HUNGARIAN JOURNAL OF
INDUSTRY AND CHEMISTRY
Vol. 54(1) pp. 73–81 (2026)
hjic.mk.uni-pannon.hu
DOI: 10.33927/hjic-2026-10

ION PAIR-BASED POTENTIOMETRIC SENSORS FOR SELECTIVE AND SENSITIVE DETERMINATION OF LOMEFLOXACIN

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This study reports the fabrication of two novel electrochemical sensors for the quantitative determination of the drug Lomefloxacin (LOM), employing LOM–silicotungstic acid (STA) and LOM–molybdophosphoric acid (MPA) ion pairs as electroactive materials. The sensors were systematically characterized in terms of gradient, linear concentration range, detection limit, response time, pH tolerance and shelf life. The applicability of the sensors was demonstrated by successfully determining LOM in pharmaceutical tablet formulations as well as in real samples such as urine using the standard addition method. The proposed sensors exhibited long operational lifetimes, high stability, sensitivity, precision, accuracy and selectivity. Moreover, they offer a cost-effective, simple and rapid analytical approach for routine LOM determination.

Keywords: Lomefloxacin, molybdophosphoric acid, phosphotungstic acid, potentiometric sensors, analytical applications

1. Introduction

Lomefloxacin hydrochloride (*Figure 1*) is a fluoroquinolone antibiotic commonly prescribed for bacterial infections such as bronchitis and urinary tract infections. Quinolones represent a broad class of synthetic antimicrobial agents that are highly effective against a wide range of infectious diseases, particularly bacterial infections. However, as bacterial resistance continues to expand, novel quinolones are being developed. Although initially considered relatively safe, long-term clinical use has revealed several adverse effects. Since 1965, multiple case reports have linked quinolone use, especially when combined with systemic corticosteroids, to spontaneous tendon ruptures and related injuries. Consequently, in early 2004, the U.S. Food and Drug Administration heightened the warnings in the package inserts of all drugs in this class to highlight these serious risks.

Lomefloxacin - 1-ethyl-6,8-difluoro-1,4-dihydro-7-(3-methyl-1-piperazinyl)-4-oxo-3-quinolinecarboxylic acid - is one of the synthetic antibacterial fluoroquinolone agents of the third generation [1]. It is a white to pale yellow powder with a molecular weight of 387.8 that is slightly soluble in water and practically insoluble in alcohol. Lomefloxacin hydrochloride is thermally stable and moisture-resistant but is sensitive to light in dilute aqueous solutions. It is a synthetic broad-spectrum antimicrobial agent for oral administration.

Lomefloxacin (LOM) is also used to prevent urinary tract infections prior to surgery. It is used as a prophylactic or preventative treatment to prevent urinary tract infections in patients undergoing transrectal or transurethral surgical procedures.

Fluoroquinolones such as lomefloxacin possess excellent activity against Gram-negative aerobic bacteria such as *E. coli and Neisseria gonorrhoeae* as well as Gram-positive bacteria including *Salmonella pneumonia* and *Staphylococcus aureus* [2]. They also possess effective activity against *Shigella, Salmonella, Campylobacter*, gonococcal organisms as well as multidrug-resistant *Pseudomonas* and *Enterobacter*. The bactericidal action of lomefloxacin results from interference with the activity of the bacterial enzymes DNA gyrase and topoisomerase IV, which are needed for

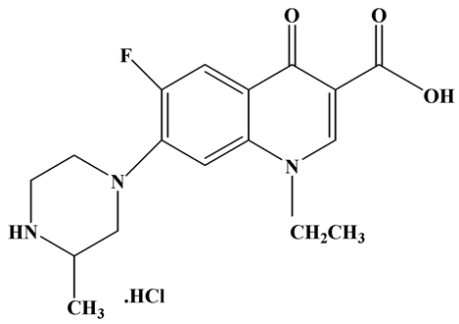

Figure 1: Structure of Lomefloxacin

Received: 4 Oct 2025; Revised: 19 Nov 2025;
Accepted: 20 Nov 2025
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the transcription and replication of bacterial DNA. DNA gyrase appears to be the primary target of quinolone for Gram-negative bacteria. Topoisomerase IV appears to be the preferential target in Gram-positive organisms. Interference with these two topoisomerases results in strand breakage of the bacterial chromosome, supercoiling and resealing. As a result DNA replication and transcription is inhibited.

