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Development of a Spectrophotometric Method to determination Minoxidil by Diazotization-Coupling reaction with Sulfanilic acid

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Abstract

A straightforward, quick, and accurate spectrophotometric method using diazotized sulfanilic acid as reagent to quantify minoxidil in both its pharmaceutical and pure forms.In this method, p-aminobenzen sulfonic acid (sulfanilic acid) was diazotized with low molar sodium nitrite ($NaNO_2$) solution in the presense of hydrochloric acid (HCl) to produce corresponding diazonium salt subsequently coupling with minoxidil drug , a yellow color of azo dye which was water soluble and has a maximum absorption (λmax) at 400 nm compared a reagent blank. An under best conditions, the linearity of Beer's law was estimated in the concentration range of (5-40) µg/ml with an exceptional determination Linearity coefficint (R^2= 0.9993) and molar absorptivity of $4.624*10^3$ l/mol.cm. The detection limit (LOD), quantitation limit (LOQ) and precision ranges (RSD%) were calculated equal to 0.012 , 0.040 µg/mLand 0.16% to 1.59%, respectively. The stoichiometry was showed to be 1:1 (minoxidil: diazotized Sulfanilic acid). The procedure has been utilized successfully to determination of minoxidil in tablets.

Introduction:

Minoxidil a white drug powder, the chemical formula $C_9H_{15}N_5O$ and molecular weight 209.25 g/mol chemically known as (2,4-diamino-6-piperidinopyrimidine 3-oxyde), has been used to treat severe hypertension in patients whose blood pressure is not adequately controlled with combinations of conventional antihypertensive medications. About 80% of patients showed good results [1], It works to hyperpolarize the cell membrane via activating potassium channels in the peripheral artery's smooth muscles [2]. The drug was showed side effects, including excessive hair growth, which was one of the reasons for discovering its effectiveness in treating androgenic alopecia (AGA), first in males and then in females [3,4].

The development of analytical methods is an important and crucial step in pharmaceutical analysis because it plays a major role in drug development and pharmaceutical quality control. This process includes selecting appropriate techniques and optimizing various parameters to ensure accurate and reliable analysis of pharmaceutical samples. Many literatures have revealed various analytical methods for the determination of minoxidil, such as high-performance liquid chromatography [5-7], electrochemistry [8-10], LC–MS[11] and Spectrophotometric methods [12-14].

Experimental

Apparatuses

Bruker ALPHA II FTIR Spectrometer, SHANGFEN N4S single beam UV-Visible Spectrophotometere with 1.0-cm matched quartz cells ,Electronic balance (Sartorius balance) was used for weighing chemical materials and pH meter.

Reagent and chemical solutions

all experiments were used materials high pure chemical:

Standard concentration of minoxidil (100 ppm):

A 10 mg dose of minoxidil that was acquired by the pharmaceutical industry from the Macklin Company (New Delhi -India) was dissolved in a quantity of ethanol, following that, the contents were transferred to a 100 mL standard flask and the volume was adjusted with distilled water.

sulphamic acid (3% W/V) solution:

Using a standard flask, 3 g of sulphamic acid was dissolved in 100 mL of distilled water..

sulfanilic acid solution (10 ppm):

A 10 mg of sulfanilic acid was dissolved in 1000 mL of distilled water using standard flask.

Sodium nitrite (0.2M):

Weighing and dissolving 1.38 g of sodium nitrite in distilled water to a final volume of 100 ml. Hydrochloric acid (1 M):

Distilled water was used to dilute 8.35 ml of 11.97 M concentrated hydrochloric acid to 100 ml.. phosphoric acid solution (1M):

A 6.29 ml of (15.9 M) concentrated phosphoric acid was diluted to 100ml using distilled water and bringing the volume to the mark. glacial acetic acid solution (1 M): A 5.75 ml of (17.4 M) concentrated glacial acetic acid was diluted to 100 ml using distilled water and bringing the volume to the mark.

Buffer solution

The buffer solution was prepared at pH = 1.3, where 1.4912 g of potassium chloride was dissolved with distilled water and transferred to a 100 ml standard flask and completed to the mark, take 50 ml from buffer solution into a 200 ml standard flask and add 67.2 ml of hydrochloric acid 0.2M, then completed to the mark with distilled water [15].

Analysis of minoxidil in the pharmaceutical preparations

The pharmaceutical preparation Minoxidil Tablets, produced by Steadfast Medishield Pvt Ltd, New Delhi - India. Each tablet contains 5 milligrams. 20 tablets were ground and dissolved in 25 ml of ethanol. The solution was filtered and transferred to a 100 ml volumetric flask then compeleted to the mark by distilled water to prpduce 1000 micrograms/ml. a

solution of 100 micrograms/ml was prepared, where 10 ml was taken, to a 100 ml volumetric bottle, and compeleted by distilled water to the mark.

Procedure method

One mL (0.2M) sodium nitrite was added to each flask in an ice path (0–5°) with a stirrer after a series of 10 mL standard flasks, 1 mL (10) µg/ml of sulfanillic acid, and 1 mL of 1 M hydrochloric acid were cooling, add 0.25 mL (3%) sulphamic acid to remove residual nitrite, After 2 minutes add minoxidil containing (5-40) µg/ ml, A yellow solution was produced. After diluting the contents of the flasks with distilled water to the appropriate level, the samples' absorbance at 390 nm was measured in comparison to a blank solution [16].

Findings and Discussion

Analytical variables' effects on Absorption were studied, and the best circumstances were chosen to create and stable the azo dye.

