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Validated Spectrophotometric Determination of Meloxicam via Azo-Coupling Reactions with Barbituric Acid and 8-Hydroxyquinoline

Khawla S. Abd-Alrassol, *Mohammed K. Mohammed, Rafid H. Alasadi
Department of Chemistry, College of Education for Pure Sciences, University of Basrah, Iraq

**Corresponding author: Mohammed K. Mohammed, E-mail: m.k.moh83chem@gmail.com or mohammed.khalaf@uobasrah.edu.iq*

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Two sensitive, simple, accurate, and rapid spectrophotometric methods have been developed for the estimation of Meloxicam in bulk drug and tablet dosage forms. Method A involves the reaction of Meloxicam with barbituric acid in a basic medium, followed by the addition of sodium nitrite to produce the corresponding primary aromatic amine, while Method B involves the azo-coupling reaction of Meloxicam with 8-hydroxyquinoline in the presence of sodium nitrite in a basic medium. Both methods exhibited excellent linear absorption–concentration dependencies, with correlation coefficients of 0.9998 and 0.9995 for the Method A and the Method B over the concentration ranges of 5.0–25.0 μg/mL and 5.0–35.0 μg/mL, respectively. Additionally, the molar absorptivity and Sandell sensitivity values were $0.94166 \cdot 10^4$ L/(mol·cm) and $6.37 \cdot 10^{-3}$ μg/cm² for the Method A, and $1.326 \cdot 10^4$ L/(mol·cm) and $5.653 \cdot 10^{-3}$ μg/cm² for the Method B, respectively. The Method A demonstrated the recovery within the range of 99.33–101.00 %, while the Method B gave the results within the range of 99.25–101.00 %, with an LOQ of 0.250 μg/mL and 0.503 μg/mL, respectively. These two methods demonstrated the ability to estimate Meloxicam in the pharmaceutical dosage form without interference from tablet additives, with an excellent agreement with the standard method. These characteristics make the proposed methods appropriate for the quality control of Meloxicam in bulk drug and tablet dosage forms.

Keywords: spectrophotometric characterization, azo dye, meloxicam drug, pharmaceutical formulation

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Валидированное спектрофотометрическое определение мелоксикама с помощью реакций азосоединения с барбитуровой кислотой и 8-гидроксихинолином

Хавла С. Абд-Альрассол, *Мохаммед К. Мохаммед, Рафид Х. Аласади
Университет Басры, Колледж фундаментальных наук, Химический факультет, Ирак

**Адрес для переписки: Мохаммед К. Мохаммед,, E-mail: m.k.moh83chem@gmail.com или mohammed.khalaf@uobasrah.edu.iq*

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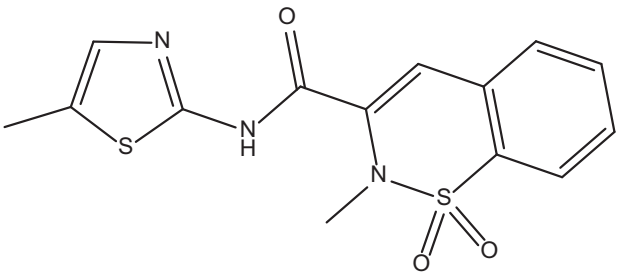
Разработаны два чувствительных, простых, точных и экспрессных спектрофотометрических метода количественного определения мелоксикама в фармацевтической субстанции и таблетированной лекарственной форме. Метод А основан на реакции мелоксикама с барбитуровой кислотой в щелочной среде с последующим добавлением нитрита натрия для получения соответствующего первичного ароматического амина, в то время как метод В включает реакцию азосочетания мелоксикама с 8-оксихинолином в присутствии нитрита натрия в щелочной среде. Оба метода продемонстрировали отличную линейную зависимость поглощения от концентрации с коэффициентами корреляции 0,9998 для метода А и 0,9995 для метода В в диапазонах концентраций 5,0–25,0 мкг/мл и 5,0–35,0 мкг/мл соответственно. Кроме того, значения молярного коэффициента поглощения и чувствительности по Санделлу составили $0{,}94166 \cdot 10^4$ л/(моль·см) и $6{,}37 \cdot 10^{-3}$ мкг/см² для Метода А, и $1{,}326 \cdot 10^4$ л/(моль·см) и $5{,}653 \cdot 10^{-3}$ мкг/см² для Метода В. Метод А характеризуется степенью извлечения в диапазоне

от 99,33 до 101,00 %, в то время, как Метод Б — в диапазоне от 99,25 до 101,00 % при пределе количественного определения (LOQ) 0,250 мкг/мл и 0,503 мкг/мл соответственно. Оба метода подтвердили возможность определения мелоксикама в фармацевтической лекарственной форме без влияния вспомогательных веществ таблеток при отличном согласовании со стандартным методом. Данные характеристики делают предложенные методы подходящими для контроля качества мелоксикама в фармацевтической субстанции и таблетированной форме.

