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## Synthesis, spectroscopic studies and electrochemical properties of Schiff bases derived from 2-hydroxy aromatic aldehydes and phenazopyridine hydrochloride

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**Abstract:** Novel Schiff bases **1–4** were synthesized by the reaction of 2-hydroxybenzaldehyde, 2-hydroxy-5-methoxybenzaldehyde, 2-hydroxy-5-nitrobenzaldehyde and 2-hydroxy-1-naphthaldehyde with phenazopyridine hydrochloride (PAP), respectively, and their structures were elucidated by means of spectroscopic techniques. The electrochemical reduction of PAP and its Schiff bases (**1–4**) were realized on a glassy carbon electrode (GCE) in dimethyl sulfoxide (DMSO) using the cyclic voltammetric (CV) technique. The effect of functional groups on reduction potential of the Schiff bases was investigated. A general electrochemical reduction mechanism of the compounds is suggested.

**Keywords:** phenazopyridine hydrochloride; Schiff base; spectroscopy; voltammetry.

### INTRODUCTION

Phenazopyridine hydrochloride (PAP) is an analgesic drug used to provide symptomatic pain relief in conditions such as cystitis and urethritis.<sup>1–5</sup> The electrochemical reduction and determination of phenazopyridine hydrochloride were realized on a carbon paste electrode by Osteryoung square wave voltammetry.<sup>5</sup> The electrochemical properties of PAP were also investigated by the adsorptive stripping voltammetric technique.<sup>6</sup> In addition, differential pulse polarography is a convenient method for the analysis of nitrofurantoin and phenazopyridine in tablet form.<sup>7</sup>

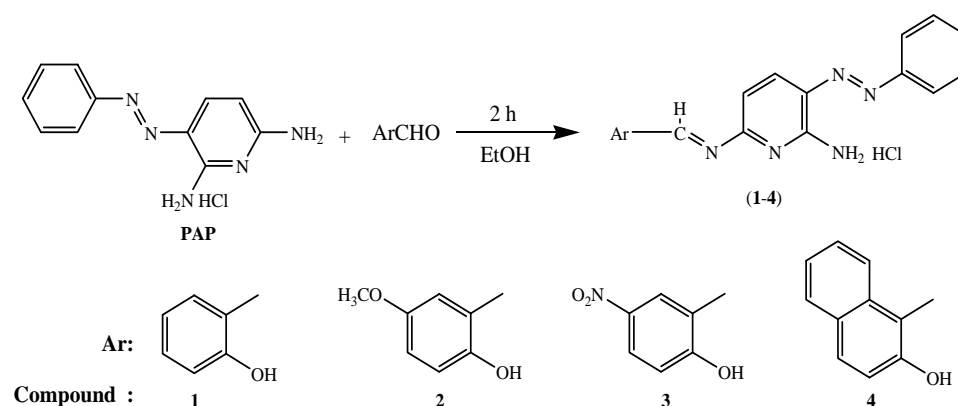
Schiff bases are used extensively as ligands in coordination chemistry.<sup>8,9</sup> They have thermochromic and photochromic properties in their solid state.<sup>10</sup>

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They are also used as model systems for biological macromolecules and catalytic reactions.<sup>11,12</sup> There are relatively few studies on the electrochemical behavior of imines or Schiff bases in aprotic media.<sup>13</sup> A different mechanism was proposed in non-aqueous media where hydrolysis does not pose problems.

In Schiff bases, the reduction potential is dependent on the size of the aromatic groups on either side of the  $-C=N-$  group,<sup>14-17</sup> the types of substituent attached to the aromatic ring<sup>15-19</sup> and intra-molecular hydrogen bonds.<sup>20,21</sup> It has also been claimed that the presence of electron withdrawing groups and hydrogen bonding facilitate the reduction. Schiff base ligands consist of a variety of substituents with different electron-donating and electron-withdrawing groups, and therefore may have interesting electrochemical properties.

