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RESEARCH ARTICLE

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Additional effect of para-hydroxyl on the reduction potentials of the *N*-benzylidenebenzenamines

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Abstract

The reduction potential E_{Red} of disubstituted *N*-benzylidenebenzenamines XArCH═NArY (abbreviated as XBAY) is a measure of the electron acceptance ability of the Lowest-energy Unoccupied Molecular Orbital (LUMO) in molecule, which is closely related to the electronic effects of substituents X and Y in molecule. Previous studies on the E_{Red} of XBAY reported by Luo, Wang, and Yuan all did not involve these molecules containing hydroxyl. In this work, 28 samples of disubstituted *N*-benzylidenebenzenamines containing para-hydroxyl, (abbreviated as 4-OHBAY or XBAOH-4′) were synthesized, and their E_{Red} values were measured. Then, 127 compounds were taken as a complicated E_{Red} data set, which involved 28 compounds (4-OHBAY and XBAOH-4′) of this work and 99 compounds XBAY reported by Luo. Based on these data E_{Red} set, through a quantitative regression analysis method and comparison of the factors affecting the E_{Red}, the following results are obtained: (a) Because the phenolic hydroxyl OH can dissociate H^+ to form phenolic oxygen anion, the change regularity of E_{Red} of compounds 4-OHBAY and XBAOH-4′ is somewhat different from that of E_{Red} of XBAY compounds without hydroxyl OH. That is, hydroxyl OH has an additional effect on the E_{Red}, which decreases the E_{Red} value. (b) The additional effects of 4-OH on the E_{Red} is different from that of 4′-OH. The effect of hydroxyl OH attached to the aldehyde aromatic ring on the E_{Red} is more than that of OH attached to the amine aromatic ring.

KEYWORDS

additional effect, para-hydroxyl *N*-benzylidenebenzenamine, reduction potential, substituent electronic effect

1 | INTRODUCTION

Schiff bases are a kind of important organic compounds containing C═N double bond. They are not only widely used as ligand in coordination chemistry[1–3] but also are able to be converted into various amines by reducing their molecular C═N double bond to CH–NH single bond.[4–8] For examples, Jia et al[4] employed sodium tetrahydroborate ($NaBH_4$) to reduce the imine C═N double bond of ruthenium (II) *p*-cymene complexes containing Schiff-base ligands [Ru(p-cymene)LCl] and prepared half-sandwich ruthenium complexes, which have active catalysts. Tan et al[5–10] used $NaBH_4$ to reduce the imine C═N double bond in the synthesis of novel 3-(1,3,4-thiadiazolyl)-1,3-bezoxazines, which have fungicidal activity. Liu et al[11] synthesized 4-[(5-aryl-1,3,4-thiadiazol-2-ylamino)methyl]phenol compounds via the nucleophilic addition reaction of 5-aryl-1,3,4-thiadiazol-2-amine with 4-hydroxybenzaldehyde and the reduction of C═N double bond in the presence of $NaBH_4$ reducing agent.

Previous studies have shown that the reducibility of C═N bond of Schiff bases is closely related to the substituents, which attach to the ring of aromatic aldehyde or aromatic amine. Not only the reducibility of Schiff bases but also the reducibility of other aromatic compounds are greatly affected by the substituents in their molecules. As regards the effect of substituents on the reducibility of organic compounds, it has been studied in-depth by many chemical researchers. Celik[12] and Zhu[13] have studied the reduction of these compounds containing XPhCH═NG or GCH═NPhY structure, and Sauro[14] has studied the reduction potential (E_{Red}) of aryl-substituted acetophenone azines (XPhC (Me)═N–N═C (Me)PhY. Celik, Zhu, and Sauro all have observed that the E_{Red} values of C═N bonds in the investigated compounds are linearly correlated to the Hammett parameter σ of substituents X and Y. Heyes[15] studied the reductions of the pyrylium and thiopyrylium cations. Yamataka[16] studied the reductions of para-substituted benzophenones. The results reported by Heyes and Yamataka also show that the E_{Red} values of the investigated compounds are greatly affected by the Hammett parameter σ of the substituents in molecules. Dosa et al[17] studied the relationship between the reduction rate and the E_{Red} of substituted azobenzene. Liu et al[18] studied the relationship between the reduction activity and the E_{Red} of para-substituted bromobenzene. The works of Dosa and Liu show that there is a linear relationship between the reduction activity and the E_{Red}. It can be seen from above mentioned study results that the substituent electronic effect, the E_{Red}, and the reduction activity of organic compounds are closely related. Therefore, exploring the relationship between the E_{Red} of organic compounds and the substituent electronic effect is of great significance for understanding the reduction activity of organic compounds.