Fundamentals of the Approach

As indicated by the following proposed mechanism of interaction (Figure 1), the basic idea of the suggested method is based on the diazotization of sulfanilic acid in an acidic medium and reaction with an amount of sodium nitrite to form diazonium salt, which was then coupled with minoxidil drug to produce a yellow azo dye [17] in water soluble, whene gave maximum absorption at 390 nm.

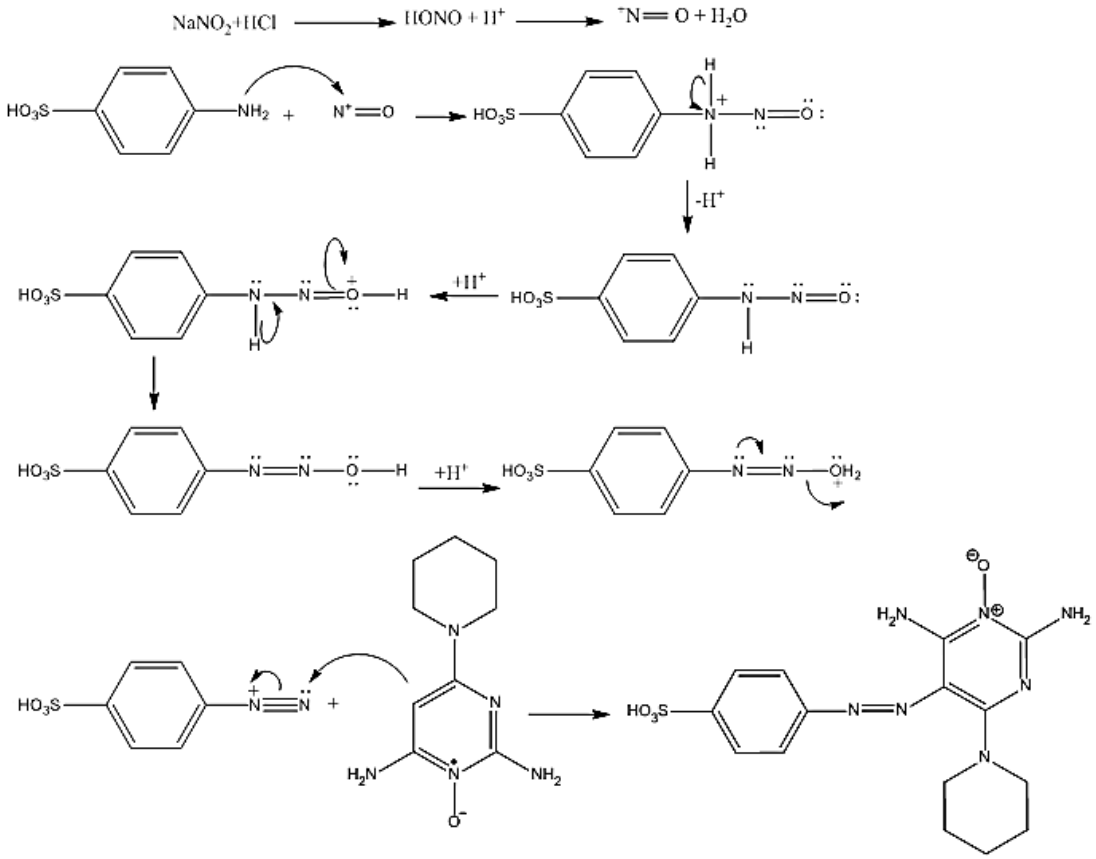

Figure 1. *The Proposed Reaction's Mechanism*

FT-IR spectral data

The infrared spectrum of the reagent, drug, and azo compound was compared (Figure 2,3,4), and the spectrum showed a new band at 1444 cm^{-1} to the azo group -N=N- and this is consistent with the literature [18], in addition to the appearance of a broad band at 3331 cm^{-1} due to the presence of water in it solution and maybe form hydrogen bonds.

