Short communication

New Spectrophotometric Methods for the Determination of Moxifloxacin in Pharmaceutical Formulations

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Abstract

Two rapid, simple and sensitive spectrophotometric methods for the quantitative analysis of moxifloxacin (MOX) in pharmaceutical formulations have been described. The first method (A) involves reaction of MOX with 1,2-naphthoquinone-4-sulphonate (NQS) in alkaline medium (pH 11.0) which results in an orange-coloured product exhibiting maximum absorption (λ_{max}) at 411 nm. The second method (B) is based on the oxidation of the MOX with a known excess of cerium (IV) sulfate and the residual oxidant is determined by treating with a fixed amount of methyl orange, and measuring the absorbance at 507 nm. The molar absorptivities for methods A and B were 4.9×10^3 and 6.5×10^4 L mol^{-1} cm^{-1}, respectively. Under the optimized reaction conditions, Beer's law correlation of the absorbance with MOX concentration was obtained in the range of 2.5–20 and 0.5–30 μgmL^{-1} for method A and B respectively. The intra-day precision expressed as relative standard deviation (RSD) was < 1.6% for both methods. The methods were validated in terms of accuracy and precision and were successfully applied to the determination of MOX in its pharmaceutical dosage form. The proposed methods are useful for routine analysis of MOX in quality control laboratories.

Keywords: Moxifloxacin; Spectrophotometry; 1,2-Naphthoquinone-4-sulphonate; Cerium(IV) Sulfate; Methyl Orange; Pharmaceutical formulations

1. Introduction

Moxifloxacin (1-cyclopropyl-7-(2,8- diazobicyclo[4.3.0] nonane)-6-fluoro-8- methoxy-1,4-dihydro-4-oxo-3-quinoline carboxylic acid) (Figure 1) is a fourth generation fluoroquinolone. Moxifloxacin is active against broad spectrum of pathogens, encompassing Gram-negative, Gram-positive bacteria and also antibiotic resistant *Streptococcus pneumonia*.[1,2]

Several analytical methods for quantitative determination of fluoroquinolones in pharmaceutical formulations are reported including high-performance liquid chromatography (HPLC)[3–8] spectrophotometry,[9–11] and capillary electrophoresis (CE).[12–18] Chromatographic and CE methods are dedicated to sophisticated and/or expensive instruments that are not available in several quality control laboratories.

The determination of MOX is not yet described in any pharmacopoeias. Therefore, a simple, accurate method is required for their determination in pharmaceutical formulations. A survey of literature revealed that MOX has been determined in biological fluids or pharmaceutical products by HPLC,[19–24] voltammetry[25] CE with laser-induced fluorescence[15] atomic absorption,[26] spectrofluorometry[27, 28] and spectrophotometry.[29–34]

Spectrophotometry is considered the most convenient analytical technique, because of its inherent simplicity, low cost, and wide availability in most quality control laboratories. However, some of spectrophotometric methods reported for determination of MOX[30–31] were associated with some major drawbacks, because of maximum absorption peak (λ_{max}) at 294.4 nm (Sahu et al., 2011) and 296 and 289 nm.[30] Because of the highly blue shifted of above λ_{max}, their determination in the dosage forms based

on the direct measurement of their absorption in UV spectral range is susceptible to potential interferences from the co-extracted common excipients. In this paper, we report two new spectrophotometric methods for the determination of MOX in pharmaceutical tablets that overcome these drawbacks.