Although the electrochemical reduction reactions of Schiff bases containing  $-N=N-$  and  $-C=N-$  groups in aqueous medium<sup>22-25</sup> have been studied, insufficient studies have been directed to the electrochemical reduction of PAP and its Schiff bases in aqueous and non-aqueous media. In the present study, the Schiff bases **1-4** were synthesized and investigated by elemental analysis, FT-IR, <sup>1</sup>H-NMR, <sup>13</sup>C-NMR, UV-Vis and MS spectroscopic techniques in order to study their electrochemical properties in dimethyl sulfoxide (Scheme 1).



Scheme 1. The synthesis of Schiff bases **1-4**.

## EXPERIMENTAL

### Instrumentation

The <sup>1</sup>H- and <sup>13</sup>C-NMR spectra were recorded on a Bruker Avance DPX NMR spectrometer operating at 400 and 101.6 MHz, respectively. The infrared absorption spectra were recorded on a Perkin Elmer BX II spectrometer in KBr discs and are reported in  $\text{cm}^{-1}$ . The UV-Vis spectra were measured using a Shimadzu 1208 series spectrometer. Carbon, nitrogen and hydrogen analyses were performed with a Leco CHNS-932 analyzer. Melting points were measured on an Electro Thermal IA 9100 apparatus using a capillary tube. LC mass spectra were recorded on an Agilent 1100 MSD spectrometer with an ion source temperature of 240 °C. A Model Metrohm 757 VA trace analyzer (Herisau, Switzerland) was employed for the

voltammetric measurements, with a three-electrode system consisting of a GCE (surface size,  $\varphi = 7$  mm, disc diameter,  $R = 2$  mm Metrohm) working electrode, a platinum wire auxiliary electrode and a Ag/Ag<sup>+</sup> (0.01 M AgNO<sub>3</sub>/DMSO) reference electrode. Before each measurement the working electrode was polished manually with polishing alumina (prepared from 0.01  $\mu$ m aluminum oxide) on an alumina polish pad, then rinsed with ultra pure deionized water and ethanol and DMSO. All measurements were performed after deoxygenating the supporting electrolyte with argon gas for 5 min and 60 s for the sample prior to each measurement.

#### Reagents

PAP was kindly supplied by Faco Inc. (Istanbul, Turkey). 2-Hydroxybenzaldehyde, 2-hydroxy-5-methoxybenzaldehyde, 2-hydroxy-5-nitrobenzaldehyde, 2-hydroxy-1-naphthaldehyde, EtOH, DMSO and tetrabutylammonium iodide (TBAI, 98 %) were obtained from BDH Chemicals and alumina and silver nitrate were purchased from Merck (Germany).

#### Synthesis of the Schiff bases

2-([6-Amino-5-(2-phenyldiazenyl)pyridin-2-yl]imino)methylphenol hydrochloride (**1**). PAP (0.1 g;  $4.0 \times 10^{-4}$  mol) was added to a dry EtOH (100 mL) solution of 2-hydroxybenzaldehyde (0.049 g;  $4.0 \times 10^{-4}$  mol). The mixture was stirred and heated for 2 h. Compound **1** was obtained after EtOH evaporation and crystallization from chloroform/*n*-heptane.

2-([6-Amino-5-(2-phenyldiazenyl)pyridin-2-yl]imino)methyl-4-methoxyphenol hydrochloride (**2**). Compound **2** was obtained in a similar manner to **1** but using a dry EtOH (100 mL) solution of 2-hydroxy-5-methoxybenzaldehyde (0.0608 g;  $4.0 \times 10^{-4}$  mol).

2-([6-Amino-5-(2-phenyldiazenyl)pyridin-2-yl]imino)methyl-4-nitrophenol hydrochloride (**3**). Compound **3** was obtained in a similar manner to **1** but using a dry EtOH (100 mL) solution of 2-hydroxy-5-nitrobenzaldehyde (0.0668 g;  $4.0 \times 10^{-4}$  mol).

