Kinetic Screening for the Acid-Catalyzed Hydrolysis of Some Hydrophobic Fe(II) Schiff Base Amino Acid Chelates and Reactivity Trends in the Presence of Alkali Halide and Surfactant

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ABSTRACT: Kinetics of acid-catalyzed hydrolysis of some high-spin Fe(II) Schiff base amino acid complexes were followed spectrophotometrically at 298 K under pseudo–first-order conditions. The studied ligands were derived from the condensation of 5-bromosalicylaldehyde with different four amino acids (phenylalanine, aspartic acid, histidine, and arginine). The acid hydrolysis reaction was studied in aqueous media and in the presence of different concentrations of the alkali halide (KBr) and cationic surfactant (cetyl-trimethyl ammonium bromide, CTAB). The general rate equation was suggested to be rate $= k_{obs}[\text{complex}]$, where $k_{obs} = k_2[H^+]$. The increase in [KBr] enhances the reactivity of the reaction, and the addition of CTAB to the reaction mixture accelerates the reaction reactivity. The obtained kinetic data were used to determine the values of $\delta_m \Delta G^{\#}$ (the change in the activation barrier) for the studied complexes when transferred from "water to water containing different [KBr]" and from "water to water containing altered [CTAB]." © 2015 Wiley Periodicals, Inc. Int J Chem Kinet 47: 501–508, 2015

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INTRODUCTION

Iron plays a unique role in biological systems. Enzymes containing iron are of crucial importance in electron transfer reactions and in the activation and transport of small molecules, such as molecular oxygen [1,2].

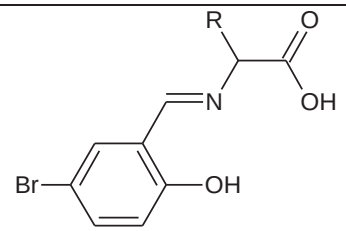
The Schiff base compounds constitute an important class of ligands, which have been extensively studied in coordination chemistry. Schiff base ligands have gained much importance in medicinal inorganic chemistry in view of their diverse biological, pharmacological, antitumor activity, and their exceptional chelating ability [3–5].

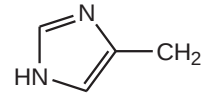
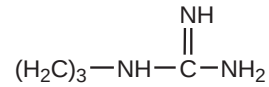
Schiff bases derived from amino acids are known to act as good chelating agents [6,7]. The interest in studying these compounds and their metal complexes arises from their behavior as efficient biological active [8] and cytotoxic [9] agents. The imine group of the Schiff base amino acid is involved in many different biological processes: decarboxylation, transamination, electron transfer, etc. For example, salicylaldehyde amino acid Schiff bases have been used to model *N*-pyridoxylidene amino acids [10].

From the literature, it was revealed that free Schiff base ligands show less or no cytotoxic activity in comparison to their metal complexes [11–14]. The use of appropriate multidentate chelating ligands removes the undesirable effects of metal ions and can deactivate either the carcinogenic metal or the enzyme, which is necessary for the rapid growth of both the healthy and malignant cells.

Iron Schiff base complexes have wide and variable applications such as catalysts for oxidation of sulfides [15], phenol [16], cycloalkanes [17], and decomposition of H_2O_2 [18]. Also they have biological importance as antimicrobial [13,19–21] compound. Iron(II) Schiff base amino acid complexes are important chelates containing a metal in unstable, low oxidation state, as well as involving unstable ligands [13,14,22,23]. Even though, little attention is devoted to their reactivities toward acid hydrolysis [24–27]. Therefore, the recent work investigates the acid-catalyzed hydrolysis of some biologically active high-spin Fe(II) Schiff base amino acid chelates in aqueous medium and in the presence of different [KBr] and [CTAB] (cetyl-trimethyl ammonium bromide) to gain more information about their stability, which can widen the area of their applications.