Recently, our team[19] studied the effect of substituents on E_{Red} of para-disubstituted *N*-benzylidenebenzenamines XArCH═NArY (abbreviated as XBAY) and pointed out that when XBAY molecule receives an electron to be reduced to radical anion ($XBAY^{-}$), the electron distribution in the molecule is similar to that of molecular excited state rather than molecular ground state. Therefore, it is better to employ both the substituent ground state constant (Hammett constant σ) and the excited state substituent constant $\sigma^{\mathrm{ex}}_{\mathrm{CC}}$P[20] to correlate the E_{Red}, which can give good results. The method of employing the σ and the $\sigma^{\mathrm{ex}}_{\mathrm{CC}}$ has been applied well to quantify the E_{Red} of disubstituted *N*-phenyl-α-phenylnitrones XArCH═N(O)ArY (abbreviated as XPNY), disubstituted *N*-(phenyl-ethylene)-anilines XArC (Me)═NArY (abbreviated as XPEAY),[21] and *N*-benzylidenebenzenamines containing meta-substituents (abbreviated as *p*/*m*-XBAY-*p*/*m*).[22] Yuan et al[23] further extended this method to quantify the E_{Red} of other kinds of compounds, such as substituted naphthalene, and also obtained good results. The above mentioned results indicate that it is a new and effective method by employing the substituent ground state constant (Hammett constant σ) and the excited state substituent constant $\sigma^{\mathrm{ex}}_{\mathrm{CC}}$ [20] to quantify the E_{Red} of organic compounds.

It should be noted that aromatic compounds containing hydroxyl groups are also a sort of important organic compounds and are widely used. For examples, they were used in plasmon-assisted reactions on a metal surface,[24] exploring and elaborating the novel excited state dynamical behavior.[25] We analyzed carefully the E_{Red} values of more than 160 compounds of three series of compounds (involving XBAY, XPNY, and XPEAY) reported by Luo[21] and noticed that none of these compounds contains para-hydroxyl OH. We want to know if the equation for calculating the E_{Red} of XBAY reported in Luo[21] can be applied to the compounds 4-OHArCH═NArY (abbreviated as 4-OHBAY) and XArCH═NArOH-4′ (abbreviated as XBAOH-4′) containing hydroxyl OH or not. Thus, we synthesized two compounds, 4-OHArCH═NC_6H_5 and C_6H_5CH═NArOH-4′, and measured their E_{Red}, which is −2.55 and − 2.38 V, respectively. Then, the E_{Red} values of the two compounds were predicted by using Equation (1) reported by Luo.[21] The predicted values are −2.17 and − 2.07 V, respectively. It can be seen that the experimental E_{Red} values of them decrease more than 0.3 V compared with the predicted E_{Red} value, and the error of the experimental E_{Red} versus the predicted E_{Red} is much higher than the experimental error. This experimental phenomenon reminds us that the E_{Red} of bi-aryl Schiff bases containing para-hydroxyl OH may have additional behavior. To explore this topic, we designed and synthesized a series of bi-aryl Schiff bases containing para-hydroxyl OH, namely, 4-OHBAY and XBAOH-4′, and determined their E_{Red} values. We, employing the compounds 4-OHBAY and XBAOH-4′, try to investigate whether the E_{Red} of para-OH bi-aryl Schiff bases has additional behavior or not and expect to provide a theoretical reference for further understanding the change regularity of the E_{Red} of bi-aryl Schiff bases.