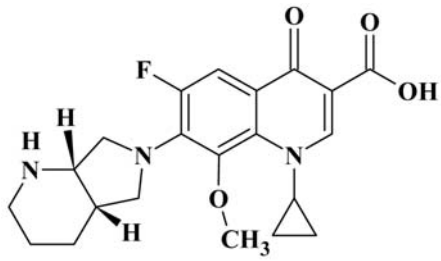

Figure 1. Chemical structure of Moxifloxacin (MOX)

2. Experimental

2. 1. Materials

All chemicals used were of analytical reagent grade. Chemicals were as follows: moxifloxacin hydrochloride (98%, Bayer AG, Leverkusen Germany); 1,2-naphthaquinone-4-sulphonate (NQS, 97%), methyl orange (MO), Sigma-Aldrich, Co., St. Louis, USA); cerium (IV) sulfate (85%, Loba- Chemie Indoaustranal Co., India); sulphuric acid (S. d. Fine Chem, Mumbai, India). Avelox tablets (Bayer AG) labeled to contain 400 mg MOX per tablet were purchased from the local market. Bi-distilled water was used to prepare all solutions.

2. 2. Reagents

A 0.5% (w/v) NQS solution was freshly prepared in bi-distilled water and was stored protected from light during use.

Buffer solution of pH 11.0 was prepared by mixing 25 mL solution of NaH_2PO_4 (0.2M) and 66 mL solution of NaOH (0.2M) in 100 mL volumetric flask, pH was adjusted by pH meter model pH211 (HANNA, Italy). Other buffer solutions of different pH values were also prepared.

A 0.01 g mL^{-1} cerium (IV) sulfate stock solution was prepared in 1 M sulphuric acid and was further diluted with 1 M sulphuric acid to obtain 250 μg mL^{-1} cerium (IV) sulfate working solution.

A 500 μg mL^{-1} MO dye solution was prepared in water and filtered and diluted 10-fold to obtain a MO working solution with the concentration 50 μg mL^{-1}.

2. 3. Preparation of Standard and Sample Solutions

A stock solution of moxifloxacin hydrochloride (MOX) 1 mg mL^{-1} was prepared in water and further diluted with water to prepare working solutions 2.5–30 μg mL^{-1}.

An accurately weighed amount of finely powdered tablet (10 tablets) equivalent to 100 mg of MOX was transferred into a 100 mL calibrated flask, dissolved in about 40 mL of water, shaked for 15 min, and filled up to volume with water. The contents were mixed well and filtered rejecting the first portion of the filtrate. The prepared solution was further diluted with water to obtain a suitable concentration for the analysis.

2. 3. 1. Method Using NQS (Method A)

Accurately measured 1 mL of MOX standard working solution (2.5–20.0 μg mL^{-1}) was transferred into 10 mL volumetric flask followed by addition of 1 mL of buffer solution pH 11.0 and 1 mL of NQS solution left at room temperature for 20 min and then filled up to volume with water. The resulting solution was measured at 493 nm against reagent blank prepared in the same manner with 1 mL water instead of 1 mL of sample solution. Double beam UV-1800 ultraviolet-visible spectrophotometer (SHIMADZU, Kyoto, Japan) with temperature controller was used for all the spectrophotometric measurements.

2. 3. 2. Method Using Cerium Sulfate & Methyl Orange (Method B)

Accurate aliquots containing 5–30 μg mL^{-1} of MOX standard solution were transferred from the stock standard solution into 10 mL calibrated flasks, following by addition of 1 mL of 1 M sulphuric acid and 1 mL of cerium (IV) sulfate working solution. After mixing the flasks were kept at room temperature for 10 min with occasional swirling, followed by addition of 1 mL methyl orange working solution, filling up to the mark with water and mixed. The absorbance of each solution was measured after 5 min at 507 nm against the reagent blank.

For both methods all measurements were made at 25 °C using temperature controller. The calibration plots were drawn in each case, and then the concentration of the unknown computed from the respective regression equation.

3. Results and Discussion

3. 1. Method A

The absorption spectrum of MOX recorded against water maximum showed absorption peak (λ_{max}) at 292 nm (Figure 2), which means that its determination in the dosage forms based on the direct measurement of its absorption in the UV spectral range is susceptible to potential interferences from the co-extracted common excipients. Therefore, derivatization of MOX with NQS (λ_{max} = 360 nm) was performed under the described optimal ex-

perimental conditions where they react instantaneously with secondary amine in aqueous solutions to give orange colored product exhibiting λ_{max} at 493 nm (Figure 2), which was used for all the measurements. The relative absorption intensity was found to be linearly correlated to MOX concentration.