Lomefloxacin is extensively used because of its strong and prolonged antibacterial activity. Therefore, the determination of lomefloxacin in capsules, injections and blood samples requires a simple, quick and sensitive analytical method. A number of analytical methods for the determination of lomefloxacin, for example, UV/Visible spectrophotometry **Hiba! A hivatkozási forrás nem található.**-[5], spectrofluorometry [6], flow injection analysis **Hiba! A hivatkozási forrás nem található.**, capillary electrophoresis **Hiba! A hivatkozási forrás nem található.**, sensitized fluorometry **Hiba! A hivatkozási forrás nem található.**, electrochemical analysis **Hiba! A hivatkozási forrás nem található.**-[17] and high-performance liquid chromatography **Hiba! A hivatkozási forrás nem található.** have been proposed. However, most of these methods require sophisticated instrumentation as well as are prohibitive in terms of cost or technical difficulty. Because of these considerations, potentiometry with ion-selective electrodes seems to have potential for the determination of lomefloxacin in pharmaceutical substances. Potentiometric sensors or so-called ion-selective electrodes (ISEs) are the subject of continuous research. This group of chemical sensors are simply prepared, robust and moderately selective in terms of analytical performance [19],[20]. Over the last three decades, being commercially available and not expensive, ion-selective electrodes have become common in analytical work since they are simple, low-cost and take rapid, accurate measurements of ionic species. The key to constructing such an electrode is to produce a sensitive and selective membrane that responds to a particular drug. Such a membrane is usually prepared by incorporating an appropriate ion exchanger and solvent mediator into a poly(vinyl chloride) membrane matrix [21].

This study focuses on developing and testing simple polymeric membrane potentiometric sensors for detecting LOM in pharmaceutical formulations. The sensors were designed using ion-association complexes formed between LOM and silicotungstic acid (STA) or molybdophosphoric acid (MPA), which served as the electroactive materials. These complexes were incorporated into plasticized poly(vinyl chloride) membranes to construct ISEs. The prepared sensors were thoroughly characterized and compared, moreover, their performance evaluated for the rapid, accurate and selective determination of LOM in pharmaceutical products, pure drug samples and even biological ones such as urine.

2. Experimental methods

2.1. Reagents and materials

All chemicals used were of analytical grade. Molybdophosphoric acid (MPA), Dibutyl sebacate (DBS) and all of the metal salts used were obtained from Merck. Bis(2-ethylhexyl) phthalate (BEP), Bis(2-ethylhexyl) sebacate (BES), Bis(2-ethylhexyl) adipate (BEA) as well as Di-n-butyl phthalate (DBP) were obtained from Lancaster (UK) and tetrahydrofuran (THF) from a local chemical supplier (S D Fine–Chem Ltd). Pure-grade LOM was donated. Pharmaceutical preparations [Lomedon (Indon, India) and Lomegen (Genix, India)] were purchased from a local market.

2.2. Apparatus

The potential measurements were carried out at 25±0.1 ºC using a Metrohm 781 ion meter. All EMF measurements were taken by the following cell assembly.

Saturated calomel electrode / internal filling solution (1×10^{-1} M NaCl solution + 1×10^{-3} M drug solution) / PVC membrane / test solution / KCl salt bridge / saturated calomel electrode.

2.3. Synthesis of the ion association complexes

The ion association complexes were prepared by mixing 75 mL of 1.0×10^{-2} M LOM with 25 mL of 1.0×10^{-2} M STA and 25 mL of 1.0×10^{-2} M MPA solutions. The precipitates formed were stirred well before being filtered through a Whatman filter paper and washed with distilled water several times. The obtained precipitates were dried at room temperature and stored in a desiccator. These ion associations were used for the fabrication of polymeric membrane sensors to determine LOM. The compositions of both ion association complexes were confirmed by elemental analysis to be 3:1 (LOM:STA and LOM:MPA) as follows:

