The following equation shows the logarithmic relationship between the concentration [M] and the voltage (E, mV):

$$X = Slog[MOX] + y$$

where S is the slope, Y is the intercept, and X is the potential (E, mV). The calibration range of the sensors at pH 3–8 was 1×10^{-2} to 4.3×10^{-6}, 1×10^{-2} to 3.3×10^{-6}, and 1×10^{-2} to 3.4×10^{-6} M for calixarene, β-CD, or γ -CD, respectively, (Figure 4). Following the IUPAC guidelines [28, 30], the LOD of MOX was found by comparing the moxifloxacin concentration to the point where the extrapolated lines of the calibration graph met. The lower limit of detection was 1.3×10^{-6}, 1.0×10^{-6}, and 1.03×10^{-6} M, whereas the LOQ was 4.3×10^{-6}, 3.3×10^{-6}, and 3.4×10^{-6} M for sensors 1, 2, and 3, respectively (Table 3).

3.6.2 Accuracy and precision

The suggested method's precision and accuracy were examined [30] by assaying MOX at 4, 40, and 400 µg/ml during a single day and over a three-day period. Five replicates' intra- and inter-day accuracy were estimated. The known MOX concentration was computed using the calibration graphs for the sensors under examination. The recovery was calculated by comparing the added concentration to the measured values, whereas the repeatability (precision) was expressed as RSD%. As shown in Table 4, the developed sensors had a 98.5% accuracy rate and an RSD of less than 2.3%.

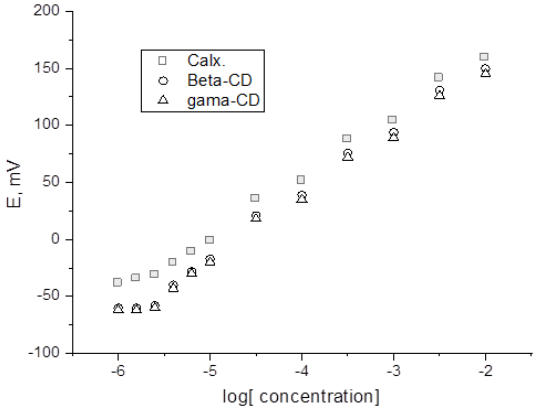

Fig. 4. Calibration curves for different MOX sensors

Table 3. Response characteristics of MOX matrix PVC matrix membrane sensors.

Parameter	**Sensor 1 calixarene**	**Sensor 2 beta**	**Sensor 3 gamma**
Slope, (mV decade^{-1})	53.5 ± 1	54.5 ± 1	55.5 ± 1
Intercept, mV	270.1	257.5	264.5
Correlation Coefficient, (r)	0.992	0.993	0.993
Calibration, rang M	4.3×10^{-6}-1×10^{-2}	3.3×10^{-6} -1×10^{-2}	3.4×10^{-6} -1×10^{-2}
Lower limit of quantification, (LOQ), M	4.3×10^{-6}	3.3×10^{-6}	3.4×10^{-6}
Lower of detection limit, (LOD), M	1.3×10^{-6}	1.0×10^{-6}	1.03×10^{-6}
Response time for1×10^{-3} M solution, s	20	20	20
Working pH range	3-8	3-8	3-8

Table 4 . Day to day reproducibility of the MOX membrane sensors.

Concentration (µg/ml)	Within-day		
	Senor 1	Sensor 3	Sensor 3
	Recovery, % ± RSD, %	Recovery, % ± RSD, %	Recovery, % ± RSD, %
4	98.5 ± 1.6	98.75 ± 1.7	98.25 ± 1.55
40	99.0 ± 1.7	99.25± 1.6	98.75 ± 1.6
400	99.0 ± 1.7	98.5 ± 2.3	98.5 ± 1.6
	Within different days		
4	98.0 ± 1.7	97.5 ± 1.8	98.0 ± 1.8
40	98.5 ± 1.6	99.0 ± 1.7	98.5 ± 1.7
400	99.5 ± 1.6	98.0 ± 1.7	98.5 ± 1.7

* Average of 5 measurements ±RSD.
*R%, recovery percentage; RSD%, relative standard deviation, %

3.6.3. Recovery

In the recommended acetate buffer, the recovery of MOX was calculated. According to the following equation, the recovery percentage of the determination was calculated.

$$\boldsymbol{Recovery}, \% = (\boldsymbol{measured\ concentraion / added\ concentration}) \times \mathbf{100}$$

The recovery percentage of MOX using the proposed sensors was found to be 98.8%, 98.83%, and 98.5% for the created sensors (1, 2, and 3), respectively (Table 5).

3.6.4. Ruggedness

Using two operators and two separate pieces of equipment on different days, the analysis of MOX was

performed to test the ruggedness of the investigated methods [30]. The results demonstrate that the studied sensors performed the analysis with very comparable results: RSD ≤2.3% was acquired both on the same day and on separate days for the assay.

3.6.5. Robustness

By examining the ideal parameters for the potentiometric method, such as response time and pH, which have an impact on the electrode response, the robustness of the procedures was examined. The procedures appear to have been reasonably robust based on the data collected under ideal conditions. The measurement medium was characterized by a pH range of 3–8, and it was determined that the optimal pH value for the experiment was 7. This was achieved by utilizing 0.05 M sodium acetate.