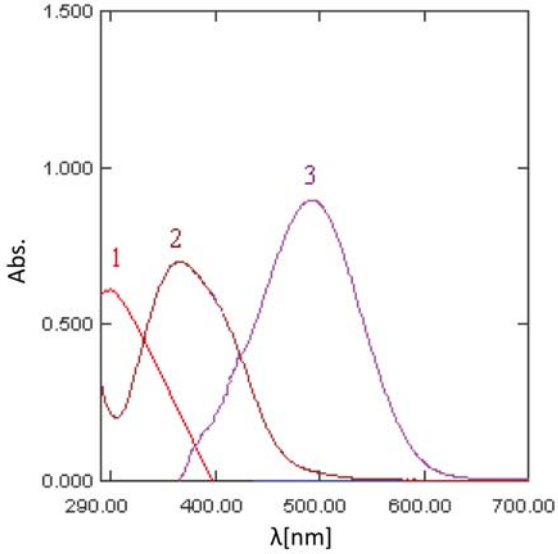

Figure 2. Absorption spectra of MOX (20 μg mL^{-1}) (1) and NQS (0.5%, w/v) (2) against water and the reaction product of MOX (20 μg mL^{-1}) with NQS against reagent blank (3).

Results indicate that the reaction was dependent on the NQS concentration with the highest absorption at NQS concentration of 0.5% (w/v), while higher concentrations of NQS up to 1% had no effect on the absorption (Figure 3). Therefore, a concentration of 0.5% NQS was considered optimum.

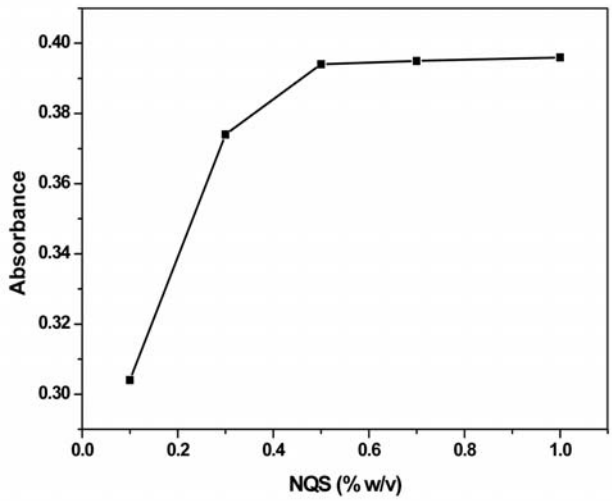

Figure 3. Effect of NQS concentration on the reaction of MOX with NQS (1 mL MOX solution 10 μg mL^{-1}, 1 mL buffer solution pH 11.0, 1 mL NQS) at T = 25°C and reaction time 20 min.

To generate the nucleophile from MOX and activate the nucleophilic substitution reaction, alkaline medium was required. The influence of pH on the absorbance of MOX–NQS product was investigated. The results revealed that the absorbance at pH < 6 were close to 0, indicating that in acidic media MOX has difficulty to react with NQS (Figure 4).

The possible reason is that amino group (–NH of MOX) is protonized and turns into protonated amine salt (–NH^{3+}) when pH of solution is low. So it loses its nucleophilic capacity on NQS, and the nucleophilic substitution reaction does not take place. When pH is more than 6.0, the absorbance of products begin to increase and becomes maximal at pH 11, indicating that the degree of the nucleophilic substitution reaction is maximal. The reason behind this change is that the protonated amine salt (–NH^{3+}) of MOX turn back into amino group when the pH is increased. The higher the pH, the more the protonated amino group (–NH^{3+}) becomes free amino group, and the more easily the nucleophilic substitution reaction proceeds. However, when pH is higher than 11.0 the absorbance of the system of MOX–NQS decrease. Presumably it is due to increase of the amount of hydroxide ion. Hydroxide ion has good nucleophilic ability and can hold back the nucleophilic substitution reaction between MOX and NQS, resulting in the descent of the absorbance of each system. Therefore, pH 11.0 was selected for the optimal experimental conditions.