1-([6-Amino-5-(2-phenyldiazenyl)pyridin-2-yl]imino)methyl-2-naphthol hydrochloride (**4**). Compound **4** was obtained in a similar manner to **1** but using a dry EtOH (100 mL) solution of 2-hydroxy-1-naphthaldehyde (0.0688 g;  $4.0 \times 10^{-4}$  mol).

## RESULTS AND DISCUSSION

Schiff bases are important in diverse fields of chemistry due to their biological activities. In addition to their biological activities, their photochromic characteristics have led to their usage in various areas, such as the control and measurement of radiation intensity, display systems and optical computers. In the field of coordination chemistry, the *ortho* hydroxylated type of Schiff bases has received overwhelming attention, particularly in the study of complex formation. Recently, it was found that the introduction of lateral polar hydroxyl groups enhanced the molecular polarizability and stabilized the liquid crystalline compounds. These compounds also contain interesting groups, such as (–N=N– and –C=N–), for electrochemical studies.

#### Analytic and spectral data of the synthesized Schiff bases

2-([6-Amino-5-(2-phenyldiazenyl)pyridin-2-yl]imino)methylphenol hydrochloride (**1**). Brown crystals; yield: 0.12 g, 86 %; m.p.: 227 °C; Anal. Calcd. for C<sub>18</sub>H<sub>16</sub>ClN<sub>5</sub>O: C, 61.10; H, 4.53; N, 19.80 %. Found: C, 61.10; H, 4.56; N,

19.79 %; IR (KBr,  $\text{cm}^{-1}$ ): 3306 (*m*, O–H); 3071 (*m*, Ar–H); 1631 (*m*, C=N); 1450 (*s*, C=C); 1264 (*m*, C–O);  $^1\text{H-NMR}$  (400 MHz, DMSO,  $\delta$  / ppm): 14.86 (1H, *s*, Ar–OH); 8.59 (1H, *s*, Ar–CH=N–); 6.29 (2H, *s*, Ar–NH<sub>2</sub>–); 6.61–7.90 (11H, *m*, Ar–H);  $^{13}\text{C-NMR}$  (100.6 MHz, DMSO,  $\delta$  / ppm): 192.0, 162.3, 155.2, 146.8, 139.1, 137.5, 136.8, 129.9, 129.6, 128.3, 119.9, 118.9, 117.7, 117.1, 117.1, 116.2.

2-([6-Amino-5-(2-phenyldiazenyl)pyridin-2-yl]imino)methyl)-4-methoxyphenol hydrochloride (**2**). Brown crystals; yield: 0.130 g, 85 %; m.p.: 195 °C; Anal. Calcd. for C<sub>19</sub>H<sub>18</sub>ClN<sub>5</sub>O<sub>2</sub>: C, 59.45; H, 4.69; N, 18.25 %. Found: C, 59.45; H, 4.73; N, 18.25 %; IR (KBr,  $\text{cm}^{-1}$ ): 3298 (*m*, O–H); 3079 (*m*, Ar–H); 1623 (*m*, C=N); 1451 (*s*, C=C); 1266 (*m*, C–O);  $^1\text{H-NMR}$  (400 Hz, DMSO,  $\delta$  / ppm): 15.85 (1H, *s*, Ar–OH); 8.63 (1H, *s*, Ar–CH=N–); 6.32 (2H, *s*, Ar–NH<sub>2</sub>–); 6.64–7.92 (10H, *m*, Ar–H); 3.79 (3H, *s*, Ar–OCH<sub>3</sub>);  $^{13}\text{C-NMR}$  (101.6 MHz, DMSO,  $\delta$  / ppm): 191.2, 155.9, 152.6, 150.1, 142.3, 130.1, 129.7, 126.6, 126.5, 122.5, 119.2, 118.8, 117.8, 116.8, 114.5, 110.3, 55.92.