2 | RESULTS AND DISCUSSION

2.1 | Determination results of reduction potential

In order to investigate systematically the effect of para-hydroxyl OH on the E_{Red} of bi-aryl Schiff bases, 28 samples of model compounds, 4-OHBAY and XBAOH-4′ (Figure 1), were synthesized according to the method reported by Cao et al,[26–28] and the E_{Red} values of the obtained model compounds were determined. The E_{Red} data were listed in Table 1.

2.2 | Quantitative correlation of E_{Red} for the model compounds

In our previous work,[19,21,22] we reported the relationship between E_{Red} and the electronic effects of substituents X and Y for the compounds XBAY, who are without hydroxyl OH, and proposed Equation (1) to express the change regularity of their E_{Red}.[21]

$$E_{\text{Red}} = -2.02 + 0.46\sigma(\text{X}) + 0.13\sigma(\text{Y}) - 0.11\sigma_{\text{CC}}^{\text{ex}}(\text{X}) - 0.20Im \tag{1}$$

$$R = 0.9573, S = 0.06, n = 99, F = 257.70,$$

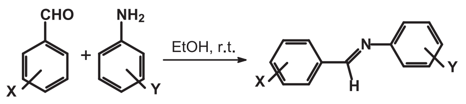
where the σ is Hammett electronic effect constant. The $\sigma_{\text{CC}}^{\text{ex}}$ is excited-state substituent constant. The X and Y in brackets indicate the substituents in XBAY molecules. The I_{m} is the indicator variable of group 3/3′-X/Y in the molecule XBAY, if there is a group at 3− or 3′ − position in the molecule XBAY, which are without hydroxyl OH, $I_{\text{m}} = 1$, otherwise, $I_{\text{m}} = 0$. The R, S, n, and F are the correlation coefficient, standard deviation, data points used in equation, and Fisher ratio, respectively.

Firstly, we took 127 compounds as a complicated E_{Red} data set, which involved 99 samples of XBAY reported by Luo[21] and 28 samples of 4-OHBAY and XBAOH-4′ in this work (Table 1). Then, based on these data E_{Red} set, a regression analysis was carried out, and Equation (2) was generated by employing the same parameters used in Equation (1).

$$E_{\text{Red}} = -2.17 + 0.53\sigma(\text{X}) + 0.14\sigma(\text{Y}) - 0.18\sigma_{\text{CC}}^{\text{ex}}(\text{X}) - 0.085Im \tag{2}$$

$$R = 0.8537, R^2 = 0.7285, S = 0.14, n = 127, F = 81.96.$$

The result of Equation (2) shows that the correlation of Equation (2) is worse than that of Equation (1) when the E_{Red} values involve these of XBAY, 4-OHBAY, and XBAOH-4′. The standard deviation S is up to 0.14 V, which is much higher than the experimental error. It means that the E_{Red} values of 4-OHBAY and XBAOH-4′ may be affected by other factors.



1-28

1-14: X= *p*-OH, Y(*p* or *m*) = CN, CF_3, F, Cl, Br, H, Me or OMe

15-28: Y = *p*-OH, X(*p* or *m*) = NO_2, CN, CF_3, F, Cl, Br, H, Me or OMe

FIGURE 1 The synthetic route of 4-OHBAY and XBAOH-4′ molecules

2.3 | Discussion

As regards the molecules 4-OHBAY and XBAOH-4′, they may occur ionization equilibrium in solution because of the presence of phenolic hydroxyl OH, as shown in Figure 2.