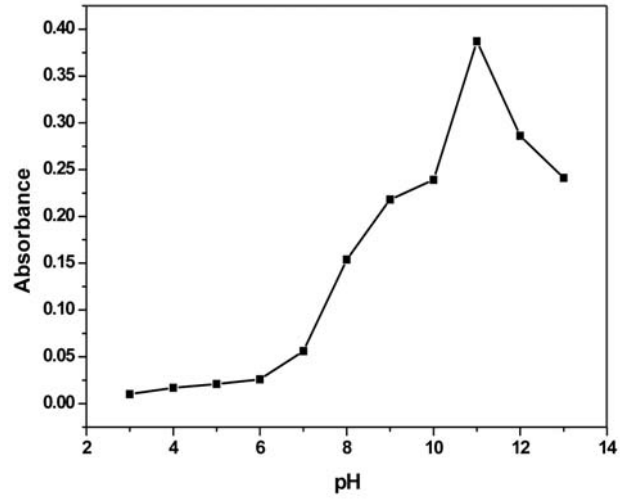

Figure 4. Effect of should be pH on the reaction of MOX with NQS (1 mL of MOX 10 μg mL^{-1}, 1 mL buffer solution, 1 mL NQS) at T = 25 °C and reaction time 20 min.

The effect of reaction time on the formation of the reaction product at room temperature was investigated in the range of 5–50 min (Figure 5). The experimental results show that MOX reacts immediately with NQS. Subsequently, the absorbance begins to increase and keeps stable after 20 min and this time was selected as the optimal.

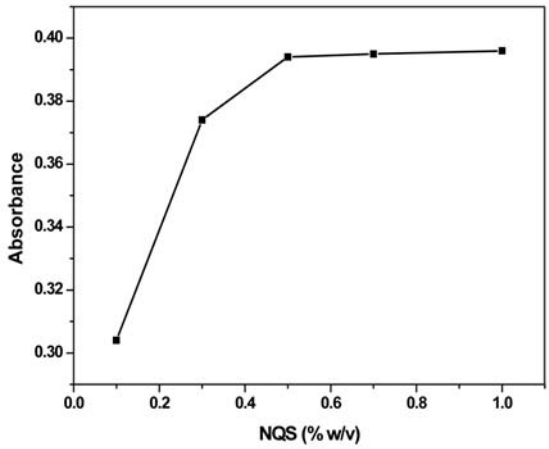

Figure 5. Effect of time on the reaction MOX with NQS at T = 25 °C (1 mL MOX 10 μg mL^{-1}; 1 mL NQS; 1 mL buffer solution pH 11).

The absorption of the mixture solution of 10 μg mL^{-1} MOX was measured after heating in thermostatically controlled water bath. The results revealed that increasing the temperature leads to decrease in the absorption values of the reaction solution. Therefore, T = 25 °C was recommended.

Under the optimum conditions, the stoichiometry of the reaction between MOX and NQS was studied by Job's method (1964).[35] Equimolar aqueous solutions of MOX and NQS (5×10^{-3} mol L^{-1}) were prepared in 10 mL volumetric flasks containing complementary proportions of the two compounds (1:9, 2:8, 3:7, 4:6, 5:5, 6:4, 7:3, 8:2, 9:1) and 1 mL of buffer solution pH 11.0. The symmetrical bell shape of Job's plot indicated that the NQS:MOX ratio was 1:1 (Figure 6).