- LOM-STA ion association
  Found (%) – C - 15.38, H - 1.34, N - 3.26
  Calculated (%) – C - 15.56, H - 1.45, N - 3.20
- LOM-MPA ion association
  Found (%) – C - 18.63, H - 1.63, N - 3.56
  Calculated (%) – C - 18.48, H - 1.72, N - 3.80

2.4. Fabrication of the LOM membrane sensor

The membrane sensors were prepared using the synthesized ionophores following the procedure reported by Cragg et al. [22]. In this approach, the ionophore, poly(vinyl chloride) (PVC), and a plasticizer were first dissolved in 5–6 mL of tetrahydrofuran (THF). The resulting solution was poured into glass rings fixed on a glass plate, covered with filter paper and left to stand overnight at room temperature to allow the solvent to evaporate slowly. This process yielded a transparent

sensing membrane approximately 1 mm thick. Circular disks about 12 mm in diameter were then cut from the membrane and attached to one end of glass tubes. The electrode bodies were filled with an internal solution containing 1.0×10^{-1} M NaCl and 1.0×10^{-3} M LOM. Prior to use, the membrane electrodes were conditioned overnight in a 1.0×10^{-3} M LOM solution.

3. Results and discussion

3.1. Potential measurement and calibration

A stock solution of LOM (1.0×10^{-1} M) was first prepared and working solutions obtained by suitably diluting with water. The membrane electrodes were then tested by immersing them in LOM solutions of varying concentrations. Their performance was evaluated by measuring the EMF values over the concentration range of 1.0×10^{-2} to 1.0×10^{-8} M. After stabilization, the potential readings were recorded and plotted against log [LOM]. The resulting calibration curve was subsequently employed for the determination of unknown LOM concentrations.

3.2. Performance characteristics of the developed sensors

The performance of any potentiometric ion-selective sensor is typically assessed through its response parameters. These electrochemical characteristics of a newly developed sensor are influenced by several factors, including the membrane composition, type of plasticizer used and the make-up of the carbon paste.

3.2.1. Optimization of the membrane composition

Table 1: Optimization of the composition of the PVC membrane sensor using LOM – STA ion association

Sensor	Composition % (w/w)			Gradient
	Ion-pair %	PVC %	Plasticizer %	mV/decade
LO_{S1}	1.0	42.4	56.6, DBP	61.5
LO_{S2}	1.2	50.2	48.6, DBP	50.4
LO_{S3}	***1.4***	***32.6***	***66.0, DBP***	***58.2***
LO_{S4}	1.0	42.4	56.6, BES	39.8
LO_{S5}	1.2	50.2	48.6, BES	50.7
LO_{S6}	1.4	32.6	66.0, BES	48.1
LO_{S7}	1.0	42.4	56.6, BEP	52.5
LO_{S8}	1.2	50.2	48.6, BEP	60.5
LO_{S9}	1.4	32.6	66.0, BEP	50.8
LO_{S10}	1.0	42.4	56.6, DBS	53.2
LO_{S11}	1.2	50.2	48.6, DBS	64.7
LO_{S12}	1.4	32.6	66.0, DBS	44.3
LO_{S13}	1.0	42.4	56.6, BEA	64.1
LO_{S14}	1.2	50.2	48.6, BEA	49.2
LO_{S15}	1.4	32.6	66.0, BEA	50.6

The operating characteristics of ion-selective electrodes can be significantly modified by changing the relative proportions of the components of the membrane electrode, which is essentially comprised of the ionophore and plasticizer. A total of fifteen different sensors were prepared for both ion associations by varying the ratios of the ionophore, plasticizer and PVC. It is well known that the construction of PVC-based ISEs requires the use of a plasticizer which mainly acts as a fluidizer allowing homogeneous dissolution and diffusional mobility of the ion pair inside the membrane. The nature and/or the amount of plasticizer must be properly controlled in order to minimize the electrical asymmetry of the membrane as well as limit fouling of the sensor. In addition, suitable selection of a plasticizer allows the electrode/solution distribution ratio to be controlled of the particular C+A− ion pair employed as an ion exchanger. The analytical performance of such an electrode is strongly dependent on a suitable ratio of plasticizer to electroactive material. Hence, the present work involved the study of the influence of the plasticizer type and also its concentration on the performance characteristics of the sensors. Accordingly, five different types of plasticizers were chosen for the study which involves BEP, BES, DBP, DBS and BEA. In the present study, several membrane compositions were investigated in which the percentage of the ion exchanger ranged from 1.0 to 1.4% (*Table 1*).