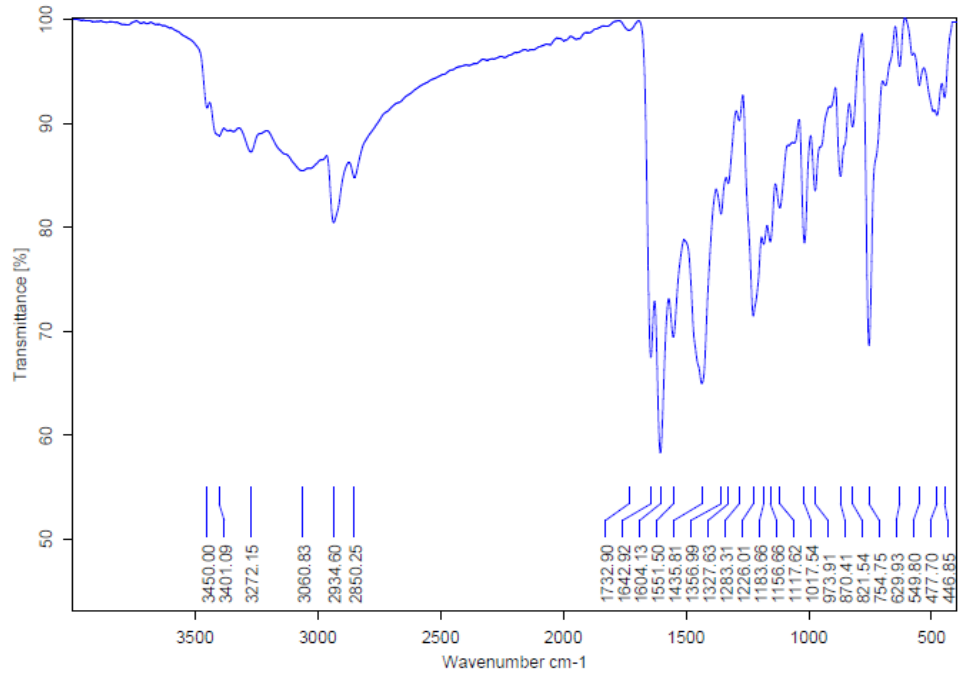

Figure 2. *Minoxidil drug*

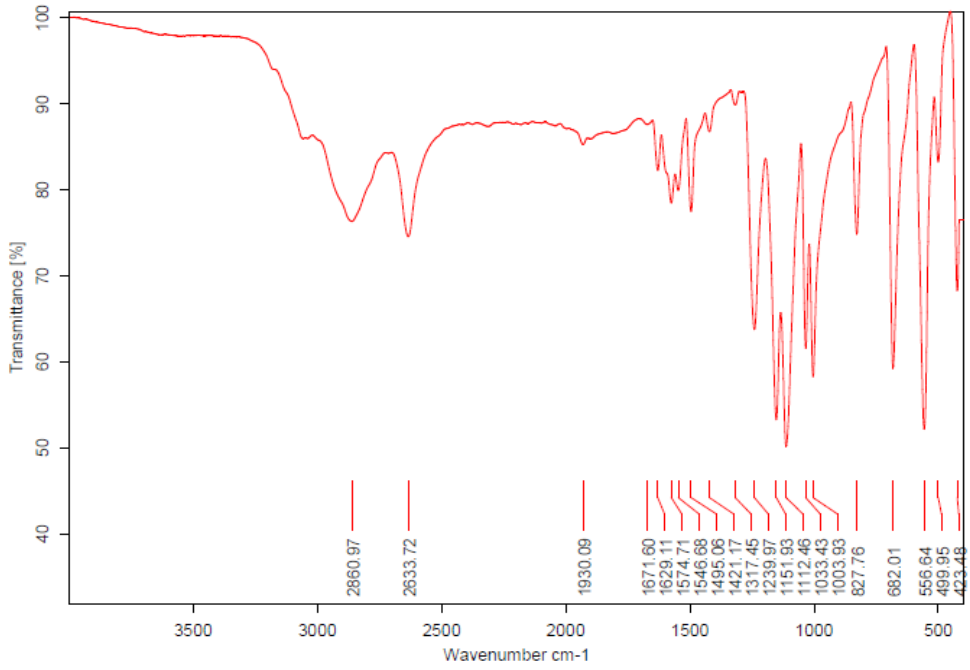

Figure 3. *Sulfanilic acid*

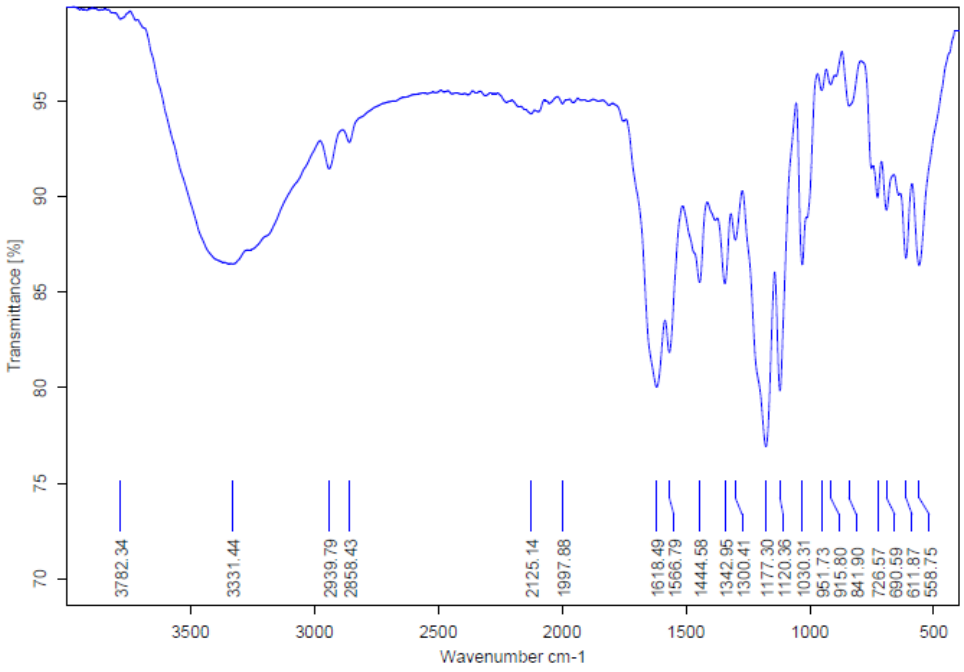

Figure 4. *Azo drug*

Impact of different acid types on azo dye absorption

Absorbance for various volumes (0.5 - 2.0 mL) in strong and weak acid solutions (1M) has been investigated. The findings are displayed in Table (1).

Table 1. Impact of different acid types on azo dye absorption by used

(1 mL) sulphanilic acid +(0.5 ml) minoxidil + (0.5 mL) $NaNO_2$

Type of acid	0.5	1	1.5	2
HCl	**0.268**	0.237	0.248	0.246
H_3PO_4	0.258	0.244	0.252	0.265
CH_3COOH	0.238	0.252	0.256	0.259

Effect of buffer solution

The effect of the buffer solution was studied at pH = 1.3, the volumes between (0.1-0.4) ml were taken and added to the reaction product, and the volume was completed by distilled water in a 10 ml standard flask, the absorbance was measured at 390 nm, as shown in Table (2). As a decreased in absorption values was observed, it was cancelled.