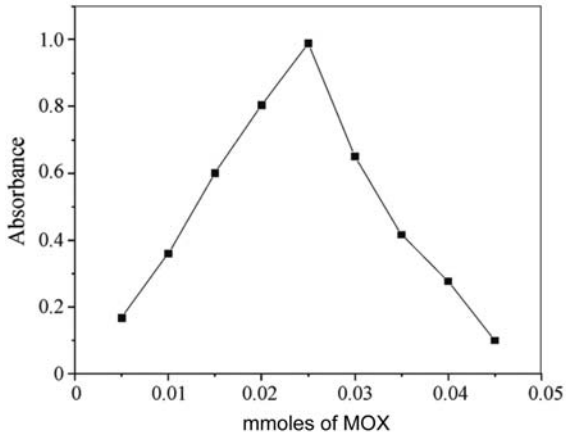

Figure 6. Job's method for NQS with MOX.

3. 1. 1. Reaction Mechanism

NQS is a highly sensitive fluorogenic and chromogenic reagent widely used for derivatization of amines.[36–42] The applications of NQS for determination of pharmaceutical bearing amine group have recently been reviewed by Elbashir *et al.* 2012.[43] MOX contains a secondary amino group, which is a suitable candidate for derivatization by NQS. So MOX can react with NQS in a nucleophilic substitution reaction. At the same time, it has been proved by the Job's method that, the composition of the product is 1:1. So it is concluded that one amino group of MOX substitutes the 4-sodium sulfonate of one NQS molecule to form orange compound. The reaction pathway was postulated to be proceeded as shown in Figure 7.

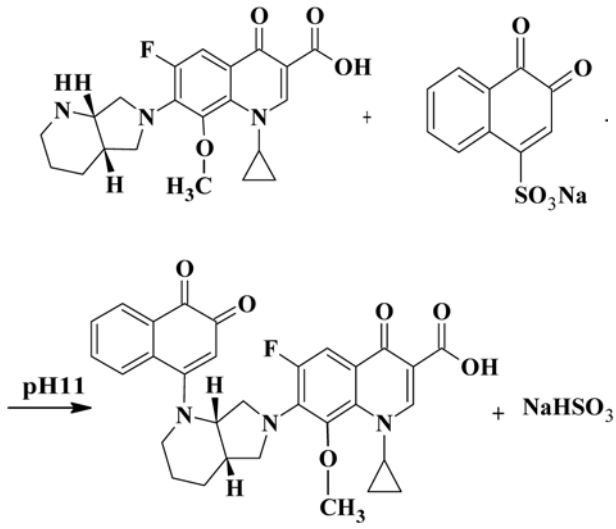

Figure 7. Scheme for the reaction pathway of MOX with NQS

3. 2. Method B

The ability of cerium (IV) sulfate to oxidize MOX and interact with methyl orange is the basis of the developed indirect spectrophotometric method B. In this method, excess of cerium (IV) sulfate reacts with MOX in acidic media, the unreacted oxidizing agent reacts with excess methyl orange and the residual methyl orange is determined by measurement of its absorbance at 507 nm (Figure 8). The absorbance was found to increase linearly with increasing concentration of MOX.

$$\text{MOX} + \text{Ce(IV)}_{\text{excess}} \rightarrow \text{MOX oxidation product} + \text{Ce(III)} + \text{Ce(IV)}_{\text{unreacted}}$$

$$\text{Ce(IV)}_{\text{unreacted}} + \text{MO} \rightarrow \text{oxidation product of MO} + \text{unreacted MO}$$

Preliminary experiments showed that the optimum concentration of methyl orange that could be determined spectrophotometrically was 5 μg mL^{-1}. Therefore, cerium (IV) sulfate concentration of 25 μg mL^{-1} was sufficient to bleach completely the red colour of 5 μg mL^{-1} methyl orange solution under acidic conditions. Hence, MOX reacted with 1 mL of 250 μg mL^{-1} oxidant solution before determining the residual cerium (IV) sulfate.