After dissociation of H^+ from molecule 4-OHBAY or XBAOH-4′, phenol-oxygen anion is formed, which can prevent the molecule from accepting electrons from the electrode and reduces the E_{Red}. In order to measure the effect of Figure 2A,B on the E_{Red} of 4-OHBAY and XBAOH-4′, we used the equalized electronegativity method proposed by Bratsch[35] (Pauling electronegativity) to calculate the partial charge Q_{O} of O atoms on the OH group in 4-OHBAY and XBAOH-4′, respectively. The O atom charges of 4-OH and 4′-OH are represented by the symbols Q_{O4} and $Q_{\text{O4'}}$, respectively, whose values can be seen in Table 1. Then, the Q_{O4} and $Q_{\text{O4'}}$ were employed as parameters to express the additional contributions of 4-OH and 4′-OH to the E_{Red}, and the contributions of σ and $\sigma_{\text{CC}}^{\text{ex}}$ of 4-OH and 4′-OH were all incorporated into the contributions of Q_{O}. Next, the Q_{O4} and $Q_{\text{O4'}}$ were added into Equation (2), and the 127 E_{Red} values were regressed once again. Then Equation (3) was obtained.

$$\begin{aligned} E_{\text{Red}} = {} & -2.02 + 0.46\sigma(\text{X}) + 0.13\sigma(\text{Y}) - 0.11\sigma_{\text{CC}}^{\text{ex}}(\text{X}) \\ & -0.20Im + 1.79\,Q_{\text{O4}} + 1.26\,Q_{\text{O4'}} \end{aligned} \tag{3}$$

$$R = 0.9750, R^2 = 0.9419, S = 0.06, n = 127, F = 324.09.$$

TABLE 1 The values of E_{Red}/V of the molecules 4-OHBAY and XBAOH-4′ and the σ_{F}, σ_{R}, and $\sigma_{\mathrm{CC}}^{\mathrm{ex}}$ of substituents X and Y

Compd.	X	Y	$\sigma_{\mathrm{F}}(\mathrm{X})^{a}$	$\sigma_{\mathrm{F}}(\mathrm{Y})^{a}$	$\sigma_{\mathrm{R}}(\mathrm{X})^{a}$	$\sigma_{\mathrm{R}}(\mathrm{Y})^{a}$	$\sigma_{\mathrm{CC}}^{\mathrm{ex}}(\mathrm{X})^{b}$	$\sigma_{\mathrm{CC}}^{\mathrm{ex}}(\mathrm{Y})^{b}$	$Q_{\mathrm{O}}{}^{c}$	$E_{\mathrm{Red,exp.}}{}^{d}$
1	4-OH	4′-OMe	0.33	0.29	−0.70	−0.56	−0.19	−0.50	−0.29	−2.68
2	4-OH	4′-Me	0.33	0.01	−0.70	−0.18	−0.19	−0.17	−0.30	−2.64
3	4-OH	H	0.33	0.00	−0.70	0.00	−0.19	0.00	−0.29	−2.55
4	4-OH	4′-F	0.33	0.45	−0.70	−0.39	−0.19	0.06	−0.28	−2.62
5	4-OH	4′-Cl	0.33	0.42	−0.70	−0.19	−0.19	−0.22	−0.29	−2.51
6	4-OH	4′-Br	0.33	0.45	−0.70	−0.22	−0.19	−0.33	−0.29	−2.50
7	4-OH	4′-CF_3	0.33	0.38	−0.70	0.16	−0.19	−0.12	−0.26	−2.58
8	4-OH	3′-OMe	0.33	0.12	−0.70	0.00	−0.19	0.10	−0.29	−2.48
9	4-OH	3′-Me	0.33	−0.07	−0.70	0.00	−0.19	−0.03	−0.30	−2.50
10	4-OH	3′-F	0.33	0.34	−0.70	0.00	−0.19	0.02	−0.28	−2.41
11	4-OH	3′-Cl	0.33	0.37	−0.70	0.00	−0.19	0.02	−0.29	−2.40
12	4-OH	3′-Br	0.33	0.39	−0.70	0.00	−0.19	−0.03	−0.29	−2.42
13	4-OH	3′-CF_3	0.33	0.43	−0.70	0.00	−0.19	0.09	−0.26	−2.47
14	4-OH	3′-CN	0.33	0.56	−0.70	0.00	−0.19	0.56	−0.29	−2.40
15	4-OMe	4′-OH	0.29	0.33	−0.56	−0.70	−0.50	−0.19	−0.29	−2.50
16	4-Me	4′-OH	0.01	0.33	−0.18	−0.70	−0.17	−0.19	−0.30	−2.44
17	H	4′-OH	0.00	0.33	0.00	−0.70	0.00	−0.19	−0.29	−2.38
18	4-F	4′-OH	0.45	0.33	−0.39	−0.70	0.06	−0.19	−0.28	−2.41
19	4-Cl	4′-OH	0.42	0.33	−0.19	−0.70	−0.22	−0.19	−0.28	−2.17
20	4-Br	4′-OH	0.45	0.33	−0.22	−0.70	−0.33	−0.19	−0.29	−2.35
21	4-CN	4′-OH	0.51	0.33	0.15	−0.70	−0.70	−0.19	−0.28	−1.91
22	4-NO_2	4′-OH	0.65	0.33	0.13	−0.70	−1.17	−0.19	−0.27	−1.87
23	3-OMe	4′-OH	0.12	0.33	0.00	−0.70	0.10	−0.19	−0.29	−2.35
24	3-Me	4′-OH	−0.07	0.33	0.00	−0.70	−0.03	−0.19	−0.30	−2.40
25	3-F	4′-OH	0.34	0.33	0.00	−0.70	0.02	−0.19	−0.28	−2.22
26	3-Cl	4′-OH	0.37	0.33	0.00	−0.70	0.02	−0.19	−0.28	−2.23
27	3-Br	4′-OH	0.39	0.33	0.00	−0.70	−0.03	−0.19	−0.29	−2.27
28	3-CN	4′-OH	0.56	0.33	0.00	−0.70	0.56	−0.19	−0.28	−2.21