Table 2. Effect of buffer solution (pH = 1.3)

Buffer solution (KCl+HCl)	Absorbance
With out	**0.268**
0.1	0.134
0.2	0.121

0.3	0.118
0.4	0.112

Effect of nitrite size with time

The effect of the amount of sodium nitrite was studied, where take 1 ml of sulfanilic acid and add 0.5 ml of hydrochloric acid (1 M),then increasing volumes of sodium nitrite (0.2 M) were added between (3.5-0.25) ml, then 0.25 ml of sulphamic acid (3%) was added, then added 0.5 ml of Minoxidil solution, its concentration was 100 µg /ml, and the solution was transferred to a 10 ml volumetric flask and the volume was completed with distilled water. The absorption was measured at 390 nm wavelength and the absorption values were recorded at different times, as shown in Table (3), where a volume of 1 ml of sodium nitrite was used at Time: 7 minutes for absorption values to stabilize.

Table 3. Effect of nitrite size with time

$NaNO_2$(0.2)	0	3	5	7	10	12
0.25	0.059	0.066	0.065	0.066	0.078	0.074
0.5	0.268	0.224	0.209	0.204	0.199	0.193
1	**0.415**	**0.403**	**0.392**	**0.380**	**0.379**	**0.363**
1.5	0.618	0.603	0.588	0.581	0.557	0.543
2	0.879	0.861	0.857	0.815	0.805	0.787
2.5	1.045	1.020	0.988	0.960	0.939	0.933
3	1.197	1.170	1.127	1.120	1.085	1.043
3.5	1.406	1.370	1.337	1.287	1.245	1.211

Effect of Sulphamic acid with time

Sulfamic acid was used to remove excess from sodium nitrite [19], which affects absorption values. After withdrawing 1 ml of sulfanilic acid, 0.5 ml of 1 M hydrochloric acid was added. Then 1 ml of sodium nitrite (0.2 M) was added, after which different volumes between (0.25-2) ml of Sulphamic acid (3%) were added. then 0.5 ml of a 100 µg/ml minoxidil solution was added. The solution was transferred to a 10 ml volumetric flask, and the volume was completed with distilled water. The absorption was measured at a wavelength of 390 nm, and the absorption values were recorded at different times, as shown in Table (4).

Table 4. Effect of Sulphamic acid with time

H_2NSO_3H(3%)	0	3	5	7	10	12
0.25	0.415	0.403	0.392	0.380	0.379	0.363
0.5	0.093	0.078	0.071	0.055	0.041	0.034
1	0.023	0.020	0.014	0.010	0.008	0.006
1.5	0.025	0.018	0.016	0.012	0.007	0.007
2	0.025	0.016	0.014	0.012	0.008	0.007

It was noted that an increase The acid leads to the disintegration of the nitrogen product and thus a decrease in the absorption values, so the volume of acid used in subsequent experiments was kept.

Effect of sulfanilic size

The optimal size of sulfanilic acid was studied, where different volumes ranging from (0.5-2) ml of the reagent(10 ppm) were withdrawn, 0.5 ml of hydrochloric (1 M) was added to it, then 1 ml of sodium nitrite (0.2) M was added, then 0.25 ml of Sulphamic acid (3%) was added, then add 0.5 ml of 100 µg/ml minoxidil , the solution transfered to a 10 ml volumetric flask and compeleted the volume with distilled water, the absorbance was measured at 390 nm wavelength. Absorption values were recorded in Table (5). It is clear that the highest absorption value at 1 ml of the reagent, which It was adopted in subsequent experiments.

Table 5. Effect of Sulfanilic acid

Reagent (10 ppm)	0	3	5	7	10	12
0.5	0.222	0.211	0.205	0.200	0.197	0.192
1	0.415	0.403	0.392	0.380	0.379	0.363
1.5	0.385	0.354	0.320	0.312	0.306	0.299
2	0.252	0.239	0.236	0.223	0.218	0.214

The effect of time

The effect of time on the stability of the product was studied by measuring the absorption versus the blank solution for different time periods and under the same conditions as the previous experiments, as shown in Table (6), where it was found that the best reaction time is after 25 minutes and it remains stable for another 20 minutes.

Table 6. Effect of time

Time	5	10	15	20	25	30	35	40	45	50	55	60
Abs	0.392	0.379	0.359	0.340	0.329	0.329	0.328	0.324	0.320	0.315	0.311	0.307

Absorption spectrum

Measurements were made of the product's absorption spectrum when 1 ml of sulfanilic acid was reacted with 0.5 ml of 1 M hydrochloric acid, 1 ml of 0.2 M sodium nitrite, and 0.25 ml of 3%) sulphamic acid, then (0.5 ml) of 100 µg/ml minoxidil solution was added. ml and transfer the solution to a 10 ml volumetric vial and fill the volume with distilled water as shown in Figure (2).