The presence of micelles in the reaction mixture has a tendency to influence the reaction rates by enhancing it or by retarding the rates of reaction because surfactants are the amphiphilic molecules consisting of the polar or charged head group and nonpolar hydrocarbon chain [28]. Moreover, the modification in the reaction medium by adding surfactant molecules [27,29–32] affects the solvating power and thus alters the hydrolytic rate. The obtained kinetic data from the current work were used to determine the change in the activation barrier ($\delta_m \Delta G^{\#}$) values for the studied complexes when transferred from water to water containing different [KBr] or [CTAB].



Complex	R
BS1	$CH_2C_6H_5$
BS2	CH_2COOH
BS3	
BS4	

Scheme 1 Structures of the ligands and abbreviations of their corresponding complexes.

EXPERIMENTAL

The investigated iron(II) complexes were prepared according to the literature method [14,22], from the condensation of α-amino acid (L-phenylalanine, L-aspartic acid, L-histidine, or L-arginine) with 5-bromosalicylaldehyde (cf. Scheme 1). The studied Schiff base ligands were synthesized by mixing equimolars from both the aldehyde and amino acid in aqueous–ethanolic solution. The mixture was refluxed for 5 h then evaporated to one-fourth of its original volume. The obtained precipitates were washed with ethanol then with diethyl ether and finally dried in vacuum over silica gel. The complexes were prepared by stirring ethanolic solutions from the prepared Schiff base ligand with Fe(II) salt (2:1). Few drops of glacial acetic acid were added to prevent the oxidation of Fe(II) [22]. The obtained complexes were recrystallized from ethanol.

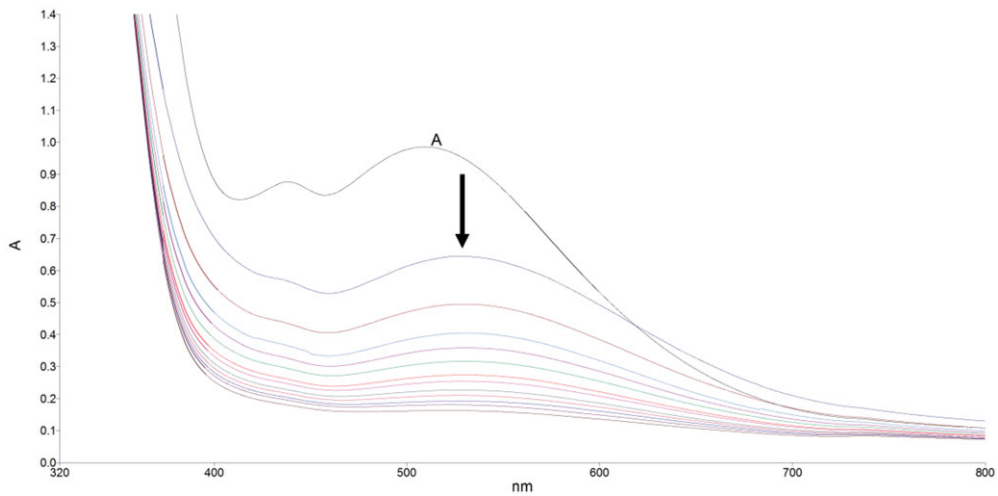


Figure 1 Repeated spectral scan of BS1 at $[H^+] = 0.13$ M, $I = 0.14$ M, $t = 3$ min, $[BS1] = 6.67 \times 10^{-4}$ M, and temperature = 298 K (**A**) spectral scan of BS1 complex before the addition of acid.

All the chemicals and reagents used such as HCl, KBr, and CTAB were of analytical grade.