Ключевые слова: спектрофотометрическая характеристика, азокраситель, лекарственный препарат мелоксикам, лекарственная форма

INTRODUCTION

Meloxicam (MEL, Scheme 1) is a non-steroidal anti-inflammatory drug (NSAID) that has a higher affinity for cyclooxygenase-2 (COX-2) enzymes and is used for the treatment of various inflammatory diseases. Despite its medicinal importance, MEL has low solubility at low pH, which may lead a gastrointestinal side effects. This emphasizes the need for precise control of dosage forms [1–5].



Scheme 1. Chemical structure of the meloxicam drug

A range of analytical techniques have been proposed for the estimation of MEL, such as spectrophotometry [6–13], high-performance liquid chromatography (HPLC) and reversed-phase high-performance liquid chromatography (RP-HPLC) [14–21], flow injection analysis [22–25], electrochemical analysis [26, 27], voltammetry [28,29], polarography [30], and several coupling reactions [31–33]. Although the above-mentioned chromatographic techniques provide high accuracy, these techniques require expensive instruments, skilled personnel, a large analysis time, and organic solvents. Although some previously reported spectrophotometric methods are simple, they typically involve complex steps, use organic solvents, exhibit low sensitivity, and lack adequate validation, which restricts their applicability, especially in resource-constrained environments [34, 35].

For this reason, there is a recognised need for simpler, environmentally friendly, and more sensitive spectrophotometric methods that operate in aqueous or semi-aqueous systems, exhibit improved chromophoric responses, and are fully validated for quality control purposes. In this investigation, this need is met by developing two novel spectrophotometric methods for meloxicam based on its diazotisation reaction, followed by azo coupling with barbituric acid and 8-hydroxyquinoline (oxyquinoline) as coupling reagents [36].

Barbituric acid provides an activated methylene group that allows for effective azo coupling through electrophilic azo compounds, resulting in intensely colored and stable azo dyes that have strong visible region absorption properties. In a similar way, 8-hydroxyquinoline reacts under alkaline conditions to form anionic species that have increased electron density at the coupling site, thus forming azo compounds that have increased conjugation and molar absorptivity properties. Compared with conventional reagents such as sulfanilic acid, these compounds exhibit greater sensitivity, stability, and signal-to-noise ratio, thereby reducing background interference [37, 38].

The current study has demonstrated, for the first time, the application of barbituric acid and 8-hydroxyquinoline as a coupling agent for the estimation of meloxicam. This method has better chromogenic activity, greener chemistry, and comprehensive method validation. The developed methods have demonstrated greater accuracy, precision, speed, and cost-effectiveness than existing methods. This study offers greater practical applicability than existing methods, particularly for pharmaceutical quality control laboratories without sophisticated chromatographic equipment.

EXPERIMENTAL

All spectral measurements were recorded using a Shimadzu UV–Vis Double Beam Spectrophotometer (model 2450) calibrated with matching quartz cells.

Chemicals and reagents

All the chemicals used were of analytical reagent grade, and double-distilled water was used in all experiments. The purity of Meloxicam was established by melting-point determination and infrared spectroscopy, which indicated the absence of impurities. Meloxicam, with a purity of 99 %, was kindly provided by Awamedica Company for Drug Industries and Medical Applications, Awa, Erbil, Iraq. Sodium hydroxide, sodium nitrite, and hydrochloric acid were purchased from Sigma, whereas barbituric acid, 8-hydroxyquinoline, and ethanol, which was of analytical grade, were purchased from Merck, England. Pharmaceutical formulations were purchased from local pharmacies.

Standard solutions

A standard stock solution of Meloxicam (1.000 μg/mL) was prepared by dissolving 0.1 g of the drug in 100 mL of distilled water in a conical flask. Furthermore, 20 mL of 2.0 M sodium hydroxide was added, and the mixture was heated in a boiling wa-

ter bath for 30 minutes. The resulting solution was cooled, and the pH was adjusted to 9 with 2.0 M hydrochloric acid. The solution was then transferred to a 100 mL volumetric flask and diluted with distilled water to obtain the standard stock solution.