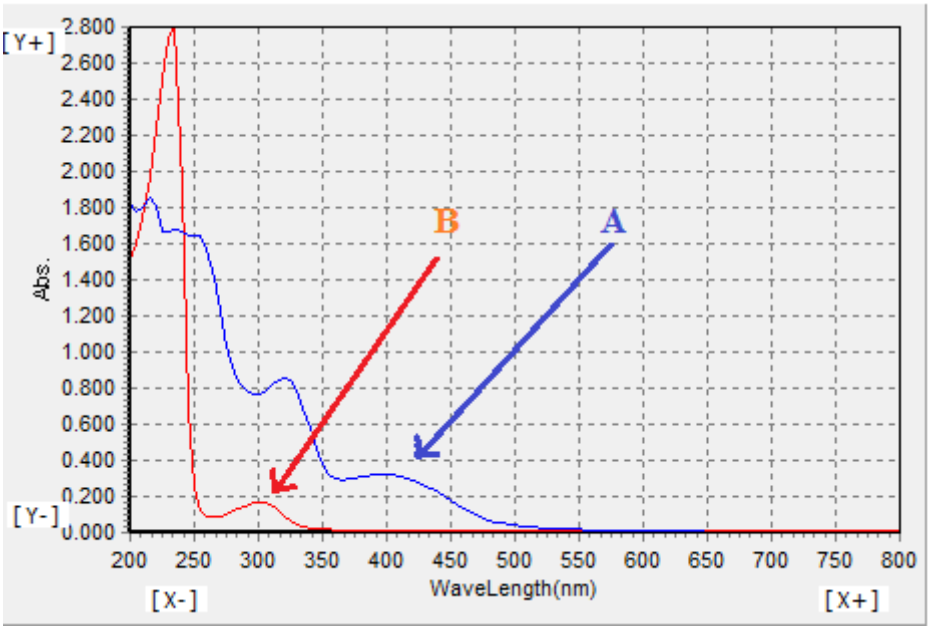

Fig 2. *(A) Absorption spectrum of the complex versus the blank solution (B) Absorption spectrum of blank solution versus distilled water*

Standard curve preparation

After established the best conditions for the determination of the Minoxidil drug, a calibration curve was prepared. 1 ml of sulfanilic acid was taken, to which 0.5 ml of hydrochloric acid (1 M) was added, then 1 ml of sodium nitrite (0.2 M) was added, then 0.25 ml of sulphamic acid (3%) was added, after which increasing volumes were added. (0.1-5.0 ml of minoxidil 100 µg/ml, the solution transferred to 10 ml volumetric flask, compeleted the volume with distilled water, then wait 25 minutes and measure the absorbance of each concentration against the blank solution at 390 nm wavelength, as Beer's law was followed within the range of concentrations (40-5) µg/ml of Minoxidil, as shown in Figure (3). It gave a coefficient of estimation of 0.9993, the slope value was 0.0221, the molar absorption value was $4.624*10^3$ L/mol.cm, and the Sandel sensitivity value was 0.0452 µg /cm^2.

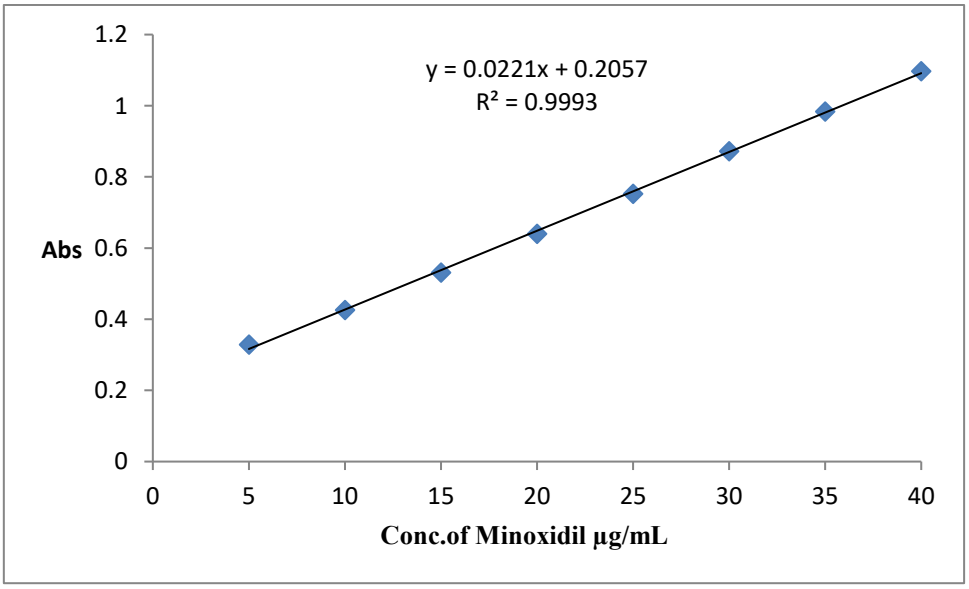

Fig 3. *Standard curve for the determination of Minoxidil by diazotization and coupling method*

Accuracy and precision

The method's accuracy and precision were tested by calculating Average of Recovery% and RSD%, and under optimal conditions by taken five readings for three different concentrations of the Minoxidil drug within the limits of Beer's Law in the calibration curve, which gave 100.1519% to Average of Recovery% and 0.0112 to RSD % no more than, as shown in Table (7). By taking five readings of the blank solution's absorbance to identify the standard deviation value, the detection limit and quantitative limit were calculated, Table (8) shows the calculated values [20].