For the membrane electrode incorporating LOM-STA as the ionophore, the best result was obtained with the sensor containing 1.4 wt% of the ionophore. The optimized composition of the corresponding sensor was the following: ion association: PVC: plasticizer (DBP) as 1.4: 32.6: 66.0 wt% (LO_{S3}). Of the five different plasticizers used, DBP was found to produce a near-Nernstian response.

Table 1a: Correlation studies between the composition variables and sensor gradient for the LOM–STA sensors

Variable Pair	Correlation coefficient (R)	Coefficient of determination (R^2)
Ion-pair % vs. Gradient	–0.224	0.050
PVC % vs. Gradient	+0.282	0.079
Ion-pair % vs. PVC %	–0.556	0.309
Plasticizer % vs. Gradient	–0.280	0.078

The correlations between the composition variables and sensor gradient for the LOM–STA sensors are shown in *Table 1a*. The strongest correlation exists between the PVC % and gradient with R = 0.282, explaining a variance of only 7.9% in the gradient response. This weak positive relationship suggests that increasing the

Table 2: Optimization of the composition of the PVC membrane sensor using the ion association LOM–MPA

Sensor	Composition % (w/w) Ion-pair %	PVC %	Plasticizer %	Gradient mV/decade
LO_{M1}	1.0	42.4	56.6, DBP	49.1
LO_{M2}	1.2	50.2	48.6, DBP	52.7
LO_{M3}	1.4	32.6	66.0, DBP	47.6
LO_{M4}	1.0	42.4	56.6, BES	64.3
LO_{M5}	1.2	50.2	48.6, BES	45.1
LO_{M6}	1.4	32.6	66.0, BES	52.4
LO_{M7}	1.0	42.4	56.6, BEP	52.3
LO_{M8}	***1.2***	***50.2***	***48.6, BEP***	***54.9***
LO_{M9}	1.4	32.6	66.0, BEP	62.7
LO_{M10}	1.0	42.4	56.6, DBS	42.7
LO_{M11}	1.2	50.2	48.6, DBS	61.4
LO_{M12}	1.4	32.6	66.0, DBS	67.1
LO_{M13}	1.0	42.4	56.6, BEA	35.5
LO_{M14}	1.2	50.2	48.6, BEA	50.9
LO_{M15}	1.4	32.6	66.0, BEA	48.5

Table 2a: Correlations between the composition variables and sensor gradient for the LOM–MPA sensors

Variable Pair	Correlation coefficient (R)	Coefficient of determination (R^2)
Ion-pair % vs. Gradient	+0.07	0.005
PVC % vs. Gradient	–0.22	0.048
Ion-pair % vs. PVC %	–0.56	0.314
Plasticizer % vs. Gradient	+0.31	0.096

PVC concentration marginally elevates the electrode gradient, however, this relationship is inconsistent. Conversely, the plasticizer percentage exhibits a weak negative correlation of R = –0.280, indicating that higher plasticizer concentrations tend to slightly reduce the gradient, accounting for a variance of 7.8%. The weak negative correlation with regard to the ion-pair percentage (R = –0.224) suggests that higher ion-pair concentrations are associated with modestly lower gradients, however, this accounts for only a variance of 5.0%.

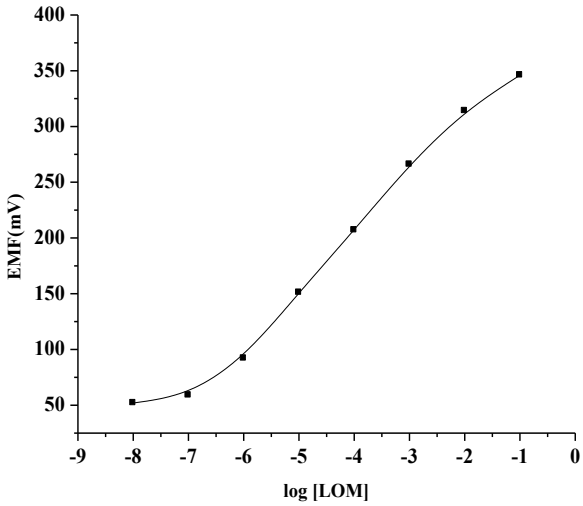


Figure 2: Calibration graph for LOM - selective PVC membrane sensor LO_{S3} at 25 °C