Sample preparation

Ten tablets of Meloxicam were crushed and made into a solution. A measured quantity of this solution was placed in a 50 mL volumetric flask, and 40 mL of acetone was added. The mixture was shaken for 30 minutes using a water bath shaker, then diluted with acetone to volume. It was then filtered, and 25 mL of the solution was placed in a 100 mL conical flask and evaporated to dryness in a boiling water bath. The concentration of Meloxicam was then calculated according to the standard procedure for the preparation of a stock solution [39].

Assay procedures:

Method A:

Standard solution of Meloxicam: For the preparation of a 100 µg/mL solution, an appropriate volume of the stock solution was diluted to 100 mL in a calibrated volumetric flask. Aliquots of 2 mL each were transferred into a 25 mL volumetric flask, and 5 mL of 0.1 % barbituric acid, 2.0 mL of 0.5 M sodium hydroxide, 1.5 mL of 1.0 % sodium nitrite, and 0.5 mL of 0.5 M hydrochloric acid were added. The volume was then adjusted to the mark with distilled water. The absorbance was read at 520 nm with a 1.0 cm quartz cell. A blank solution was also prepared in the same way without Meloxicam.

Method B:

Standard solution of Meloxicam: This entailed preparing a 100 µg/mL solution from the stock solution by making up to 100 mL in a calibrated volumetric flask. Two millilitres of this solution were then added to a 25 mL volumetric flask and mixed with 2.0 mL of 0.5 M sodium hydroxide solution, 5 mL of 0.1 % solution of 8-hydroxyquinoline, 0.5 mL of 0.5 M hydrochloric acid solution, and 1.5 mL of 1.0 % sodium nitrite solution. The solution was then made up to the mark with distilled water, and its absorbance at 360 nm in a 1.0 cm quartz cell was recorded in the absence of Meloxicam.

Optimization of experimental parameters

Several factors that could influence the absorbance, stability, and sensitivity of the coloured dyes were investigated. Because each spectrophotometric method has ideal conditions, parameters were investigated to identify the optimal conditions for measurement.

RESULTS AND THEIR DISCUSSION

Absorption spectra

The absorption spectra of the azo compounds and the blank are presented in Fig. 1 and 2. The azo dyes had a peak absorbance of 520 nm for Method A and 360 nm for Method B, whereas the blank had negligible absorption for the same wavelengths.

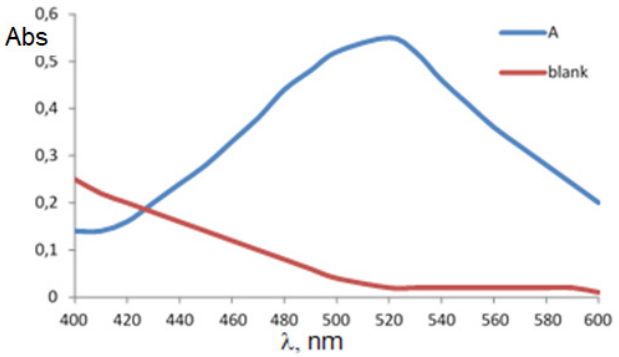


Fig. 1. Absorption spectrum for Method A: (A) azo compound against blank (distilled water)

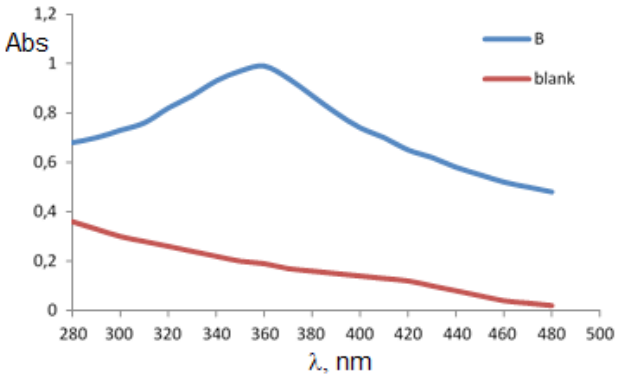


Fig. 2. Absorption spectra for Method B: (B) azo compound against blank (distilled water)

Study the effect of sodium nitrite concentration

The effect of different concentrations of sodium nitrite on the colour intensity of azo dyes was evaluated by Methods A and B, as shown in Fig. 3. Fixed amounts of drug samples reacted with various amounts of 0.1 % $NaNO_2$ solution. The reaction was complete at 1.5 mL and 1.0 mL of 0.1 % $NaNO_2$ solution for Methods A and B, respectively, where the absorbance was at its peak, as shown in Fig. 4.