Table 7. Accuracy and precision

Taken (ppm)	Abs	Found (ppm)	Rec%	Average of Recovery%	RSD%
10	0.429	10.10407	101.0407	100.1669	0.0112
20	0.642	19.74208	98.71040		0.0057
35	0.985	35.26244	100.7498		0.0032

Table 8. The detection limit and quantitative limit

Slope	SD	LOD(µg/mL)	LOQ(µg/mL)
0.0221	0.0011	0.1642	0.4977

Stoichiometry to Azo dye

To determine the rate and nature of the compound's bonding, the molar ratios and continuous changes (JOB) methods were performed as follows:

Molar ratio method [21]

It involved taken sulfanilic acid concentrations increasing in quantities (0.25-2.0) ml of 1x 10^{-4} M into a series of 10 ml volumetric flask, adding 0.5 ml of hydrochloric acid (1M), then adding 1 ml of sodium nitrite (0.2M), then adding 0.25 ml of sulphamic acid (3%) After that, a fixed volume of 1 ml of minoxidil solution was added at the same concentration, 1x 10^{-4} M, the volume was finished to the appropriate level. Then passed 25 minutes, the absorbance values were recorded against the blank solution at 390 nm wavelength, showed a ratio of 1:1, as shown in the figure (4).

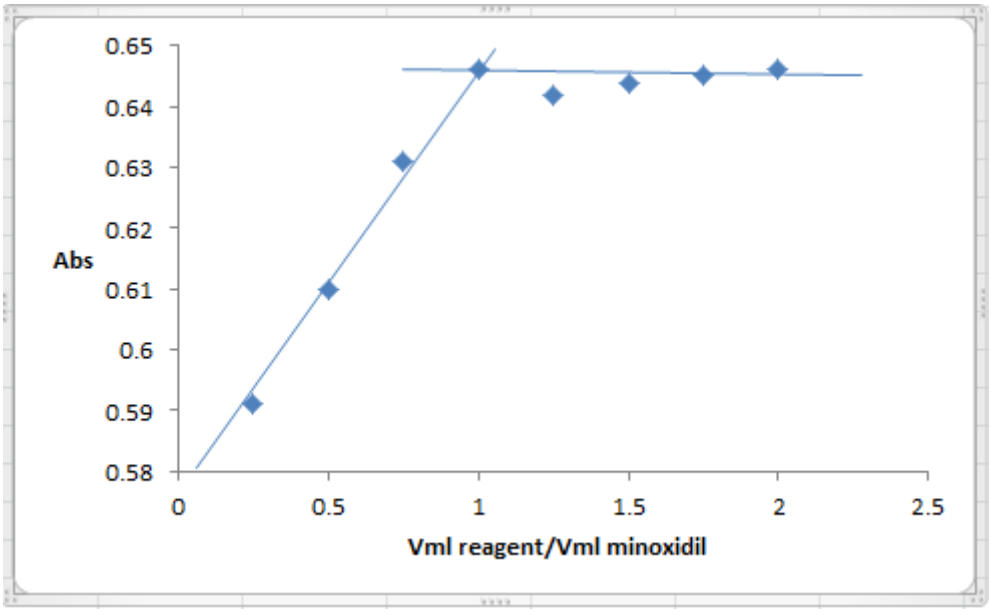

Fig 4. *Curve of the molar ratio method for the determination of Minoxidil*

Continuous changes method (Job method) [22]

It included taking different volumes of sulfanilic acid (0.1-0.9) ml (1x 10-4M) into a series of 10 ml volumetric flask, added 0.5 ml of hydrochloric acid (1M) , then added 1 ml of sodium nitrite (0.2) molarity, then added 0.25 ml of sulphamic acid (3%), Then increasing volumes (0.9-0.1) ml of Minoxidil solution (1x 10^{-4}M) were added so that the finaly volume of the reagent and the drug was 1 ml , the volume was completed to the mark. Then, after 25 minutes, the absorbance values were recorded against the blank solution at 390 nm of wavelength, where it showed The ratio was 1:1, as shown in Figure (5).

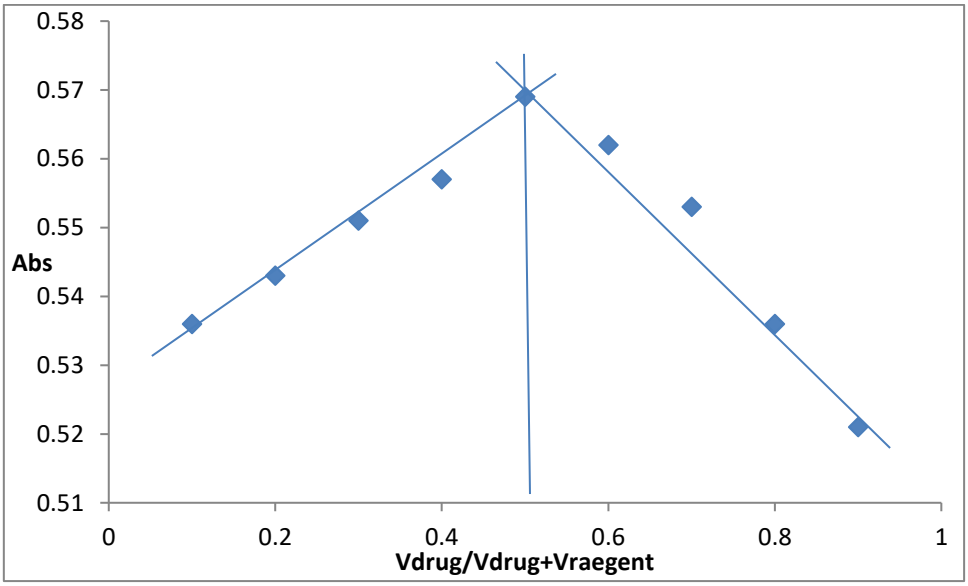

Fig 5. *Curve of the Job method for the determination of Minoxidil*

Application of the method

The proposed method was applied to the pharmaceutical preparation of Minoxidil, where three different concentrations (10, 25 and 35) µg/ml were taken from the preparation with a concentration of 100 µg/ml by withdrawing volumes of 1.0, 2.5, and 3.5 ml, respectively, into a 10-ml volumetric flask under the same conditions, then add distilled water to the volume to

the required level. The solutions' absorbance was measured at a wavelength of 390 nm, where the value of the average of recovery% reached 100.852% in the pharmaceutical preparation, and the value of the RSD% not exceed 0.0111%. as shown in Table (9).