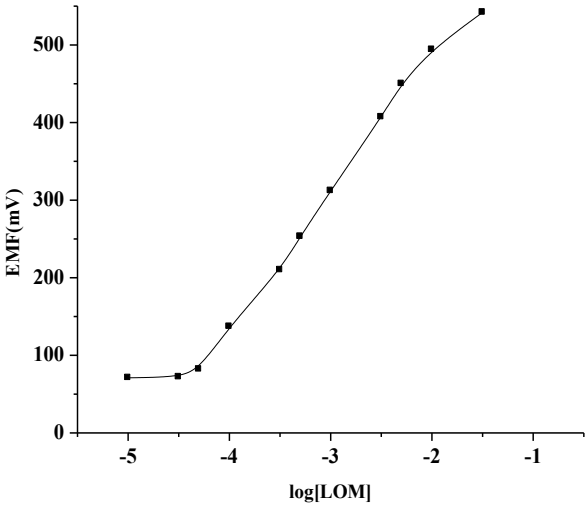


Figure 3: Calibration graph for LOM-selective PVC membrane sensor LO_{M8} at 25 °C

The sensor LO_{S3} yielded a near-Nernstian gradient of 58.2 mV/decade over a concentration range of 1.0×10^{-2} – 1.0×10^{-6} M (*Figure 2*). Hence, the following studies were performed using the LO_{S3} sensor.

The membrane composition of the sensors incorporating the ion association LOM–MPA was also varied (*Table 2*).

It was found that the sensor with a 1.2 wt% ion pair yielded a near-Nernstian gradient of 54.9 mV/decade (*Figure 3*).

The correlation coefficients observed between the different variable pairs for the LOM–MPA sensors are summarized in *Table 2a*, providing a quantitative measure of how membrane composition parameters are related to the resulting electrode gradients.

In the case of LOM–MPA sensors, the ion-pair concentration exhibited almost no correlation with the gradient (R = 0.07, R^2 = 0.005), indicating minimal influence on electrode performance. PVC content

Table 3: Response characteristics of the developed sensors LO_{S3} and LO_{M8}

Parameter	PVC membrane sensor LO_{S3}	PVC membrane sensor LO_{M8}
Gradient (mV per decade)	58.2	54.9
Linear range (M)	1.0×10^{-2} – 1.0×10^{-6}	1.0×10^{-2} – 5.0×10^{-5}
pH range	6 – 8	7 – 9
Detection limit (M)	2.69×10^{-6}	5.23×10^{-5}
Response time (s)	< 20	< 30
Shelf life	3 weeks	4 weeks

Table 3a: Correlation studies between the performance parameters of the developed sensors LO_{S3} and LO_{M8}

Parameter	PVC membrane sensor LO_{S3}	PVC membrane sensor LO_{M8}	Correlation coefficient (R)
Gradient (mV per decade)	58.2	54.9	0.56
pH range	6 – 8	7 – 9	
Response time (s)	< 20	< 30	
Shelf life	3 weeks	4 weeks	

showed a weak negative correlation (R = –0.22, R^2 = 0.048), suggesting that increasing it slightly reduces sensor sensitivity. The plasticizer percentage exhibited a marginally higher but still weak positive correlation (R = 0.31, R^2 = 0.096), implying that a higher plasticizer content mildly enhances the gradient, possibly due to improved ion mobility within the membrane. A moderate negative correlation (R = –0.56, R^2 = 0.314) between the ion-pair % and PVC % suggests compositional interdependence during membrane formulation.

Out of the five plasticizers used, BEP yielded the best result in terms of the gradient. The optimized membrane composition can be represented as follows: ion association: PVC: plasticizer (DBP), that is, 1.2: 50.2: 48.6 wt% (LO_{M8}). The response characteristics of both the sensors, namely LO_{S3} and LO_{M8}, are summarized in *Table 3*.