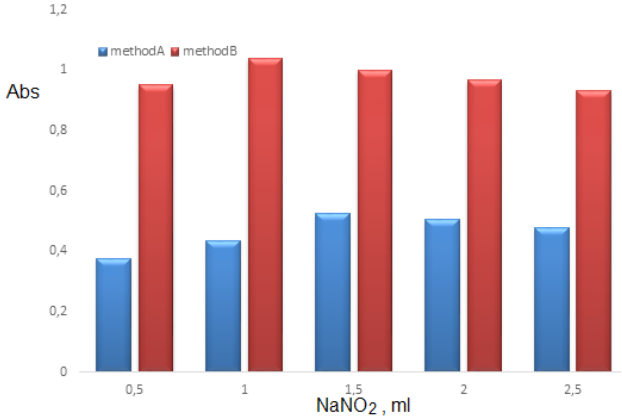


Fig. 3. Effect of volume of 1.0 % $NaNO_2$ solution on absorption in Method A

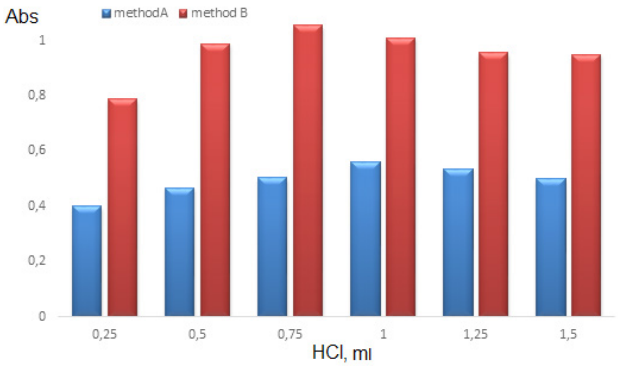


Fig.4. Effect of volume of hydrochloric acid solution on color intensity in Method A and Method B

The impact of the acid type

Various acids, including hydrochloric, acetic, sulfonic, and nitric acids, were studied for optimal absorption. The results of hydrochloric acid were superior. As shown in Fig. 4, the colour intensity increased with increasing HCl volume up to 1.0 mL, and then decreased. Hence, 1.0 mL of 0.5 M hydrochloric acid was used in further experiments.

Study the effect of the basic solution

It is worth noting that azo dye formation is usually carried out under basic conditions. The influence of different bases, such as 0.5 M KOH, NaOH, and Na_2CO_3, on absorbance was studied. It was noted that sodium hydroxide showed the best absorbance. Consequently, the influence of sodium hydroxide concentration on absorbance was studied by adding different volumes of 0.5 M NaOH solution (0.25–1.5 mL) to a known volume of the drug. It was noted that maximum absorbance was achieved by adding 0.5 mL of NaOH solution for Method A and 1.0 mL for Method B. These volumes were then used for further study because they exhibited high colour intensity with minimal absorbance in the blank solution. Figure 5 illustrates the relationship between NaOH concentration and colour intensity for both methods.

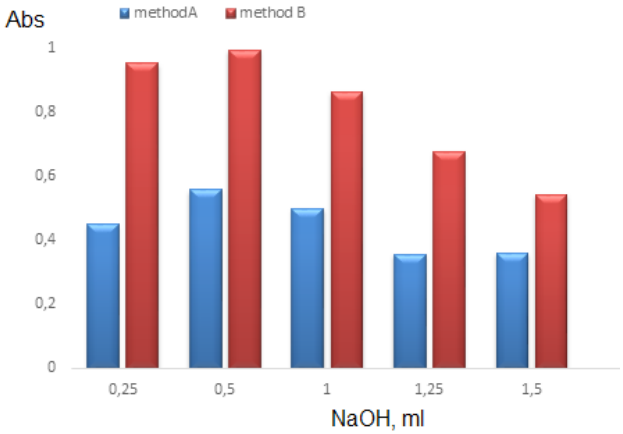


Fig. 5. Effect of varying the volume of 0.5 M sodium hydroxide solution on the absorbance in Methods A and B, using 15 µg/mL Meloxicam, 1 mL of 0.5 M hydrochloric acid, and 1.5 mL of 0.1% sodium nitrite solution

Calibration Curves

For the proposed methods, the calibration curves showed linearity in the concentration range of 5.0–25.0 µg/mL for Method A and 5.0–35.0 µg/mL for Method B. For the calibration curves of the proposed methods, five concentrations were used, with four replicates per concentration. The regression equations and optical properties of the proposed methods are presented in Table 1.