2-([6-Amino-5-(2-phenyldiazenyl)pyridin-2-yl]imino)methyl)-4-nitrophenol hydrochloride (**3**). Brown crystals; yield: 0.136 g, 86 %; m.p.: 167 °C; Anal. Calcd. for C<sub>18</sub>H<sub>15</sub>ClN<sub>6</sub>O<sub>3</sub>: C, 54.20; H, 3.79; N, 21.08 %. Found: C, 54.21; H, 3.79; N, 21.07 %; IR (KBr,  $\text{cm}^{-1}$ ): 3306 (*m*, O–H); 3063 (*m*, Ar–H); 1619 (*m*, C=N); 1451 (*s*, C=C); 1336 (*m*, C–O);  $^1\text{H-NMR}$  (400 MHz, DMSO,  $\delta$  / ppm): 12.21 (1H, *s*, Ar–OH); 8.90 (1H, *s*, Ar–CH=N–); 8.44 (2H, *s*, Ar–NH<sub>2</sub>–); 6.21–8.51 (10H, *m*, Ar–H);  $^{13}\text{C-NMR}$  (101.6 MHz, DMSO,  $\delta$  / ppm): 192.2, 166.1, 142.3, 139.3, 130.1, 129.2, 128.8, 127.3, 125.2, 124.3, 123.1, 121.1, 119.3, 118.2, 116.4, 115.2.

1-([6-Amino-5-(2-phenyldiazenyl)pyridin-2-yl]imino)methyl)-2-naphthol hydrochloride (**4**). Brown crystals; Yield: 0.133 g, 83 %; m.p.: 237 °C; Anal. Calcd. for C<sub>22</sub>H<sub>18</sub>ClN<sub>5</sub>O: C, 65.43; H, 4.46; N, 17.35 %. Found: C, 65.43; H, 4.49; N, 17.34 %; IR (KBr,  $\text{cm}^{-1}$ ): 3282 (*m*, O–H); 3062 (*m*, Ar–H); 1633 (*m*, C=N); 1455 (*s*, C=C); 1365 (*m*, C–O);  $^1\text{H-NMR}$  (400 MHz, DMSO,  $\delta$  / ppm): 15.34 (1H, *s*, Ar–OH); 8.61 (1H, *s*, Ar–CH=N–); 6.60 (2H, *s*, Ar–NH<sub>2</sub>–); 7.58–8.96 (13H, *m*, Ar–H);  $^{13}\text{C-NMR}$  (101.6 MHz, DMSO,  $\delta$  / ppm): 193.2, 164.5, 155.1, 138.8, 130.5, 130.1, 129.8, 129.7, 129.6, 128.2, 128.0, 124.6, 122.8, 121.7, 119.7, 112.9, 109.9, 108.9, 108.3, 106.4.

#### FT-IR, $^1\text{H-NMR}$ , $^{13}\text{C-NMR}$ and UV spectroscopy

The vibration bands with the wave numbers 3306, 3298, 3306 and 3282  $\text{cm}^{-1}$  (O–H); 3071, 3079, 3063 and 3062  $\text{cm}^{-1}$  (C–H, Ar–H); 1450, 1451, 1451 and 1455  $\text{cm}^{-1}$  (C=C) and 1264, 1266, 1336 and 1365  $\text{cm}^{-1}$  (C–O, Ar–O) were observed for compounds **1–4**, respectively. The C=N bond was observed at 1631, 1623, 1619 and 1633  $\text{cm}^{-1}$  for **1–4**, respectively. The stretching frequency observed at 2875–2745  $\text{cm}^{-1}$  in compounds **1–4** showed the presence of O–H...N

intramolecular hydrogen bonds.<sup>26,27</sup> The C=N bond, which is partially accountable for the existence of enol–imine form, can also be inferred from the IR spectra of compounds **1–4**. Compounds **1–4** with strong bands at 1278, 1280, 1282 and 1284 cm<sup>-1</sup>, respectively, possessed high percentages of the enol–imine tautomer due to stabilization of the phenolic C–O bond.<sup>28</sup>