From *Table 3*, the correlation between the quantitative parameters of sets of 2 numerical values was calculated. R was calculated from the gradients (mV/decade), detection limits ($\times10^{-6}$ M), response times (s) and shelf lives (weeks) presented in *Table 3a*. The linear range and pH range were excluded because they are intervals not single values.

A correlation coefficient (R) of 0.56 was obtained, which suggests a moderate positive correlation between the performance parameters of the two sensors, indicating that while their characteristics exhibit comparable trends, the relationship between them is not strongly linear.

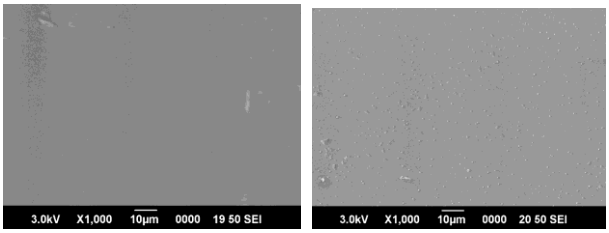
3.2.2. *Effect of the concentration of the internal filling solution*

The influence of the concentration of the internal filling solution on the potential response of the LOM selective membrane sensors was studied. The LOM concentration was varied from 1.0×10^{-4} to 1.0×10^{-2} M. In all the cases, the EMF vs. log [LOM] plot was obtained. It was found that varying the concentration of the internal reference solution does not cause any significant differences in the electrode response. Hence, a 1.0×10^{-3} M internal filling solution was selected for further assays. The optimum conditioning time for the membrane sensors in the 1.0×10^{-3} M LOM solution was approximately 12 and 24 hours for the LOM-STA and LOM-MPA sensors, respectively, which then generate stable potentials when placed in contact with LOM solutions. The electrodes were rinsed with distilled water before each measurement and stored in the preconditioning solution when not in use.

3.2.3. *Working concentration range, gradient and response time*

Sensor LO_{S3} had a working concentration range from 1.0×10^{-2} to 1.0×10^{-6} M with a gradient of 58.2 mV/decade. In the case of the LO_{M8} sensor, a linear response was obtained within the concentration range of 1.0×10^{-2} to 5.0×10^{-5} M, which was chosen as the working concentration range of this sensor. The sensor exhibited a near-Nernstian gradient of 54.9 mV/decade within this working concentration range. The limit of detection, as determined from the intersection of the two extrapolated linear segments of the calibration graph, was 2.69×10^{-6} M and 5.23×10^{-5} M for LO_{S3} and LO_{M8}, respectively. The time required for all the constructed sensors to reach values within ±1 mV of the final equilibrium potential after immersion in LOM solutions was investigated. The response time of the sensor LO_{S3} was found to be <20 s, whereas that of LO_{M8} was <30 s.

A SEM analysis was conducted in order to determine the surface morphology of the developed membranes. The scanning electron microscopic images of the surfaces of membranes LO_{S3} and LO_{M8} are presented in *Figures 4a* and *4b*, respectively. These images indicate the homogeneity of the membranes which is a major factor affecting the response characteristics of the sensors. One of the disadvantages of a PVC-based membrane is that it is not possible to control the uniform distribution of the electroactive material in the membrane. From the figures, it is clear that a smooth and homogeneous surface is obtained for membrane LO_{S3}. The better response parameters of LO_{S3} compared to LO_{M8} may be due to its much more



(a) (b)

Figure 4: SEM images of the polymeric membrane of sensors LO_{S3} (a) and LO_{M8} (b)

homogeneous and uniform surface. A high degree of sensitivity as indicated by the gradient, lower detection limit and fast response time are characteristic of sensor LO_{S3}.