3.7. Application of MOX-PVC sensors

The sensors were applied to pharmaceutical formulations and pure solutions to evaluate MOX concentration. Using the developed sensors, the pure MOX (4–400 µg/ml) was measured in five preparations, with mean recovery values of 98.5%, 98.6%, and 98.14%. The sensors exhibit RSD values (%) of 2.4%, 2.61%, and 2.1% for calixarene, β-CD and γ-CD sensors, respectively (Table 5). The quantification of MOX in its pharmaceutical formulations shows a distinctive recovery of 98.5%, 98.76%, and 98.5% with RSD values of 1.5%, 1.6%, and 1.5% for calixarene, β-CD, and g-CD, respectively (Table 6). On the other hand, the assay of MOX in spiking urine showed good accuracy and recovery, as presented in Table 7. Table 6 presents the comparison between the assay of MOX in its pharmaceutical form using the developed sensors and that produced by a spectrophotometric approach [5]. The results are in good agreement with each other in terms of accuracy and precision.

Table 5. Direct determinations of MOX using PVC membrane sensors.

Added (µg/ml)	Sensor 1	Sensor 2	Sensor 3
	Recovery, % ± RSD, %	Recovery, % ± RSD, %	Recovery, % ± RSD, %
2	97.5± 2.8	98.0±2.8	97.5± 2.7
5	98.0± 2.7	98.2±2.8	97.5 ± 2.7
10	98.5± 2.1	98.5±2.6	98.0± 2.1
50	98.5 ± 2.0	98.5±2.6	98.0± 1.8
100	99.0 ± 1.8	99.0±2.5	98.5±1.8
200	99.0 ± 1.8	99.0±2.5	98.5±1.8
400	99 ± 1.8	99.0±2.5	99.0±1.8

* Average of 5 measurements ± RSD.

Table 6. Determination of moxifloxacin in its dosage forms

Dosge form	MOX Nominal value, mg	Sensor 1		Sensor 2		Sensor 3		Reported method	
		R, %	RSD, %	R, %	RSD, %	R, %	RSD, %	R, %	RSD, %
Moxavudex	400	98.5	1.5	98.8	1.6	98.8	1.6	98	2.1
Shamsomox	400	98.6	1.5	98.7	1.6	98.7	1.6	98.5	1.9
Moxiflox	400	98.5	1.5	98.8	1.6	98.8	1.6	97.5	2.5
T test		0.6		0.77		0.78			
F test		2.73		2.38		2.39			

*Average of five determinations.

Table 7. Determination of moxifloxacin in spiking urine sample by the proposed method.

Added (µg/ml)	Sensor 1			Sensor 2			Sensor 3		
	Found	R, %	RSD, %	Found	R, %	RSD, %	Found	R, %	RSD, %
4	3.94	98.5	1.6	3.95	98.75	1.7	3.93	98.25	1.55
40	39.6	99.0	1.7	39.7	99.25	1.6	39.5	98.75	1.60

*Average of five determinations.

A statistical analysis of the MOX test using both the developed sensors and the published method showed that there was no significant difference in terms of how accurate and precise the results were. The comparison was performed using the null hypothesis method with a significance level of $p \leq 0.05$ and a sample size of $n = 5$. These findings are summarized in Table 6. T = 0.6, 0.77 and 0.78which is less than the tabulated value (3.36) [30]. In addition, F = 2.73, 2.38 and 2.39 which is less than the tabulated value (6.38) [30].

3.8.Application of MOX-PVC sensors as indictor electrodes

The created electrodes have been tested as an end point indication electrode for potentiometric drug titrations in conjunction with an Ag/AgCl reference electrode. The suggested sensors have been used to titrate MOX with sodium tetraphenylborate (Figure 5). The data clearly show that MOX and NaTPB react at a molar ratio of 1:1. The symmetrical titration curves with a sharply defined potential jump demonstrated the electrodes' great sensitivity.

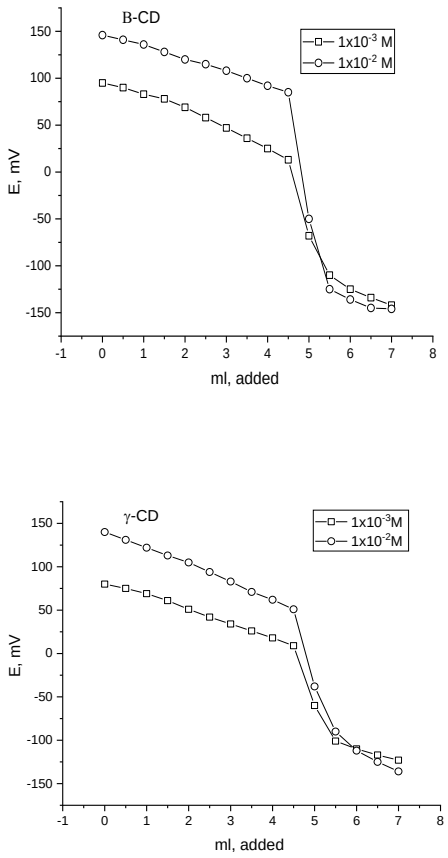

Fig. 5. Titration curve of 0.001M moxifloxacin with 0.001M NaTPB using the proposed sensors.

4. Conclusion

Three PVC membrane sensors were created to detect moxifloxacin. These sensors utilized calixarene, β-CD, and γ-CD as ionophores, KTPClPB as an anionic additive, o-NPOE as a plasticizer, and PVC as the polymeric matrix. The sensors proved to have excellent moxifloxacin selectivity and sensitivity. The sensors exhibited a nearly Nernstian response when measuring moxifloxacin, with calibration slopes of approximately 54 mV, 55 mV, and 56 mV per decade for calixarene, β-CD, and γ-CD, respectively. The examined sensors demonstrated excellent selectivity, rapid response time (20 seconds), and a broad operating pH range of 3–8. The proposed sensors were able to attain a wide calibration range for moxifloxacin, demonstrating a lifespan exceeding two months. The sensors proved effective in accurately and precisely determining moxifloxacin content in bulk samples, pharmaceutical formulations, and spiked urine samples.

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