The <sup>1</sup>H-NMR data for compounds **1–4** show that the tautomeric equilibrium favors the enol–imine in DMSO. The OH protons were observed as singlets at 14.86, 15.85, 12.21 and 15.34 ppm for compounds **1–4**, respectively. The azomethine protons were observed as singlets at 8.59, 8.63, 8.90 and 8.61 ppm for compounds **1–4**, respectively. The amine protons were found as singlets at 6.29, 6.32, 8.44 and 6.60 ppm for compounds **1–4**. The phenyl protons resonated as multiplets at 6.61–7.90, 6.64–7.92, 6.21–8.51 and 7.58–8.96 ppm for compounds **1–4**, respectively. The singlet of the Ar–OCH<sub>3</sub> protons was observed at 3.79 ppm for compound **2**. The <sup>13</sup>C-NMR spectra of compounds **1–4** have 16, 17, 16 and 20 signals, respectively.

The UV–Vis spectra of the compounds were studied in DMSO. The Schiff bases exhibited absorptions in the range greater than 400 nm in polar and non-polar solvents.<sup>26,27</sup> The UV–Vis spectrum of *ortho* hydroxylated Schiff-bases that exist mainly as the enol–imine structure is indicated by the presence of a band at <400 nm, while compounds existing either as keto–amine or as mixture of enol–imine/keto–amine forms show a new band, especially in polar and non-polar solvents in both acidic and basic media, at >400 nm.<sup>26–29</sup> Compounds **1–4** showed absorption above 400 nm in DMSO. The enol–imine ⇌ keto–amine tautomerisms of the compounds **1–4** are found to be 40, 38, 48 and 49 % in DMSO, respectively.

In conclusion, UV–Vis, <sup>1</sup>H-NMR and <sup>13</sup>C-NMR results showed that the compounds existed in the enol–imine form in DMSO.

#### *Electrochemical studies*

The reduction properties of PAP and its Schiff bases were investigated using a GCE by cyclic voltammetry in the potential range 0 to –3 V. The cyclic voltammetry (CV) curves of these compounds are given in Fig. 1. The curves were recorded in DMSO solution containing 0.1 M tetrabutylammonium iodide (TBAI) as a supporting electrolyte at a scan rate of 3 V s<sup>-1</sup>. Three cathodic peaks were observed for PAP and compounds **1**, **2** and **4** and four peaks for compound **3**. A new reduction peak, which was thought to belong to the reduction of the NO<sub>2</sub> group, appeared at –2.7 V on the CV curve for compound **3**.

Since the –N=N– group (all compounds) is more susceptible to reduction than the –C=N– (compound **1–4**) and NO<sub>2</sub> (compound **3**), the –N=N– group is reduced at less negative potential than the other groups.<sup>22–25</sup> As could be seen from Fig. 1, the first peak (about –1.0 V) can, therefore, be attributed to the

reduction of the  $-\text{N}=\text{N}-$  (azo) group. The second peak (about  $-1.5$  V) may most probably be due to the reduction of  $-\text{C}=\text{N}-$  (azomethine) group of the adsorbed molecule and the third peak (about  $-2.0$  V) was thought to be due to the reduction of  $-\text{C}=\text{N}-$  group of the molecule in solution.<sup>30</sup> The  $\text{NO}_2$  group is reduced at a more negative potential than the others. The fourth peak (about  $-2.7$  V) can conveniently be attributed to reduction of the  $\text{NO}_2$  group. Electrochemical data of these compounds are given in Table I.