3.2.4. Effect of pH

It is necessary to determine the optimum pH range over which the electrode functions without interference from the protons and hydroxide ions. The pH dependence of the test solution on the electrode potential was tested over the range of 1 to 12 at two different LOM concentrations, namely 1.0×10^{-3} and 1.0×10^{-4} M. The pH of the test solutions was adjusted by using appropriate buffer solutions. In the case of LO_{S3}, it is clear from *Figure 5* that the potential remained constant between pH 6 and 8, beyond which it changed considerably. In *Figure 5*, the EMF vs. pH plot reveals a noticeable change in the gradient around pH 8–9 for both the sensors, marking a clear shift in the reaction mechanism of the electrode. Above this pH range, the gradient steepens, indicating that the electrode potential becomes more sensitive to changes in pH. According to the Nernst equation, this behavior suggests an increase in the effective proton-to-electron mass ratio (m/n), implying that proton involvement in the electrode reaction becomes more significant or occurs through a different pathway. This observation confirms that beyond pH 8, the reaction mechanism changes and protons begin to play a stronger or altered role in influencing the electrode reaction.

The pH vs. potential profile of LO_{M8} is presented in *Figure 6*, showing that the potential remained constant between pH 7 and 9. The observed potential drift at lower pH values may be attributed to the membrane response to protons. The decrease in potential readings when pH >9.0, on the other hand, is probably attributed to the interference of the hydroxide ions and the decrease in concentration of the LOM ions. Furthermore, the increase in the gradient above pH 8 confirms a distinct mechanism change around pH 8–9. Beyond this point, the electrode reaction becomes more proton-dependent, suggesting the involvement of a different redox species or a change in the rate-limiting step with altered proton-to-electron stoichiometry.

3.2.5. Potentiometric selectivity

Selectivity is one of the most important characteristics of a membrane sensor because it helps to determine whether

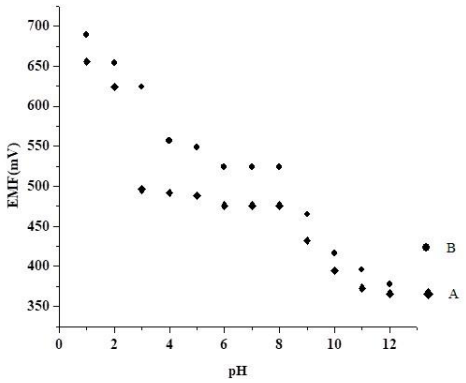


Figure 5: Effect of pH on the cell potential of the LOM-selective PVC membrane sensor LO_{S3} at (A) 1.0×10^{-4} M and (B) 1.0×10^{-3} M

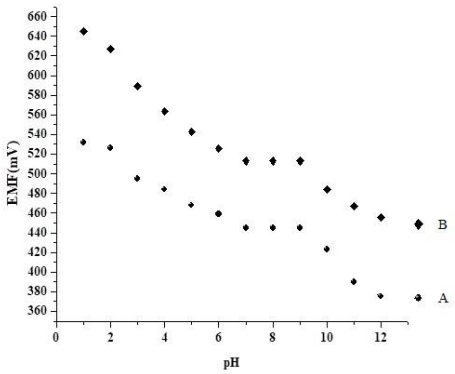


Figure 6: Effect of pH on the cell potential of the LOM-selective PVC membrane sensor LO_{M8} at (A) 1.0×10^{-4} M and (B) 1.0×10^{-3} M

measurements can be reliable in the target sample or not. To investigate the selectivity of the proposed membrane sensors, their potential responses were investigated in the presence of various interfering ions. Selectivity is measured in terms of the potentiometric selectivity coefficient $K_{A,B}^{pot}$, which measures the response of the electrode to the primary ion in the presence of foreign ions. The extent of interference of various interfering species was studied by the fixed interference method **Hiba! A hivatkozási forrás nem található.**. The resulting selectivity coefficients are summarized in *Table 4*, revealing a high degree of selectivity for LOM in the presence of the other common species tested. Common excipients used in pharmaceutical preparations like lactose and glycine did not interfere.