[a] Values were from Hansch et al.[29]

[b] Values were from author's previous studies.[20,30–34]

[c] For 4-OH, Q_{O} indicates Q_{O4}; For 4′-OH, Q_{O} indicates $Q_{\mathrm{O4'}}$.

[d] Values determined by this work.

Compared with Equation (2), the correlation coefficient R of Equation (3) is significantly increased, and the standard deviation S is reduced from 0.14 to 0.06 V, which is less than half of the S in Equation (2) and is within the experimental error. The intercept of Equation (3), the coefficients in front of the variables $\sigma(\mathrm{X})$, $\sigma(\mathrm{Y})$, $\sigma_{\mathrm{CC}}^{\mathrm{ex}}(\mathrm{X})$, and I_{m} all are exactly the same as those in Equation (1). Equation (3) indicates that (a) 4-OH and 4′-OH groups have indeed additional effects on the E_{Red}, and the more negative the charge of O atom is, the more the E_{Red} will be reduced. (b) The effects of 4-OH and 4′-OH on the E_{Red} are different from those of other groups X and Y of XBAY molecules; (c) the additional effect of 4-OH on the E_{Red} is different from that of 4′-OH, and the former is greater than the latter. It should be pointed out that because the charge Q_{O} of O

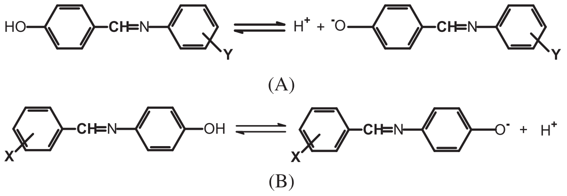


FIGURE 2 The ionization equilibrium for 4-OHBAY A, and XBAOH-4′ B

atom is usually negative, the additional effect of OH makes the E_{Red} decrease. For example, in molecule, 4-OHArCH=NC$_6$H$_5$, the Q_{O4} of OH is −0.29, the additional effect of 4-OH is $1.79 \times (-0.29) = -0.52$ V; in molecule C$_6$H$_5$CH=NArOH-4′, the additional effect of 4′-OH is $1.26 \times (-0.29) = -0.37$ V.