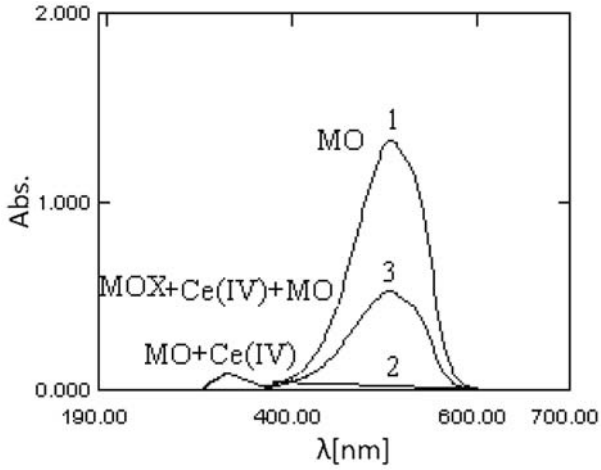

Figure 8. Absorption spectra of solutions containing 1 M H_2SO_4 + methyl orange working solution (1); 1 M H_2SO_4 + methyl orange working solution + Ce(IV) sulfate working solution (2); and 1 M H_2SO_4 + methyl orange working solution + Ce(IV) sulfate working solution + 3.0 μg mL^{-1} MOX (3).

It was observed that regardless the amount MOX added, methyl orange is almost totally bleached if the reagents addition order was dye + MOX + oxidant or dye + oxidant + MOX. The reason for this is that cerium (IV) sulfate did not have enough time to oxidize MOX because it rapidly bleaches methyl orange. Therefore, MOX and oxidant solution must be added first, their addition order does not influence the reaction and methyl orange have to be added after 10 min during which MOX is totally oxidized by $Ce(SO_4)_2$.

It was observed that if methyl orange is immediately added to the solution containing MOX and cerium (IV) sulfate in acidic medium, the resulted solution is bleached rapidly and the absorbance is very low. This can be explained by the fact that MOX oxidation by cerium (IV) sulfate is a time dependent reaction and therefore the influence of the reaction time was studied.

The reactions between MOX and cesium (IV) sulfate was studied quantitatively at different times before adding the indicator and the absorbance was measure. It was observed that the absorbance of these solutions increases with the time up to 10 minutes then it remained constant (data not shown). Thus, for further measurements a reaction time of 10 minutes was selected. The standing time of 5 min was necessary for the bleaching of dye colour by the residual oxidant. The measured colour was stable for hours in the presence of reaction product.

3. 3. Validation of the Methods

Calibration curves for the methods A and B were linear in the ranges 2.5–20.0 μg mL^{-1} and 0.5–3.0 μg mL^{-1} with regression equations Y = –0.007 + 0.11276X (r = 0.9992) and Y = 0.028 + 0.13629X (r = 0.9988), respectively. The molar absorptivities (å) at 493 nm and 507 nm for Methods A and B were 4.9×10^3 and 6.5×10^4 L mol^{-1}, respectively. LOD, LOQ values were found to be 0.75 and 2.50 μg mL^{-1}, respectively, for method A and 0.16 and 0.48 μg mL^{-1}, respectively, for method B.

Accuracy was within 0.48% and 1.76% for methods A and B, respectively, with corresponding intra-day precision expressed as RSD was < 1.6% for the two methods. The results are compiled in Table 1. The inter-day precision expressed as RSD was < 3.7% reflecting the validity of the methods for routine analysis in quality control laboratories.

3. 3. 1. Recovery Studies of the Proposed Methods

To a known amount of the drug in the dosage form, pure drug (the standard) was added at three different levels and the total was found by the proposed methods. Each test was performed in triplicate. The percent recoveries of MOX were in the ranges of 101.28–104.68 and 99.80–100.28 for methods A and B, respectively (Table 2). This shows the absence of interference from tablet excipients.