3.2.6. Shelf life or lifetime

The calibration curve was periodically plotted with standard solutions and the response gradients calculated in order to determine the shelf life of the fabricated sensors. It was observed that the investigated electrode LO_{S3} exhibited a good level of stability in terms of the gradient in the linear domain of concentration and could be used continuously for about 3 weeks without the

Table 4: Potentiometric selectivity coefficients of various interfering species, K_{pot}

Interfering Species	K_{pot} PVC membrane sensor LO_{S3}	K_{pot} PVC membrane sensor LO_{M8}
NH_4^+	2.6×10^{-2}	3.1×10^{-2}
K^+	1.5×10^{-3}	2.8×10^{-3}
Na^+	7.3×10^{-2}	5.4×10^{-2}
Mg^{2+}	4.3×10^{-3}	4.1×10^{-2}
Co^{2+}	6.4×10^{-2}	5.7×10^{-2}
Ca^{2+}	3.5×10^{-3}	4.9×10^{-3}
Ni^{2+}	8.6×10^{-3}	7.3×10^{-3}
Zn^{2+}	6.7×10^{-3}	8.4×10^{-3}
Ascorbic acid	5.6×10^{-2}	4.8×10^{-2}
Starch	6.1×10^{-2}	4.5×10^{-2}
Talc	3.9×10^{-2}	4.8×10^{-2}
Glycine	3.8×10^{-3}	5.3×10^{-3}
Lactose	2.9×10^{-2}	4.1×10^{-2}

Table 5: Determination of Lomefloxacin in the pharmaceutical formulations

Sample	Declared Amt (mg/tablet)	Method adopted	Found * (mg/tablet)	SD	CV
Lomedon (Indon, India)	400	LO_{S3}	398	0.96	0.24
		LO_{M8}	397	0.85	0.21
		Standard method	397	0.91	0.23
Lomegen (Genix, India)	400	LO_{S3}	397	0.94	0.24
		LO_{M8}	397	0.93	0.23
		Standard method	398	0.97	0.24

*Average of six replicates

Table 6: Determination of Lomefloxacin in a urine sample using the developed sensors

Drug taken (M)	Sensor	Drug found* (M)	Recovery %
2.00×10^{-3}	LO_{S3}	1.98×10^{-3}	99.0
	LO_{M8}	2.04×10^{-3}	102.0

*Average of six replicates

gradient decreasing much. Sensor LO_{M8} could be used continuously for about 4 weeks without a considerable decrease in its gradient. After this period, it appeared that the response characteristics altered and the sensors could not be used for analytical purposes.

4. Analytical applications

The prepared sensors have been successfully used for the determination of LOM in pharmaceutical formulations and real samples like urine.

4.1. Determination of LOM in pharmaceutical formulations (tablets)

The developed sensors LO_{S3} and LO_{M8} were applied to determine LOM in pharmaceutical formulations commercialized by Lomedon (Indon, India) and Lomegen (Genix, India). The LOM content was determined by the proposed ion-selective sensors using the calibration method. LOM present in its tablets was determined by the proposed method and a standard spectrophotometric method **Hiba! A hivatkozási forrás nem található.**. The results are summarized in *Table 5*, which clearly indicate a satisfactory level of agreement between the LOM content determined by the proposed membrane sensors and the standard spectrophotometric method as well as the declared amounts in the prepared drug used. This is indicative of the noninterference of the other ingredients and excipients present in the pharmaceutical formulations.

4.2. Recovery of LOM from a urine sample

In order to investigate the applicability of the proposed LOM membrane sensors in determining this drug in biological fluids, they were applied to recover a 2.00×10^{-3} M solution of the drug from a urine sample. The sensors proved useful for determining LOM content in a urine sample using the standard addition method as illustrated in *Table 6* from which acceptable levels of recovery are clearly obtained by the proposed membrane electrodes. The recovery percentages of LOM obtained using the sensors LO_{S3} and LO_{M8} were 99.0 and 102.0, respectively.

5. Conclusions

Novel polymeric membrane sensors were developed for the drug LOM based on the ion-pair complexes LOM-STA and LOM-MPA. The proposed sensors were stable, sensitive, precise, accurate and selective as well as had a long lifetime. The sensors are cost-effective as well as easy to prepare and use. Their usefulness for LOM determination in real samples, particularly for some commercial pharmaceutical preparations, was demonstrated, suggesting their suitable application as

reliable and advantageous alternatives to most other previously reported methods regarding the routine control of LOM concentration in these samples. The sensors were also applied to determine this drug in real samples like urine. The analytical method proposed proved to be simple, rapid and accurate.

Acknowledgements

The authors would like to express their sincere gratitude to Sophisticated Test and Instrumentation Centre (STIC) in Kochi, India for the analysis.

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