The rate measurements of the acid hydrolysis reaction of the studied complexes were followed spetrophotometrically on a Perkin Elmer Lambda 35 spectrophotometer, via monitoring the absorbance (A) – time (t) dependence at λ_{max} appropriate to each complex. The observed first-order rate constants (k_{obs}) were calculated using least-squares procedures of ln(A) versus t plots. The kinetic measurements were performed in aqueous medium under pseudo–first-order conditions by mixing a multifold greater concentration of the acid than that of the complex $\{(0.033 \leq [H^+], M \leq 0.167)$ and $[complex] = 6.67 \times 10^{-4}$ M)} at 298 K and constant ionic strength. Moreover, the investigated reaction was carried out in the presence of different concentrations of both KBr ($0.12 \leq [KBr], M \leq 0.74$) and CTAB ($0.67 \times 10^{-3} \leq [CTAB], M \leq 5 \times 10^{-2}$) at 298 K for BS2, BS3, and BS4. The effects of KBr and CTAB on the reactivity of acid hydrolysis of complex BS1 were not stated because of the lower solubility of BS1 resulted in formation of precipitation on mixing its solution with other component under the mentioned reaction conditions. Furthermore, the change in the activation barrier ($\delta_m \Delta G^{\#}$) values for the studied complexes (BS2, BS3, and BS4) when transferred from water to water containing different concentrations of KBr or CTAB was determined from the kinetic data.

RESULTS

Effect of $[H^+]$

On the addition of the acid to the studied Fe(II) Schiff base complexes, the color of the complex was changed from dark red to violet, then the formed violet color decayed slowly with time to be colorless. Figure 1 illustrates the bathochromic shift occurred in λ_{max} of BS1 complex in the presence of the acid, which can be inferred from the protonation of the complex [24]. The protonated intermediate precursor decayed in a first-order kinetics manner (cf. Fig. 1).

Table I The Observed First-Order Rate Constant ($10^4\ k_{obs.}$, s^{-1}) Values for the Acid Hydrolysis of the Investigated Fe(II) Schiff Base Amino Acid Complexes in Aqueous Medium at Different $[H^+]$, $[Complex] = 6.67 \times 10^{-4}$ M, I = 0.17 M, and 298 K

	Complex			
10 $[H^+]$ (M)	BS1	BS2	BS3	BS4
1.67	3.19	19.80	9.34	9.07
1.33	2.61	14.80	7.46	6.89
1.00	1.74	11.40	5.29	4.83
0.83	1.41	9.60	4.17	3.58
0.67	1.11	7.02	3.19	3.02
0.50	0.74	4.99	2.46	1.87
0.33	0.45	2.75	1.65	1.25

The first-order rate constant values (k_{obs}) are given in Table I. The relation between k_{obs} and $[H^+]$ was found to be linear (cf. Fig. 2) and in a good agreement with the following equation:

$$k_{obs} = k_2[H^+] \tag{1}$$

Therefore, the overall rate equation under the implemented conditions of pseudo–first-order kinetics can be suggested as follows [24]:

$$\text{Rate} = \{k_2[H^+]\}[\text{complex}] \tag{2}$$

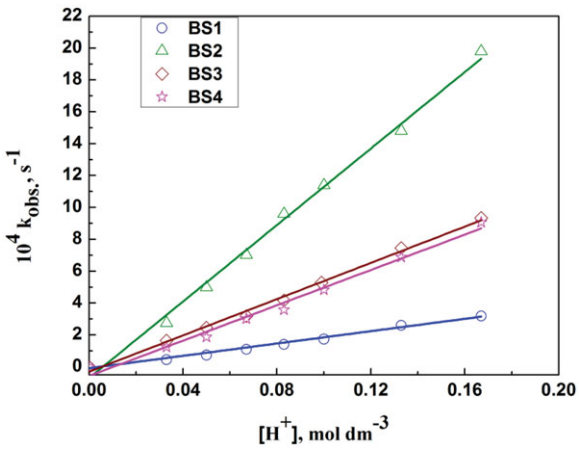


Figure 2 The relation between $[H^+]$ and k_{obs} values for the acid hydrolysis reaction of the investigated complexes in aqueous solution at [complex] = 6.67 × 10^{-4} M, I = 0.17 M, and 298 K.