3. 3. 2. Robustness

The experimental parameters were slightly changed in order to investigate the reliability of the results. The variation of NQS concentration by 0.05, the pH of the buffer by 0.25, temperature by 2 °C and reaction time by 2 minutes did not have a significant effect on the results, indicating the robustness of the developed method.

Table 1: Evaluation of accuracy and precision

Method	Amount of MOX standard [μg]	Amount of MOX Found •[μg/mL]	Range	Relative Error %	SD	RSD (%) (n=5)
A	4.5	4.60	0.001	0.48	0.001	1.58
	7	7.01	0.001	0.13	0.001	0.99
	14	13.96	0.002	0.30	0.001	0.77
B	1.7	1.67	0.012	1.76	0.006	1.30
	2.2	2.19	0.020	0.45	0.008	1.50
	2.7	2.69	0.020	0.37	0.001	1.42

• MOX taken/found, range and SD are in μg/mL

Table 2. Results of recovery study by standard-addition methods, using Avalox tablets (400 mg)

Method	Amount of MOX in formulation, [μg]	Amount of standard MOX added, [μg]	Total found, [μg]	% recovery of pure drug* ± SD (n =3)
A	5.0	4.0	9.196	102.18 ± 0.014
	7.0	8.0	15.70	104.68 ± 0.014
	10.0	8.0	18.23	101.28 ± 0.018
B	1.0	0.5	1.497	99.80 ± 0.008
	1.0	1.0	2.003	100.15 ± 0.002
	1.0	1.5	2.507	100.28 ± 0.002

3. 4. Analysis of MOX in Dosage Forms

It is evident from the above-mentioned results that the two proposed method gave satisfactory results for MOX. Thus its pharmaceutical dosage forms (tablets) were subjected to the analysis of their MOX contents by the proposed methods. The label claim percentage was 102.10 ± 1.83% and 99.50 ± 0.96% for method (A) and (B) respectively (Table 3).

Table 3. Results of analysis of dosage forms in Avalox tablets containing MOX

Method	Label claim, mg/tablet	Amount found mg/tablet	% found* ± RSD (n =5)
A	400	408.4	102.10 ± 1.83
B	400	398.05	99.50 ± 0.96

4. Conclusions

Developed spectrophotometric methods for determination of MOX in its dosage forms are simple, sensitive, selective, rapid and superior to the previously reported spectrophotometric methods[30–31] in terms of their simplicity. Furthermore, all the analytical reagents are inexpensive, have excellent shelf life, and are available in any analytical laboratory. The methods are practical and valuable for routine application in quality control laboratories for analysis of MOX.

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Povzetek

Razvili smo dve enostavni in občutljivi spektrofotometrični metodi (A in B) za kvantitativno določanje moksifloksacina v farmacevtskih pripravkih. Metoda A temelji na reakciji moksifloksacina z 1,2-naftokinon-4-sulfonatom v alkalnem mediju (pH 11,0), pri čemer nastane oranžno obarvani produkt z absorpcijskim maksimumom pri 411 nm. Metoda B pa temelji na oksidaciji moksifloksacina spresežkom cerijevega (IV) sulfata, katerega preostanek določamo po dodatku metiloranža z merjenjem absorbance pri 507 nm. Določeni sta bili molarni absorptivnosti $4{,}9 \times 10^3$ $Lmol^{-1}$ cm^{-1} (A) in $6{,}5 \times 10^4$ $Lmol^{-1}$ cm^{-1} (B). Pri optimiziranih reakcijskih pogojih velja Beerov zakon za koncentracijsko območje moksifloksacina od 2,5–20 μgmL^{-1} za metodo A in 0,5–30 in μgmL^{-1} za metodo B. V okviru validacije obeh metod smo testirali še točnost in natančnost. Metodi smo uspešno uporabili za določanje moksifloksacina v farmacevtskih pripravkih in potrdili njuno uporabnost za rutinske analize v laboratorijih za izvajanje kontrole kakovosti.