Table 1

The optical characteristics of the proposed Methods A and B

Parameters	Method A	Method B
λ_{max}, nm	520 nm	360 nm
Beer's law range (µg/ml)	5.0–25.0	5–35
Detection coefficient (R^2)	0.9998	0.9995
Molar absorptivity (L/mole.cm)	0.941×10^4	1.326×10^4
Sandell's sensitivity (µg/cm²)	6.3×10^{-3}	5.653×10^{-3}
Detection of limits (µg/ml)	0.075	0.15
Intercept (a)	0.0007	0.0233
Slope	0.0362	0.0517

The stability of the dye over time

The stability of the dye was assessed by measuring the absorbance of Meloxicam at three concentrations (5.0, 10, and 20 µg/mL) at specified time intervals using the proposed methods. The results showed that the stability of the azo dyes was maintained for at least 2 hours, with no significant change in the analytical signal observed over 5–120 minutes after reagent mixing. The stability of the dye was shown in Method A and Method B, regardless of the concentration of Meloxicam.

Limit of detection (LOD) and limit of quantification (LOQ)

The Limit of Detection (LOD) and Limit of Quantification (LOQ) were determined according to IUPAC guidelines [40] using the following formulas:

$$LOD = \frac{3\sigma}{S},\ LOQ = \frac{10\sigma}{S}.$$

Here, σ is the standard deviation of blank absorbance values, and S is the slope of the calibration curve [41, 42]. The high molar absorptivity, along with low S and LOD values, indicates the high sensitivity of the proposed methods. The LOD values for Methods A and B are 0.075 µg/mL and 0.15 µg/mL, and the LOQ values are 0.250 µg/mL and 0.503 µg/mL. These values represent the lowest concentrations that can be measured accurately and precisely by analysing samples containing a known amount of analyte.

The Precision and Accuracy

The accuracy and precision of the suggested methods were investigated by analyzing pure

Table 2

Determination of accuracy and precision of the Method A on pure drug samples

Concentration of standard solution taken (μg/ml)	Concentration of standard solution found (μg/ml)	*RE*, %	*Recovery, %	*RSD*, %
5	5.05	1.00	101.00	1.25
10	9.95	0.50	99.5	1.33
15	14.90	0.60	99.33	0.95

*Each value is the mean of six observations

Table 3

Determination of accuracy and precision of the Method B on pure drug samples

Concentration of standard solution used (μg/ml)	Concentration of standard solution found (μg/ml)	*RE*, %	*Recovery, %	*RSD*, %
10	10.09	0.90	100.90	1.13
15	15.15	1.00	101.00	1.07
20	19.85	0.75	99.25	0.99

*Each value is the mean of six observations

meloxicam solution at different concentration levels within the predetermined working range. Six replicates of each concentration were performed for each method: 5, 10, and 10 μg/mL for method A and 5, 15, and 20 μg/mL for method B. The accuracy of the suggested methods was evaluated using relative error (% *RE*), whereas precision was evaluated using relative standard deviation (% *RSD*). The mean values of accuracy and precision for method A were 1.17 and 0.70 %, respectively, whereas for method B, they were 1.13 and 0.88 %, respectively. The recovery results were excellent for methods A and B, with values ranging from 99.33 to 101.00 % and 99.25 to 101.00 %, respectively (Tables 2 and 3), indicating good accuracy and precision for the proposed methods.

Interference

The effect of commonly used excipients on method selectivity was also investigated through measurement of absorbance for solutions containing 2 mL of each excipient at a concentration of 100 μg/mL, and 1 mL of drug at a concentration of 50 μg/mL. The volume of each solution was made up to 10 mL. The results showed that these excipients have little effect on the intensity of the produced colored compound, thus confirming the selectivity of the proposed methods.

Effect of Temperature

The effect of temperature on the intensity of dye colour was also investigated at temperatures ranging from 15–35 °C. According to the results, the optimal temperature for the highest colour intensity is 25 °C. Temperatures above 25 °C caused turbidity; therefore, 25 °C was used in all experiments.

Stoichiometric ratio for the formation-colored dyes

The stoichiometry of the product formed was investigated using the continuous variation method, commonly known as Job's plot, to determine the properties of the formed complex and the extent of drug binding to the diazotized reagent [43]. As illustrated in Fig. 6, both reactions have a stoichiometry of 1:1 for the drug and reagent, thus confirming the formation of mono-azo compounds for Methods A and B. The mechanisms for the formation of the dyes are illustrated in Schemes 2 and 3.