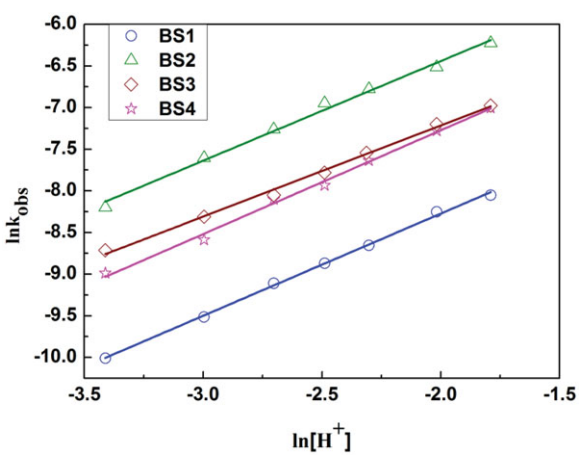


Figure 3 Plots of $\ln(k_{obs})$ as a function of $\ln[H^+]$ in aqueous solution at [complex] = 6.67 x 10^{-4} M, I = 0.17 M, and 298 K.

$$\text{Rate} = k_{obs}[\text{complex}] \tag{3}$$

the value of the reaction order (n) as a function of $[H^+]$ was determined by a least-squares procedure from the slopes of the linear plots of $\ln(k_{obs})$ versus $\ln[H^+]$ (cf. Fig. 3 and Table II) according to the following equation [18,24]:

$$\ln(k_{obs}) = \ln k' + n \ln[H^+] \tag{4}$$

Effect of [Alkali Halide]

The kinetics of the acid hydrolysis of the studied hydrophobic Fe(II) Schiff base complexes has been studied in the presence of various concentrations of the alkali halide KBr (0.12–0.74 M). The progress of the reaction was monitored by following the change in absorbance versus time at λ_{max} for each complex.

It was found that the values of the observed first-order rate constants (k_{obs}) increase with increasing [KBr] at a constant $[H^+]$ (cf. Fig. 4 and Table III). The increases in k_{obs} values differ from one complex to other depending on the used ligand and the hydrophobic character of each complex.

The change in the activation barrier ($\delta_m \Delta G^{\#}$) values for the studied complexes when transferred from aqueous solution into aqueous solution containing different [KBr] is shown in Table III and Fig. 5 from the ratio of rate constants of the acid hydrolysis reaction in the presence of various [KBr] (k'_{2S}) to the corresponding values in the aqueous solution (k'_{2W}) according to the following relation [33–35]:

$$\delta_m \Delta G^{\#} = -RT \ln(k'_{2S}/k'_{2W}) \tag{5}$$

Table II The Reaction Order (n) and k' Values as Determined by the Least Squares of the Plots $\ln[H^+]$ versus $\ln(k_{obs})$ Obtained for the Acid Hydrolysis of the Investigated Complexes in Aqueous Medium, at [complex] = 6.67 × 10^{-4} M, I = 0.17 M, and 298 K

	Complex			
Parameter	BS1	BS2	BS3	BS4
n	1.23	1.19	1.09	1.25
$10^3 k'$	2.97	17.40	6.54	8.39

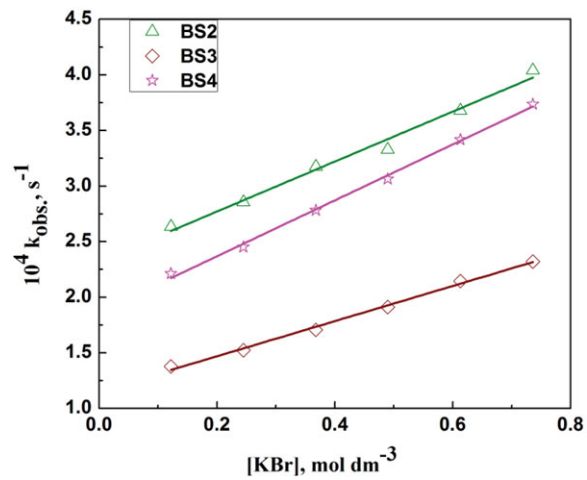


Figure 4 The relation between [KBr] and k_{obs} values for the acid hydrolysis reaction of the investigated complexes in aqueous solution at $[H^+]$ = 0.05 M, [complex] = 6.67 × 10^{-4} M, and 298 K.