The additional effect of para-hydroxyl OH on the E_{Red} can be attributed to the ionization shown in Figure 2. In order to illustrate the effect of ionization on the E_{Red}, we briefly discuss it as follows. Taking substituents NMe$_2$, OH, and OMe for examples, their Hammett constant σ values are −0.83, −0.37, and − 0.27,[29] respectively, while the E_{Red} values of 4-NMe$_2$BAH, 4-OHBAH, and 4-OMeBAH are −2.11, −2.55, and − 1.98 V, respectively. The E_{Red} values of HBANMe$_2$-4′, HBAOH-4′, and HBAOMe-4′ are −2.01, −2.38, and − 2.06 V, respectively. Among the three substituents mentioned above, the σ value of OH is in the middle position, while the E_{Red} value of the compounds containing OH is the smallest in above two series of compounds, respectively. This phenomenon cannot be reasonably explained by the contribution of σ value of substituents but can only be explained by the additional effect of OH, that is, the E_{Red} decrease originates from the ionization of OH. In order to compare intuitively the difference of effect of 4-OH versus 4′-OH on the E_{Red}, we plot the E_{Red} with 4-OHBAX and XBAOH-4′ isomers containing the same group X and got Figure 3. It can be seen from Figure 3 that 4-OH reduces E_{Red} more than 4′-OH does.

There are 127 compounds employed in Equation (3). The substituents in these compounds are distributed in the para- and meta-positions of aldehyde and amine aromatic rings, and the substituents involve a wide electronic effect range, which is in going from strong electron-donating group[19] NMe$_2$ (σ = −0.83) to strong electron-withdrawing group NO$_2$ (σ = 0.78). The E_{Red} range is also wide, which is in going from −1.43 (compound 4-NO$_2$BACN-4′) to −2.68 V (compound 4-OHBAOMe-4′). The obtained Equation (3) has good correlation; its standard error is only 0.06 V. The average absolute error of the calculated value by Equation (3) versus the experimental value is only 0.05 V, which is within the experimental error. Figure 4 is the plot of the calculated value $E_{\mathrm{Red,cal.}}$ Versus the experimental value $E_{\mathrm{Red,exp}}$. From the calculation results and Figure 4, it can be seen that Equation (3) expresses well the additional effect of 4-OH and 4′-OH on the change regularity of E_{Red} of bi-aryl Schiff bases.

Also the results of this paper are consistent with the experimental facts reported. For example, the reduction of compounds containing 4-OH-ArCH=N structure[11] and other X-ArCH=N structure[5–10] by NaBH$_4$, the former takes longer than the latter under the same reduction conditions, that is, the former is more difficult to be reduced.

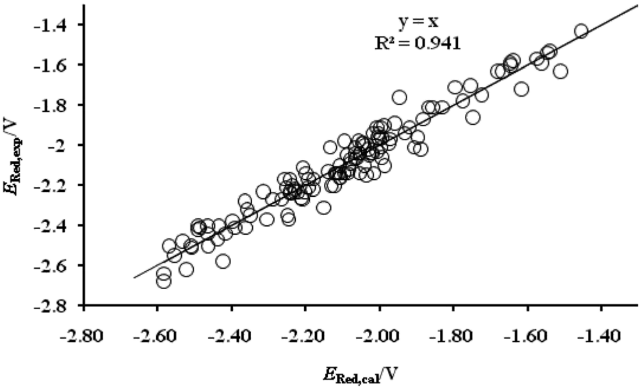


FIGURE 4 Plot of the calculated $E_{\mathrm{Red,cal}}$ with Equation (3) versus the experimental $E_{\mathrm{Red,exp}}$ for the 127 model compounds