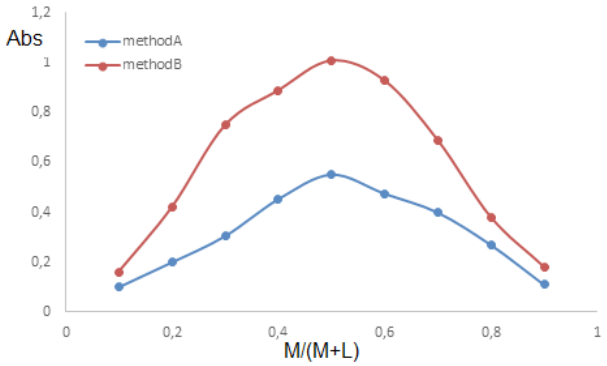


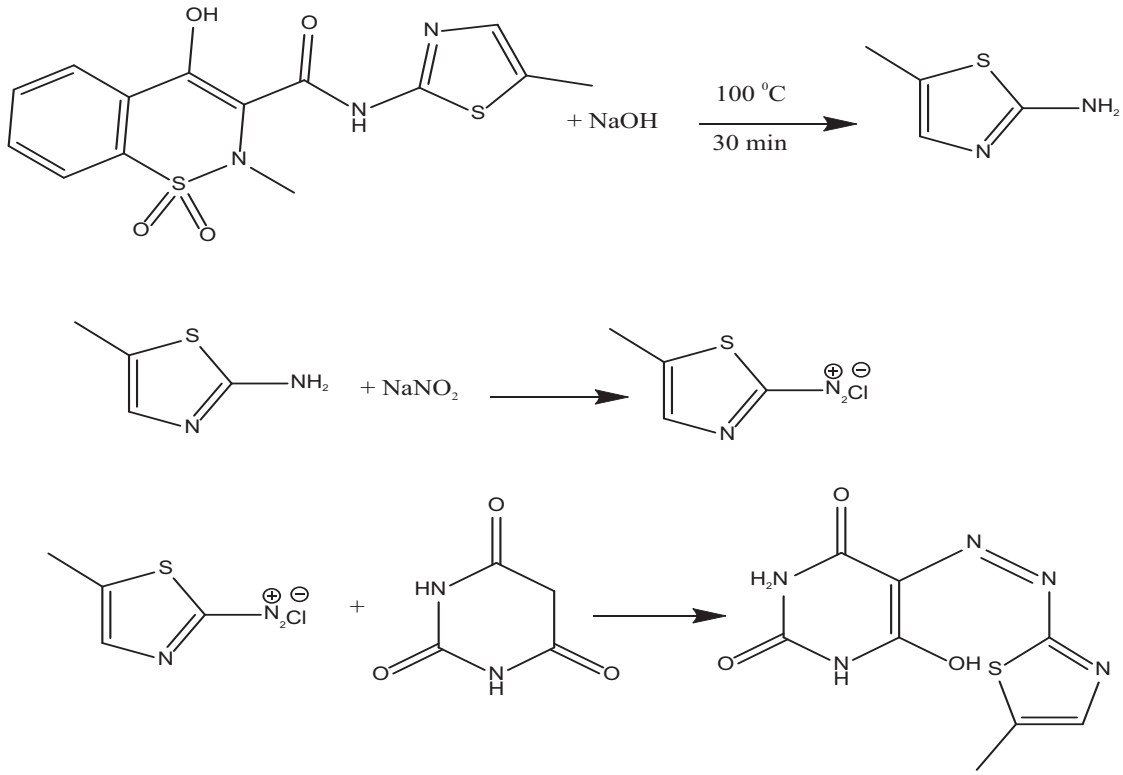
Fig. 6. Continuous variation plots for the azo compounds in Methods A and B