Table III The Observed First-Order Rate Constant (10^4 k_{obs}, s^{-1}) Values for the Acid Hydrolysis of the Investigated Fe(II) Schiff Base Amino Acid Complexes in Different Added Molar Concentrations of KBr, at $[H^+] = 0.05$ M, [complex] $= 6.67 \times 10^{-4}$ M, and 298 K and the Change in the Activation Barrier ($\delta_m \Delta G^{\#}$, kJ mol^{-1}) Values for the Complexes When Transferred from Water to Water Containing Different [KBr]

[KBr] (M)	Complex					
	BS2		BS3		BS4	
	k_{obs}	$\delta_m \Delta G^{\#}$	k_{obs}	$\delta_m \Delta G^{\#}$	k_{obs}	$\delta_m \Delta G^{\#}$
0	2.32	0	1.15	0	1.87	0
0.12	2.64	–0.31	1.38	–0.44	2.21	–0.40
0.25	2.86	–0.51	1.52	–0.68	2.45	–0.65
0.37	3.17	–0.77	1.71	–0.96	2.78	–0.96
0.49	3.33	–0.88	1.91	–1.23	3.06	–1.19
0.61	3.68	–1.12	2.15	–1.52	3.42	–1.46
0.74	4.04	–1.34	2.32	–1.70	3.74	–1.68

Effect of [CTAB]

The observed first-order rate constants (k_{obs}) for the acid hydrolysis reaction in the presence of different concentrations of the cationic surfactant CTAB were calculated and cited (cf. Table IV and Fig. 6). The values of k_{obs} increase with increasing the concentration of the added surfactant (CTAB) at a constant $[H^+]$. Moreover, the values of $\delta_m \Delta G^{\#}$ (change in the activation barrier) for the studied complexes when transferred from aqueous solution into aqueous solution containing different [CTAB] are calculated (Fig. 7) from reaction rates in the appropriate media, using the relationship

$$\delta_m \Delta G^{\#} = -RT \ln(k''_{2Surf}/k''_{2W}) \quad (6)$$

where k''_{2Surf} is the rate constants of the acid hydrolysis reaction in the aqueous solutions containing various [CTAB] and k''_{2W} is that for the aqueous medium only.

Table IV The Observed First-Order Rate Constant (10^4 k_{obs}, s^{-1}) Values for the Acid Hydrolysis of the Investigated Fe(II) Schiff Base Amino Acid Complexes in Aqueous Medium at Different [CTAB], $[H^+] = 0.13$ M, [complex] $= 6.67 \times 10^{-4}$ M, and 298 K and the Change in the Activation Barrier ($\delta_m \Delta G^{\#}$, kJ mol^{-1}) Values for the Complexes When Transferred from Water to Water Containing Various [CTAB]

10^3 [CTAB] (M)	BS2		BS3		BS4	
	k_{obs}	$\delta_m \Delta G^{\#}$	k_{obs}	$\delta_m \Delta G^{\#}$	k_{obs}	$\delta_m \Delta G^{\#}$
0	9.09	0	4.55	0	4.85	0
0.67	10.1	−0.26	4.72	−0.09	5.05	−0.10
2	10.8	−0.42	4.82	−0.14	5.37	−0.25
4	11.6	−0.59	4.94	−0.20	5.51	−0.31
6	12.6	−0.79	5.30	−0.37	5.85	−0.45
8	13.1	−0.89	5.50	−0.46	6.12	−0.56
10	14.3	−1.10	6.09	−0.71	6.24	−0.61
15	14.7	−1.16	7.00	−1.04	6.43	−0.68
20	15.3	−1.26	7.32	−1.15	6.54	−0.72
30	16.2	−1.40	7.54	−1.22	6.77	−0.81
40	16.6	−1.46	7.76	−1.29	6.91	−0.86
50	17.5	−1.59	8.02	−1.37	7.10	−0.92