Table 9. Application of the method for the determination of Minoxidil in a pharmaceutical preparation

Pharmaceutical preparation (ppm)	Abs	Found µg/ml	Rec%	RSD%
10	0.431	10.1945	101.9457	0.0111
25	0.756	24.9004	99.6018	0.0045
35	0.987	35.3529	101.0084	0.0032

Summary of optical characteristics for the method proposed

The analytical characteristics for the determination of Minoxidil by the diazotization and coupling method and at the optimum conditions are summarized in Table (10).

Table 10. Summary of optical characteristics for the method proposed

Parameter	Value
λmax nm	390
Beer's Law Limit (µg mL^{-1})	5-40
Molar Absorptivity ($Lmol^{-1}cm^{-1}$)	$4.624*10^3$
Limit of Detection (µg mL^{-1})	0.1642
Limit of Quantification (µg mL^{-1})	0.4977
Color	Yellow
Slope	0.0221
Intercept	0.2057
Correlation Coefficient (R^2)	0.9993
Sandell's Sensitivity (µg cm^{-2})	0.0452
RSD%	$\leq$0.0112
Recovery%	98.6651-101.0407

Conclusion

In this work, minoxidil and diazotization of sulfanilic acid in an acidic solution were combined to create a yellow azo dye that was stable, soluble in water, and showed a maximum absorption peak at 390 nm in comparison to the blank solution. Beer's law demonstrated linearity within the concentration range of 5-40 µg/ml, with a molar absorptivity of 4.624*103 l/mol.cm. , the RSD% was discovered to range from 0.0032% to 0.0111%. The recommended method for detecting minoxidil in pharmaceutical samples has the benefits of being inexpensive, straightforward, and sensitive. The method was effectively used to determination in pharmaceuticals..

References

1- LINAS, S. L., & NIES, A. S. (1981). Minoxidil. Annals of Internal Medicine, 94(1), 61-65. doi:10.7326/0003-4819-94-1-61

2- Shorter, K., Farjo, N. P., Picksley, S. M., & Randall1, V. A. (2008). Human hair follicles contain two forms of ATP-sensitive potassium channels, only one of which is sensitive to minoxidil. *The FASEB Journal*, *22*(6), 1725-1736. https://doi.org/10.1096/fj.07-099424

3- Zins, G. R. (1988). The history of the development of minoxidil. *Clinics in dermatology*, *6*(4), 132-147. https://doi.org/10.1016/0738-081X(88)90078-8

4- Gupta, A. K., Talukder, M., & Williams, G. (2022). Comparison of oral minoxidil, finasteride, and dutasteride for treating androgenetic alopecia. *Journal of Dermatological Treatment*, *33*(7), 2946-2962. https://doi.org/10.1080/09546634.2022.2109567

5- Ichida, M., Fujita, C., Sumie, R., Miyano, R., & Inoue, H. (2020). Simultaneous determination of minoxidil and minoxidil sulfate by high-performance liquid chromatography with UV-detection and its applications. *Medicine in Drug Discovery*, *7*, 100050. https://doi.org/10.1016/j.medidd.2020.100050

6- Shah, D. S., Jha, D. K., & Amin, P. D. (2021, March). Development, validation, and application of an RP-HPLC method for concurrent quantification of Minoxidil and Finasteride in a topical solution for hair regrowth. In Annales Pharmaceutiques Françaises (Vol. 79, No. 2, pp. 194-206). Elsevier Masson. https://doi.org/10.1016/j.pharma.2020.10.003

7- Basuri, P. P., Vijayakumar, R., & Nalini, C. N. (2022). Estimation of minoxidil in human plasma using UHPLC-MS/MS and its application in pharmacokinetic study. Journal of Chromatography B, 1192, 123104. https://doi.org/10.1016/j.jchromb.2022.123104

8- Dehdashtian, S., & Hashemi, B. (2020). Voltammetric sensing of minoxidil using a molecularly imprinted polymer (MIP)-modified carbon paste electrode. *Chemical Papers*, *74*, 157-165. https://doi.org/10.1007/s11696-019-00860-4

9- Teerasong, S., Praditweangkum, W., & Chompoosor, A. (2022). A new mechanism for resonance Rayleigh scattering detection of minoxidil based on catalytic oxidation of silver nanoparticles. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, *275*, 121147. https://doi.org/10.1016/j.saa.2022.121147

10- Caldas, N. M., do Nascimento, S. F., de Faria, L. V., de Oliveira Alves, A., Peixoto, D. A., Semaan, F. S., ... & Dornellas, R. M. (2024). Cost-effective lab-made graphite-and aluminum oxide-integrated polylactic acid composite electrodes for electrochemical sensing of minoxidil. *Microchemical Journal*, *199*, 109948. https://doi.org/10.1016/j.microc.2024.109948