Application

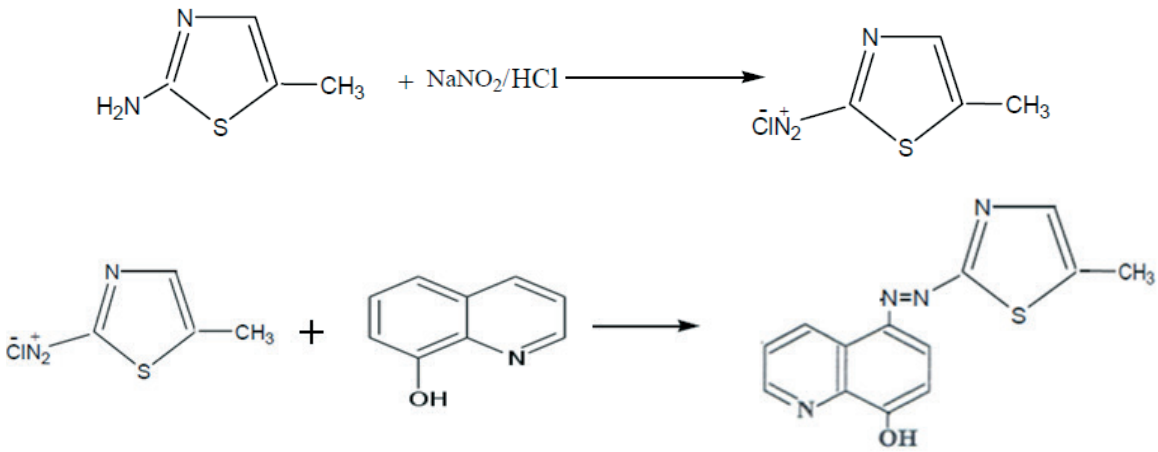
The proposed methodologies were applied to pharmaceutical formulations of Meloxicam, such as Awamedica-Meloxicam Awa (Erbil, Iraq, 7.5 mg) and Boehringer Ingelheim-Mobic (Germany, 15 mg), in tablet form. The results of these analyses validate the efficacy of these methodologies in drug estimation. The analyses were performed at three concentrations, with six replicates at each concentration (n = 6). The results are given in Tables 4 and 5 in mean and standard deviation (99.10 ± 0.047) %. The assay results indicate that these methodologies are accurate and precise for drug estimation.

Comparison

The proposed methods have been compared with the previously published analytical techniques, and the major performance characteristics of the pro-



Scheme 2. The suggested mechanisms for Method A



Scheme 3. The suggested mechanisms for Method B

Table 4

Results of applying Method A for quantifying meloxicam in a pharmaceutical formulation

Pharmaceuticals tablets	Concentration ppm		*RE, %	*Recovery, %	*RSD, %	Reference method (HPLC) [21]
	Taken	Found				
Awamedica-Meloxicam Awa, Erbil, Iraq (7.5 mg)	5	5.05	1.00	101.00	1.11	% Assay ± SD = (99.10 ± 0.047) *RSD*, % (0.06) *RE*, % (0.64)
	10	9.95	0.50	99.50	0.95	
	15	15.05	0.33	100.33	1.05	
Boehringer Ingelheim Mobic, Germany (15 mg)	5	4.98	1.40	101.40	1.33	
	10	9.99	1.00	99.90	0.75	
	15	15.05	0.47	100.46	1.00	

*Average of six times

Table 5

Results of applying Method B for quantify meloxicam in a pharmaceutical formulation

Pharmaceuticals tablets	Concentration ppm		*RE, %	*Recovery, %	*RSD, %	Reference method (HPLC)
	Taken	Found				
Awamedica-Meloxicam Awa, Erbil, Iraq (7.5 mg)	5	5.03	0.60	100.60	1.05	% Assay ± SD (99.47±0.012)
	10	10.10	1.00	101.00	1.12	
	15	14.96	0.36	99.73	0.98	*RSD*, % (0.08)
Boehringer Ingelheim Mobic, Germany (15 mg)	5	5.07	1.40	101.40	1.22	
	10	10.07	0.70	100.70	0.99	*RE*, % (0.74)
	15	14.90	0.67	99.33	0.89	

*Average of six times

Table 6

Comparison of suggested Methods A and B with other reported techniques

Methods	λ_{max}, nm	Linear range, μg ml^{-1}	*RSD*, %	Recovery, %	LOD, μg ml^{-1}	Literature method Ref.
Method A	520	5–25	1.25–0.95	101.00–99.33	0.075	–
Method B	360	5–35	1.13–0.99	101.00–99.25	0.15	–
Literature method spectroscopy	708	0.1–11	0.25–0.73	98.7–99.5	0.009	[44]
Literature method FIA	530	10–160	1.2	97.00–104.00	6.00	[45]
Literature Method (HPLC)	355	0.5–20	0.45–0.80	99.20–100.30	0.02	[46]

posed and literature methods are listed in Table 6. This comparison reveals that although the analytical properties of previously published methods are comparable, the current spectrophotometric methods offer considerable advantages in practical applicability for quality control testing. Methods A and B have been compared with conventional spectrophotometry, flow injection analysis (FIA), and high-performance liquid chromatography (HPLC). Both methods exhibit high accuracy and precision for quantitative analysis, with recoveries between 99.25 % and 101.00 % and relative standard deviations (*RSD*s) < 1.3 %, confirming their suitability for quantitative determinations.