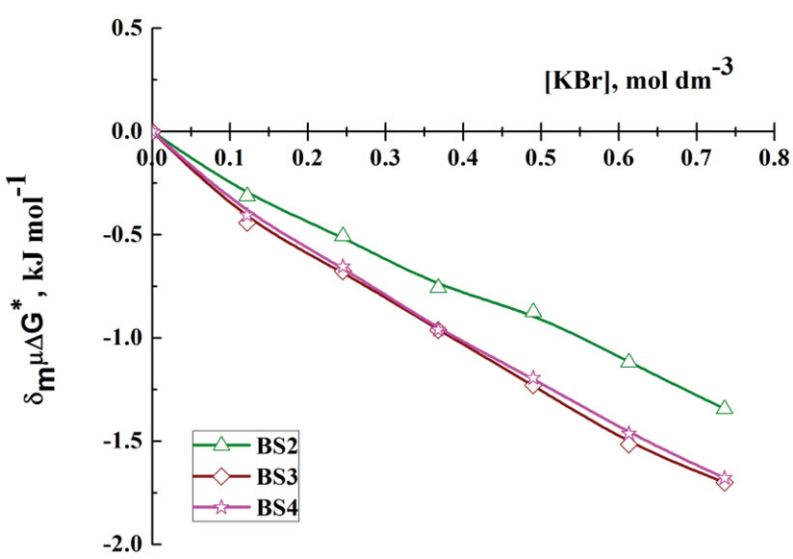


Figure 5 Trends in transfer chemical potential of the investigated complex in the presence of different [KBr].

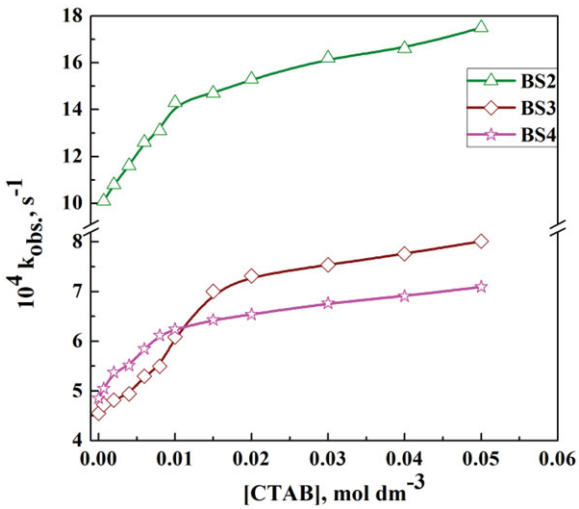


Figure 6 The variation of k_{obs} values for the acid hydrolysis reaction of the investigated complexes in aqueous solution with [CTAB] at $[H^+] = 0.13$ M, [complex] $= 6.67 \times 10^{-4}$ M, and 298 K.

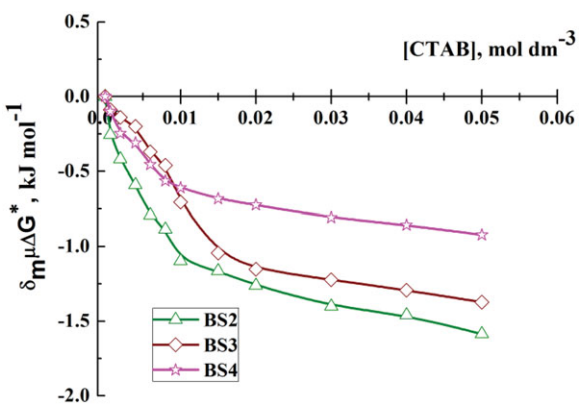


Figure 7 Trends in transfer chemical potential of the investigated complex in the presence of different [CTAB].