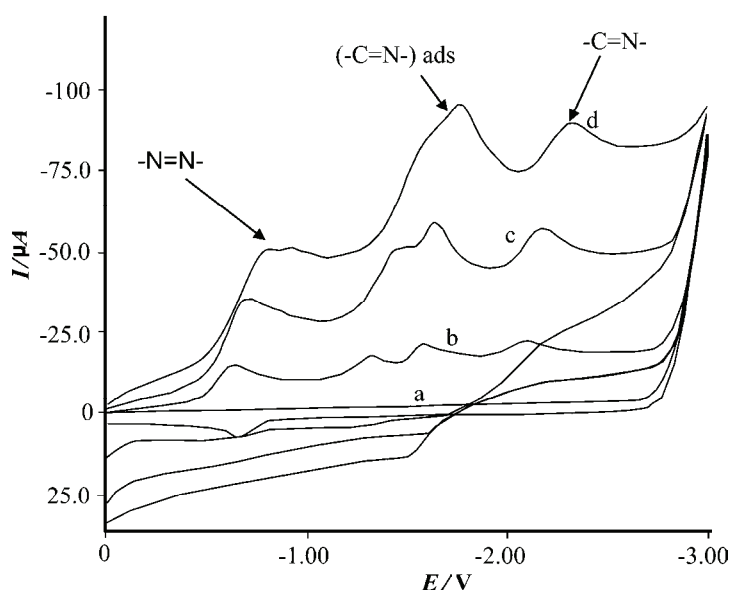


Fig. 1. The cyclic voltammograms of  $2 \times 10^{-3}$  M PAP and its Schiff bases in 0.1 M TBAI/DMSO at a GCE. Scan rate =  $3.0 \text{ V s}^{-1}$ .

TABLE I. Peak potential and peak current values of  $2 \times 10^{-3}$  M PAP and its novel Schiff bases in 0.1 M TBAI/DMSO at a GCE from CV. Scan rate =  $3 \text{ V s}^{-1}$

Compound	Peak 1		Peak 2		Peak 3		Peak 4	
	$E_{p1} / \text{V}$	$I_{p1} / \mu\text{A}$	$E_{p2} / \text{V}$	$I_{p2} / \mu\text{A}$	$E_{p3} / \text{V}$	$I_{p3} / \mu\text{A}$	$E_{p4} / \text{V}$	$I_{p4} / \mu\text{A}$
PAP	-0.73	20.70	-1.68	31.00	-2.22	12.80	-	-
<b>1</b>	-0.78	22.50	-1.76	33.50	-2.30	20.30	-	-
<b>2</b>	-0.79	23.70	-1.77	34.90	-2.33	20.60	-	-
<b>3</b>	-0.61	13.60	-1.26	14.30	-1.69	26.60	-2.77	14.50
<b>4</b>	-0.76	23.90	-1.71	35.30	-2.26	24.50	-	-

As can be seen from Table I, the current values of the two  $-\text{C}=\text{N}-$  groups of the Schiff bases were found to be nearly twice that of the single  $-\text{C}=\text{N}-$  group of PAP. The reduction potentials depend on the electronegativity of the groups in Schiff bases.<sup>14-21</sup> While the reduction potential had a negative value for compound **3**, it was observed that compound **2** had a more negative potential value.

The observed reduction potentials of the Schiff bases were  $-0.78$ ,  $-0.79$ ,  $-0.61$  and  $-0.76$  V ( $-\text{N}=\text{N}-$ ),  $-1.76$ ,  $-1.77$ ,  $-1.26$  and  $-1.71$  V ( $-\text{C}=\text{N}-$ )<sub>ads</sub> and  $-2.30$ ,  $-2.33$ ,  $-1.69$ ,  $-2.26$  V ( $-\text{C}=\text{N}-$ ) for compounds **1–4**, respectively. The effects of various scan rates between  $0.1$ – $10$  V s<sup>-1</sup> on the peak potential and the peak current of  $2 \times 10^{-3}$  M PAP and its Schiff bases were evaluated. Scan rate studies were performed to assess whether the processes on the GCE were under diffusion- or adsorption control.<sup>31–40</sup>

The cyclic voltammograms of  $2 \times 10^{-3}$  M PAP and its Schiff bases were obtained in  $0.1$  M TBAI/DMSO on a GCE. A linear relationship existed between the peak current and square root of the scan rate between  $0.1$ – $10$  V s<sup>-1</sup> (correlation coefficient about  $0.99$ ), which showed that the reduction process was predominantly under diffusion-control over the complete scan rate range studied. In addition, a plot of the logarithm of the peak current vs. the logarithm of the scan rate gave a straight line (correlation coefficient about  $0.99$ ) with a slope of about  $0.5$ , which is the expected value for an ideal reaction of solution species.<sup>33–43</sup> Therefore, a diffusion component must be taken into account. Other studies were conducted in line with this phenomenon.