11- Oliveira, P. M., Sampaio, T. R., França, L. C., Gratieri, T., Cunha-Filho, M., & Gelfuso, G. M. (2020). LC–MS bioanalytical method for simultaneous determination of latanoprost and minoxidil in the skin. Journal of pharmaceutical and biomedical analysis, 187, 113373. https://doi.org/10.1016/j.jpba.2020.113373

12- Annapurna, M. M., Polipalli, V. N., & Angirekula, N. (2024). New spectrophotometric methods for the determination of Minoxidil. Research Journal of Pharmacy and Technology, 17(5), 2372-2378. 10.52711/0974-360X.2024.00371

13- Afika, N., Saniy, A. F., Ko, C. K., Kamran, R., & Permana, A. D. (2024, May). Application of validated UV spectrophotometric and colorimetric method to quantify minoxidil in the development of trilayer dissolving microneedle: Proof of concept in ex vivo and in vivo studies in rats. In Annales Pharmaceutiques Françaises (Vol. 82, No. 3, pp. 531-544). Elsevier Masson. 10.1016/j.pharma.2023.12.006
14- Elmansi, H., Belal, F., M. Hosny, M., & M. EL-Abassy, O. (2021). Utility of NBD-Cl as an electrophilic reagent for the determination of the two antihypertensive drugs hydrochlorothiazide and minoxidil in dosage forms and human urine samples. *Chemical Papers*, *75*, 1925-1935.
15- Perrin, D. D. (2012). *Buffers for pH and metal ion control*. Springer Science & Business Media. http://doi 10.1007/978-94-009-5874-611
16- Ahmed, A. I., & Omar, K. M. (2024, May). Development of new spectrophotometric method for quantitative analysis of sulfasalazine in pure and tablet dosage forms based on diazo coupling reaction. In *AIP Conference Proceedings* (Vol. 3097, No. 1). AIP Publishing. https://doi.org/10.1063/5.0211441
17- Patel, A. R., Patel, G., Srivastava, A., & Banerjee, S. (2023). A Review on Traditional and Modern Methods for the Synthesis of Aromatic Azo Compounds. *Current Organic Chemistry*, *27*(18), 1611-1628. https://doi.org/10.2174/0113852728301737240307111549
18- Nejati, K., Rezvani, Z., & Seyedahmadian, M. (2009). The synthesis, characterization, thermal and optical properties of copper, nickel, and vanadyl complexes derived from azo dyes. *Dyes and Pigments*, *83*(3), 304-311 https://doi.org/10.1016/j.dyepig.2009.05.007.
19- Granger, J., & Sigman, D. M. (2009). Removal of nitrite with sulfamic acid for nitrate N and O isotope analysis with the denitrifier method. *Rapid Communications in Mass Spectrometry: An International Journal Devoted to the Rapid Dissemination of Up-to-the-Minute Research in Mass Spectrometry*, *23*(23), 3753-3762. https://doi.org/10.1002/rcm.4307
20- Dayyih, W. A., Al Mufty, M., Hailat, M., Awad, R., Awad, M. R., & Hamad, M. F. (2024). Development and Validation of a Method for Analyzing the Combination of Etoricoxib and Acetaminophen in Tablet Dosage Form. *Journal of Hunan University Natural Sciences*, *51*(5). https://doi.org/10.55463/issn.1674-2974.51.5.1
21- Momoki, K., Sekino, J., Sato, H., & Yamaguchi, N. (1969). Theory of curved molar ratio plots and a new linear plotting method. *Analytical Chemistry*, *41*(10), 1286-1299. https://doi.org/10.1021/ac60279a008
22- Huang, C. Y. (1982). [27] Determination of binding stoichiometry by the continuous variation method: the job plot. In *Methods in enzymology* (Vol. 87, pp. 509-525). Academic Press. https://doi.org/10.1016/S0076-6879(82)87029-8

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تطوير طريقة طيفية لتقدير المينوكسيديل عن طريق تفاعل الازوتة والاقتران مع حمض السلفانيليك

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الخلاصة:
طريقة طيفية واضحة وسريعة ودقيقة باستخدام حمض السلفانيليك المؤزوت ككاشف لتحديد كمية المينوكسيديل في كل من صورته الصيدلانية والنقية. في هذه الطريقة، تم ازوتة *4– أمينوبنزين حامض السلفونيك* (حمض السلفانيليك) إلى محلول نتريت الصوديوم ($NaNO_2$) تركيزه قليل. بجود حامض الهيدروكلوريك (HCl) لإنتاج ملح الديازونيوم المقابل بعد ذلك يقترن مع عقار المينوكسيديل، لينتج لون أصفر من صبغة الآزو التي كانت قابلة للذوبان في الماء ولها أقصى امتصاص (λmax) عند 390 نانومتر مقارنة بالمحلول الصوري للكاشف. وفي الظروف الفضلى تم تقدير الخطية لقانون بير في مدى التركيز (5-40) ميكروغرام/مل مع معامل تقدير (R^2=0.9993) وامتصاصية مولية قدرها $4.624 *10^3$ لتر/مول.سم. تم حساب حد الكشف (LOD) والحد الكمي (LOQ) ومدى الدقة (RSD٪) بما يساوي 0.012 و0.040 ميكروجرام/مل 0.16% إلى 1.59% على التوالي. وقد تبين أن قياس العناصر الكيميائية هو 1-1 (مينوكسيديل: حمض السلفانيليك المؤزوت). تم استخدام الإجراء بنجاح لتقدير المينوكسيديل في الأقراص.