DISCUSSION

In the recent work, the kinetics of the acid-catalyzed hydrolysis of some high-spin Fe(II) Schiff base amino acid complexes were monitored in aqueous medium at 298 K. The stability range [14] of the prepared complexes was found to be from pH 4 to 10, which is similar to Fe(II) Schiff base amino acid complexes derived from both 2-hydroxy-1-naphthaldehyde and salicylaldehyde [22] or sodium 2-hydroxybenzaldehyde-5-sulfonate [20].

The distinguished bands for the investigated complexes in the UV/visible spectra lies at $\lambda_{max} = 458$–490 nm (dark red color of the complex′s solution). But on addition of the acid, a bathochromic shift occurred

$$\underset{\mathbf{A}}{[Fe(II)L_2]} + H^+ \underset{\text{Fast}}{\overset{K_a}{\rightleftharpoons}} \underset{\mathbf{A^+}}{[Fe(II)L_2H]^+} \quad (7)$$

$$\underset{\mathbf{A^+}}{[Fe(II)L_2H]^+} + H_2O \xrightarrow[\text{Slow}]{k_b} \underset{\mathbf{B}}{[Fe(II)L_2(H)(OH)]} + H^+ \quad (8)$$

$$\underset{\mathbf{B}}{[Fe(II)L_2(H)(OH)]} \xrightarrow[\text{Fast}]{k_c} \text{Products} \quad (9)$$

Scheme 2 The suggested reaction mechanism of the acid-catalyzed hydrolysis of the studied Fe(II) complexes.

at 530 nm as shown in Fig. 1 (the color of the complex′s solution is converted to violet). Then, this new formed band decays in a first-order kinetics trend in [complex]. This can be attributed to the protonation of the complex in a fast step to form a protonated precursor, which is attacked by water on its anil carbon [24].

The suggested mechanism of the investigated reaction is presented in Scheme 2 [24].

Under the recent study conditions ($0.033 \leq [H^+]$, M ≤ 0.167), the investigated chelates are protonated and exist as their conjugate acids because of the fast pre-equilibrium step involved the attack of H^+ on (HC=N) to give ($HCNH^+$). After that the formed intermediate decayed slowly in the rate-determining step of the reaction. This suggestion has a realistic theoretical evidence, because the anil carbon assumes considerable carbonium ion characteristics induced by conjugate acid formed in these acidic media and that it is more susceptible to nucleophilic attack by water [24,36]. After this, the protonated complex decays rapidly into the final products of the reaction, which were suggested to be amino acid and the aldehyde with the oxidation of Fe(II) to Fe(III) under the effect of the dissolved oxygen in the reaction mixture.

On applying the steady-state approximation for the determination of the concentrations of the reaction intermediates A^+ and B, where $[A]_T = [A] + [A^+] + [B]$, the following reaction rate can be derived as

$$\text{Rate} = \frac{K_a k_b k_c [A]_T [H^+]}{1 + K_a k_b [H^+] + K_a k_c [H^+]} \quad (10)$$

and k_{obs} can be deduced to be

$$k_{obs} = \frac{K_a k_b k_c [H^+]}{1 + K_a k_b [H^+] + K_a k_c [H^+]} \quad (11)$$

Table I illustrates the reactivity trends of acid hydrolysis of the investigated Fe(II) complexes with

various amino acid moieties in the presence of different [H^+]. It seems that these reactivity trends are controlled by the hydrophobic character of the studied complexes [24]. The reactivity decreases in the following order: BS2 > BS4 ≥ BS3 > BS1 (according to the slope values of the lines in Fig. 2), whereas the order of the hydrophobicity resulted from the amino acid moieties increases in the following order: BS2 < BS4 < BS3 < BS1. It is clear that the less hydrophobic complex is the more reactive one.