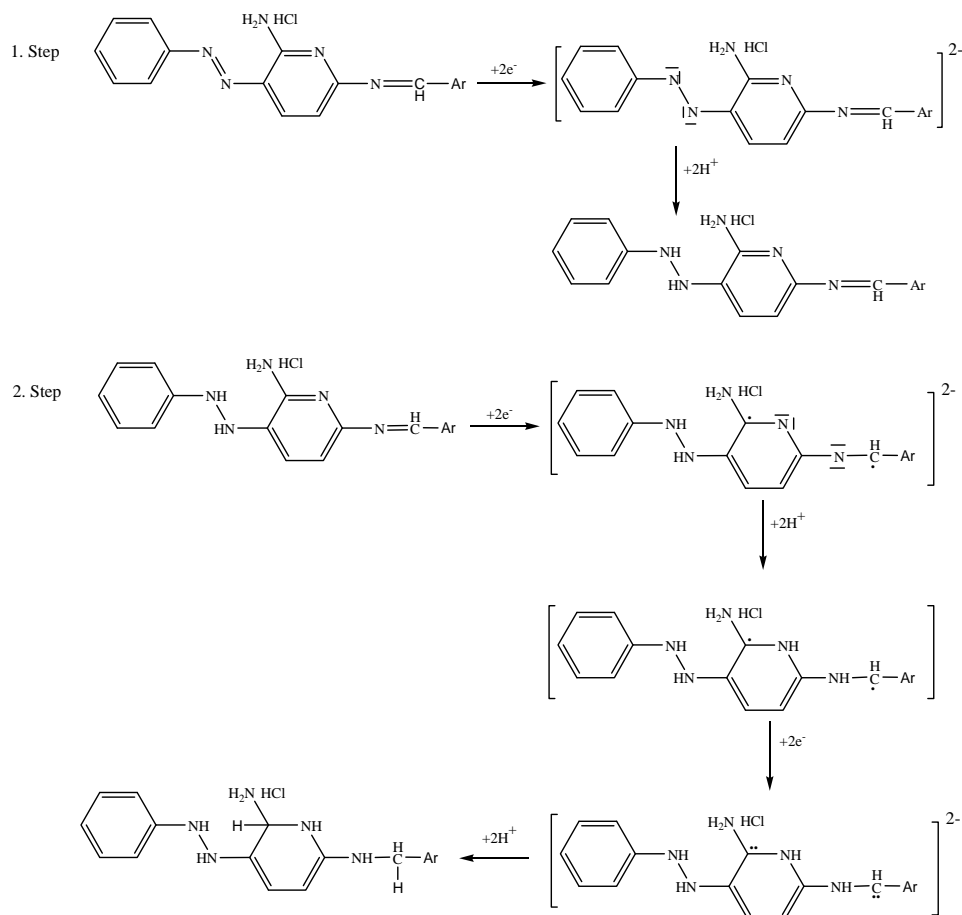
#### *Mechanism of the electrochemical reduction of the compounds*

When describing electrochemical reactions, an “*E*” and “*C*” formalism is often employed.<sup>32,33</sup> *E* represents an electron transfer; sometimes  $E_{\text{O}}$  and  $E_{\text{R}}$  are used to represent oxidations and reductions, respectively. *C* represents a chemical reaction, which can be any elementary reaction step, and is often called a “following” reaction.