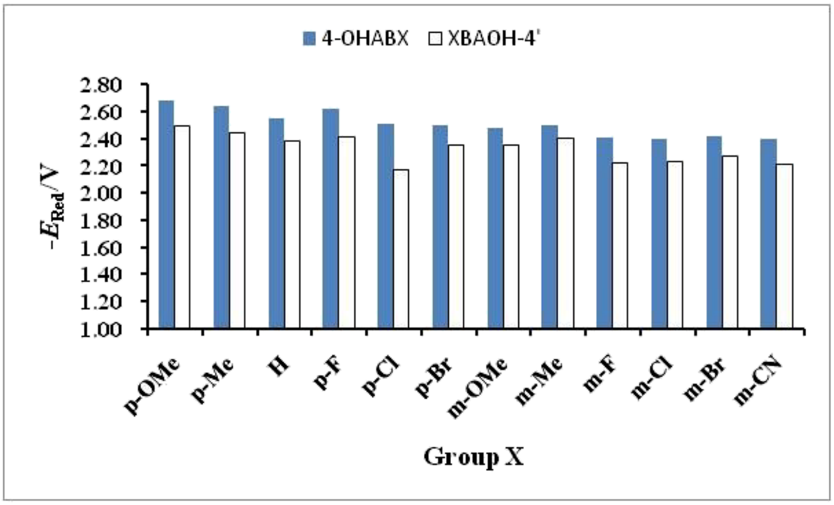


FIGURE 3 Comparison of the E_{Red} for the isomers 4-OHBAX and XBAOH-4′ containing the same group X

3 | CONCLUSION

By systematically and quantitatively analyzing the factors affecting the E_{Red} of 4-OHBAY, XBAOH-4′, and XBAY without hydroxyl OH, we can obtain the conclusions: (a) Because phenolic hydroxyl OH can dissociate H^+ to form phenolic oxygen anion, the change regularity of E_{Red} of compounds 4-OHBAY and XBAOH-4′ is somewhat different from that of E_{Red} of compounds XBAY without hydroxyl OH. That is to say, OH group has a additional effect on the E_{Red}, which decreases E_{Red} value. (b) The additional effect of 4-OH on the E_{Red} is different from that of 4′-OH. The effect of OH group attached to the aldehyde aromatic ring on the E_{Red} is more than that of OH attached to the amine aromatic ring.

The new experimental phenomena observed in this paper are helpful to deepen the understanding of the change regularity of E_{Red} of disubstituted *N*-benzylidenebenzenamines XBAY. It should be pointed out that this paper focused on the influence of 4/4′-OH on the E_{Red} of Schiff bases; it needs further to be investigated whether 3/3′-OH and 2/2′-OH in molecules XBAY also have additional effect on the E_{Red} or not.

4 | EXPERIMENTAL SECTIONS

4.1 | Synthesis of model compounds

Firstly, 20 mL absolute ethanol was added into 50 mL round bottom flask, then substituted benzaldehyde (10 mmol) and substituted aniline (10 mmol) were added into the flask. Next, the mixture was stirred at room temperature for about 30 minutes, let it stand until solid is precipitated, and the crude product was filtered out and recrystallized with absolute ethanol to purify the product. The molecular structure of the target product was characterized by ^{1}H NMR and ^{13}C NMR (AV500 MHZ) after vacuum drying. The detailed data and NMR spectra of model compounds in Table 1 were reported in the Supporting Information.

4.2 | Determination of E_{Red} of the model compounds

The reduction potentials E_{Red} of the model compounds were determined via the following method, employing anhydrous acetonitrile as solvent and 0.1 M (n-Bu)$_4$NPF$_6$/CH$_3$CN solution as supporting electrolyte, using a glassy carbon disk consisting of a standard three-electrode cell as work electrode, taking 0.01 mol/L AgNO$_3$/Ag (in 0.1 mol/L n-Bu$_4$NPF$_6$/CH$_3$CN) as reference electrode and a platinum wire as counter electrode. Then the E_{Red} values were obtained by using cyclic voltammetry (CV), scanning with CS300 electrochemical instrument (Ferrocene as an internal standard).

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