Although conventional spectrophotometry is relatively easy, it has been shown that it sometimes lacks sensitivity and may require several steps, as well as organic solvents, which may be detrimental to the efficiency of the analysis. Flow injection analysis offers a wide linear range but has a higher detection limit and requires specialised equipment that may not be readily available. High-performance liquid chromatography has the lowest detection limit, but its routine use is limited by high operating costs, specialised operating skills, and the large amounts of organic solvent required.

In contrast, Methods A and B achieve the goals of simplicity, environmental friendliness, cost-effectiveness, and acceptable sensitivity via the use of conventional UV–Vis spectrometry and predominantly aqueous solvents. Method A shows increased sensitivity (LOD = 0.075 μg·mL^{-1}) and better colour stability, making it applicable for low-level analyses, while Method B offers a wider range of working concentrations (5–35 μg·mL^{-1}), which is advantageous for analyses involving high doses. Although Methods A and B do not achieve better sensitivity than HPLC, a balance is achieved in terms of analytical power and practical simplicity. Therefore, Methods A and B are reliable, cost-effective, and environmentally friendly, particularly for quality control analyses in pharmaceutical settings where sophisticated chromatographic equipment is not readily available.

Evaluation of the Best Spectrophotometric Method (A or B) for Meloxicam Determination

In this investigation, spectrophotometric methods A and B were developed for the quantitative determination of meloxicam in tablet formulations. Both methods were accurate, precise, and reproducible, as indicated by recovery rates of 99.25 %–101.00 % and relative standard deviations of less than 1.3 %. Method A, which has a λ_{max} of 520 nm, produced intensely colored azo compounds with high molar absorption coefficients and good colour stability, which greatly increased the sensitivity of the analytical method. Method B, with a λ_{max} of 360 nm, exhibits a wider linear dynamic concentration range of 5–35 μg/mL, thereby increasing the method's versatility for assaying higher drug concentrations.

Although it is a simple, quick, and cost-effective technique, it is also environmentally safe. However, Method A is more sensitive, with a lower detection limit of 0.075 μg/mL, whereas Method B has a limit of 0.15 μg/mL. In addition, the stability of the colored complex of Method A minimizes errors that may occur as a result of photodegradation of the complex, and therefore, it is more suitable for quality control analysis of meloxicam at low concentration levels.

Although Method B has a wider linear dynamic range, Method A offers the most favourable balance of sensitivity, accuracy, and simplicity for the spectro-

photometric determination of meloxicam in pharmaceutical formulations.

CONCLUSION

This study reports the successful development of two new spectrophotometric methods, Methods A and B, for the quantitative determination of meloxicam via the formation of an azo derivative. Both methods were characterized as simple, sensitive, selective, accurate, and rapid. In addition, Method A showed linearity within a range of 5–25 μg/mL, and Method B showed linearity within a range of 5–35 μg/mL. Both methods also showed high correlation coefficients of 0.998 and 0.995, respectively. Additionally, high molar absorptivity values were obtained for both methods, namely 0.9416×10^4 $L \cdot mol^{-1} \cdot cm^{-1}$ for Method A and 1.326×10^4 $L \cdot mol^{-1} \cdot cm^{-1}$ for Method B. In terms of sensitivity, Sandell's sensitivity values were obtained as 6.37×10^{-3} $\mu g \cdot cm^{-2}$ and 5.653×10^{-3} respectively, indicating high analytical sensitivity.

The limits of detection for the methods were 0.075 μg/mL for Method A and 0.15 μg/mL for Method B, demonstrating the ability of the proposed methods to accurately detect low concentrations of meloxicam. In the recovery studies, the results for Method A ranged from 99.33 % to 101.00 %, whereas for Method B, the results were 99.25 %-101.00 %, thereby confirming excellent accuracy and precision in the presence of common pharmaceutical excipients.

The methods were successfully demonstrated to be applicable to commercial pharmaceutical formulations of meloxicam, and no interfering effect of commonly used excipients was found. The results confirm the robustness and versatility of the proposed methods for quality control analyses in pharmaceutical laboratories. In addition, spectrophotometric methods are more environmentally friendly compared to chromatography because of their use of small amounts of solvents.

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