The electrochemical reduction mechanisms are given in Scheme 2 for PAP **1–4**. The electrochemical reduction occurred in three steps. The  $-\text{N}=\text{N}-$  and the  $-\text{C}=\text{N}-$  groups were reduced in the first and the second step in compounds PAP and compounds **1–4** and in the last step, the  $-\text{NO}_2$  group was reduced in compound **3**. Both the  $-\text{C}=\text{N}-$  groups in PAP and the Schiff bases were reduced at the same potential.

As can be seen from the mechanism given in Scheme 2, the first step is the  $2e$  reduction of the diazenyl group of PAP, in which the  $-\text{N}=\text{N}-$  bond opens resulting  $-\text{N}-\text{N}^{2-}$  dianion. Then, the hydrazine anion binds  $2\text{H}^+$ , resulting in the formation of a hydrazine compound ( $-\text{NH}-\text{NH}-$ ). In the second step, the imine groups of PAP and its Schiff bases form a radical anion ( $\text{C}-\text{N}^{\cdot-}$ ) with a  $1e$  electron transfer. Then, the negatively charged nitrogen atom binds to a proton resulting in  $\text{C}-\text{NH}$ . This radical carbon atom takes one more electron giving a carbanion  $\text{C}^--\text{NH}$ . Finally, the carbanion reacts with a proton from the solution, resulting in an amine ( $\text{CHNH}$ ). In the third stage, the  $\text{NO}_2$  group in compound **3** is reduced. According to these data, it can conveniently be claimed that the reactions occur according to an EC mechanism.<sup>32</sup>



Scheme 2. Electrochemical reduction mechanism of PAP and the Schiff bases **1–4**.

### CONCLUSIONS

In the present work, the Schiff bases **1–4** were synthesized and characterized, and the electrochemical properties of PAP and its Schiff bases were investigated by cyclic voltammetry. A simple, sensitive and selective CV technique was developed for the examination of the electrochemical reduction behavior of PAP and its Schiff bases **1–4** on a GCE. While the electrochemical reduction occurred over three steps for PAP and compounds **1**, **2** and **4**, compound **3** had a fourth step. As could be seen from the cyclic voltammograms, the first, second and third peaks belong to the  $-\text{N}=\text{N}-$ ,  $-\text{C}=\text{N}-$  and  $-\text{C}=\text{N}-(\text{ads})$  groups, respectively, and the fourth peak belongs to the  $\text{NO}_2$  group.

It could also be concluded that the reduction potentials and currents depended on the electron withdrawing groups and hydrogen bonding on either side of the  $-\text{N}=\text{N}-$  and  $-\text{C}=\text{N}-$  groups.<sup>14–17</sup>



## ИЗВОД

## СИНТЕЗА, СПЕКТРОСКОПСКО ИСПИТИВАЊЕ И ЕЛЕКТРОХЕМИЈСКЕ ОСОБИНЕ ШИФОВИХ БАЗА ДОБИЈЕНИХ ОД 2-ХИДРОКСИ АРОМАТИЧНИХ АЛДЕХИДА И ФЕНАЗОПИРИДИН-ХИДРОХЛОРИДА

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У реакцијама између 2-хидроксибензалдехида, 2-хидрокси-5-метоксибензалдехида, 2-хидрокси-5-нитробензалдехида, 2-хидрокси-1-нафталдехида и феназопиридин-хидрохлорида (РАР) синтетизоване су нове Шифове базе (1–4), које су окарактерисане при меном спектроскопских метода. Електрохемијска редуција феназопиридин-хидрохлорида (РАР) и одговарајућих Шифових база (1–4) је извођена на електроди од стакластог угљеника (GCE) у диметил-сулфоксиду (DMSO) применом цикличне волтаметрије (CV). Испитиван је утицај функционалних група на потенцијал редуције Шифових база. Поред тога, предложен је механизам електрохемијске редуције испитиваних једињења.

(Примљено 24. маја, ревидирано 13. децембра 2012)

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