As shown from Table III and Fig. 4, there is an enhancement in the rate constant upon adding KBr to the reaction mixture. The enhancement of the rate constant with increasing [KBr] can be ascribed to the dominating hydration characteristics of KBr leading to the disintegration of the aqua cage around the reactants thus giving them more opportunity to react [37].

Figure 5 illustrate the dependence of the transfer potentials on [KBr]. It is clear that the general trend for the transfer potentials is to decrease with increasing the concentration of the added alkali halide to the reaction mixture because of the increase in the reactivity of the acid hydrolysis reaction in the same direction (cf. Table III). The less hydrophobic complex (BS2) exhibited less negative values of transfer potentials than did higher hydrophobic chelates (BS4 and BS3).

Micelles, as well as other association colloids, can act as microreactors concentrating, separating, or diluting reactants, and in this manner, they may have considerable effects on chemical reactivity of different reactions [38]. Because of their abnormal characteristics, micelles have been extensively exploited as models for understanding processes that are important for grasping the complex behavior encountered in biological assemblies [39,40]. It was observed that the observed rate constant values of the reaction increase with increasing [CTAB] (Table IV and Fig. 6), which clearly demonstrate that the CTAB catalytic effect is not only above but also even below the CMC (i.e., micellar as well as premicellar catalyses are observed) [41–43]. The interaction between the surfactants and the complexes decreases the surface area of the reactants and increases the reaction rates. Micellization increases the reaction rate because on micellization counterions can be attracted or repelled more effectively. The frequency of molecular collisions increases as a consequence of the close association of the two reacting species at the micellar interface. The location of the exact site of the reaction is not accurate, but localization of the reactants can be considered [41]. This trend may result from surface catalysis. Thus in the micellar medium, adsorption of reactants on the micellar surface enhances the reaction rate to a great extent. For this, with increasing [CTAB], the observed rate constant (k_{obs}) values increase too. Thus the catalytic effect of micelles is observed, which can be attributed to surface catalysis. Moreover, on increasing [CTAB], the total area of the micellar surface increases and therefore the degree of the surface catalytic effect and reaction rate increase [29]. The plots of k_{obs} against [surfactant] (Fig. 6) show a gradual increase in rates with [surfactant]. Such a type of behavior of the studied surfactants may be due to more and more solubilization of both the reactants and/or its intermediate complex with increasing [surfactant] and overall reaction rates [27,30]. The change in the activation barrier ($\delta_m \Delta G^{\#}$) values for the studied complexes transferred from "water to water containing different concentration from CTAB" was determined from the kinetic data (cf. Fig. 7). It seems that the reactivity was controlled by the hydrophobicity of the studied complexes. The addition of CTAB to the reaction medium enhances the reaction rate for the less hydrophobic complex (BS2) greater than that for the higher hydrophobic chelates (BS4 and BS3).

CONCLUSIONS

In this work, the acid-catalyzed hydrolysis of some hydrophobic Fe(II) Schiff base amino acid chelates was screened at pH < 2 in aqueous medium and in the presence of variable concentrations from alkali halide (KBr) and cationic surfactant (CTAB). That is to gain more information about the stability of these important complexes as their reactivities in basic medium were studied previously [44,45]. The rate of acid hydrolysis reaction of the studied compounds increases with the decrease in pH. The proposed reaction mechanism points toward the slow attack of water on the anil carbon conjugate acid ($HCNH^+$), formed in a fast preequilibrium step. The steady-state derived rate equation was formulated in accordance with the reaction mechanism, which suggests the formation of an intermediate species during the course of the reaction. The reactivity trends in the studied reactions are controlled by hydrophobic/hydrophilic nature of the studied complexes. It was found that the addition of alkali halide (KBr) enhances the acid hydrolysis of the investigated hydrophobic chelates. Moreover, the addition of CTAB catalyzes the reaction rate due to the enhancement of the interaction of the reactant in the micellar. The change in the activation barrier ($\delta_m \Delta G^{\#}$) values for the studied complexes when transferred from "water to water containing different concentration from KBr or CTAB " was determined from the kinetic data.

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