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# Reactions of polyhomofunctional organic compounds: 3: Kinetics of hydrolysis of ethylene bridged salicylaldimine Schiff base and its mononuclear copper (II) complex

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Kinetic studies of hydrolysis of the two isochemical azomethine sites present in 1,2-bis(salicylidinimino)ethelene (**1**) abbreviated as EDA-Sal and one of its novel mononuclear copper (II) complex abbreviated as Cu(EDA-Sal) (**2**) have been studied in the varied *p*H range 1-12, and at different temperatures. The investigations indicate that based on *p*H, the aliquot (**1**) exists in several acid-base forms. The acid-base equilibrium and resonance effects have been found to be responsible for near simultaneous hydrlysis of azomethine sites present in the molecules. The repetitive spectral profiles of both the selected compounds reveal that the kinetic mixtures exhibit isosbestic points in the entire *p*H range of study, indicating the involvement of only two absorbing species throughout the reaction path. The hydrolysis follows pseudo-first order kinetics for both Schiff base, and its corresponding complex. Interestingly, except in the acidic range, the complex is found to be stable. The difference in rate of hydrolysis of (**2**) from that of (**1**) and the probable influencing factors are discussed. By Arrhenius and Eyring methods, activation energy $E_a$ and other thermodynamic parameters such as $\Delta H^{\#}$, $\Delta G^{\#}$ and $\Delta S^{\#}$ are evaluated.

**Keywords:** EDA-Sal, Cu(EDA–Sal), Hydrolysis, Isosbesticity, Thermodynamic Parameters

Organic compounds contain homo-polyfunctional sites are expected to exhibit difference in their characteristics when compared to their monofunctional analogues. Some of the typical characteristics observed in this context are magnetic, electronic, and electrochemical properties due to intramolecular interactions[1,2]. The well-known example is the case of isocheimal dicarboxylic acids, HOOC-R-COOH whose $pK_{a1}$ and $pK_{a2}$ values considerably differ based on the nature of the bridging moiety. The $pK_{a1}$ and $pK_{a2}$ values will almost converge to $pK_a$ of its mono functional equalling H-R-COOH, only when R is sufficiently longer. However, intramolecular interactions besides hydrogen bonding force $pK_{a1}$ and $pK_{a2}$ values to become considerably different when the value of R is small. The Schiff bases are one of the very important classes of compounds for organic as well as inorganic chemists in view of their salient features *viz*., potentiality as ligands[3-5] and vulnerability for hydrolysis at the existed azomethine sites[6,7]. A good amount of work has been published on the characterization and the kinetics of hydrolysis of Schiff bases having only one azomethine site[1-7,19]. The similar kind of studies on either Schiff bases or on their complexes containing two or more than two azomethine sites is very important field of interest in physical organic chemistry. As the reports on these contexts are not abundant, we in our laboratory made attempts to verify whether or how some of the physicochemical properties of homopolyfunctional organic compounds where the reactive sites are separated by some bridging organic moieties differ in their reactivity with respect to their corresponding monologues. In our previous attempts, we reported the kinetics studies of the hydrolysis of the Schiff base bridged with 1,3-methyl cyclohexane and its trinuclear cobalt (II) complex[8,9]. Though the protonated and deprotonated Schiff base moieties at different *p*H ranges found undergo near simultaneous hydrolysis, the coulombic repulsions between the protonated azomethine sites found to play key role in doubly protonated conditions of Schiff bases[8]. It was found in our reports that either protonated or deprotonated species present predominantly at different *p*H zones. The coulombic repulsions and other steric factors between protonated and deprotonated azomethine sites found to play key role in successive hydrolysis with varied speeds. The details of synthesis

and characterization of mononuclear copper (II) complex Cu(EDA-Sal) were reported earlier from this laboratory[10]. Here in, we report the results of kinetics of hydrolysis of two azomethine sites in 1,2-bis(salicylidinimino)ethylene (**1**) and its mononuclear copper (II) complex, Cu(EDA-Sal) (**2**). This work was initiated with the intension of investigating how the intra-molecular electronic interaction and coordination with the Cu metal ion influences the hydrolytic cleavage of the azomethine sites in the Schiff base molecules.

## Experimental Section

### Materials and Methods

The EDA-Sal was prepared by mixing methanolic solutions of ethylene diamine and salicylaldehyde in 1:2 molar ratios under warm conditions. The reaction mixtures on standing, resulted in the formation of solid which was filtered, dried, and recrystallized from methanol.

The synthesis of mononuclear copper (II) complex involved the mixing of hot acetone solutions of EDA-Sal and cupric acetate in 1:1 molar ratio. The reaction mixture immediately turned green. This solution gave dark green crystals upon standing over night. These crystals were filtered, washed with methanol, and air-dried. The complex was recrystallized from acetone.

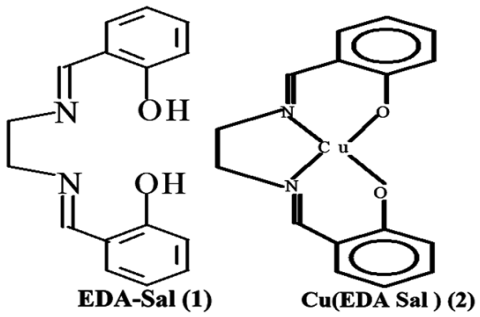
Buffers of different *p*Hs and other solutions were prepared by standard methods using AR grade reagents[11].

Orion Research Expandable Ion Analyser Model EA 940 was used for the measurement of *p*H values of the buffer solutions. A Shimadzu Model UV 160A spectrophotometer possessing the wavelength range of 200 – 1100 nm was used for spectral monitoring and for other photometric measurements with its double walled cuvette holders thermostatted by an INSREF model Cryostatic circulating liquid bath with temperature stability of ± 0.1°C.

Every time, the reaction mixture was prepared by quick mixing of 9 mL of aqueous buffer and DMF stock solution of either (**1**) or (**2**). An aliquot of these reaction mixtures was transferred immediately into the double walled cuvette and the absorbance of this solution was monitored while employing the "Photometric Mode "of the spectrophotometer at regular intervals. The studies were conducted after constant lapse of 20 seconds time from the quick mixing and then onwards at 1minute regular interval. Appropriate reagent solutions were used as reference solutions. For the entire period of study, the ionic strength was carefully adjusted by the addition of exact required amount of 1M KCl solution to the stock buffer solution

## Results and Discussion

The structure of EDA -Sal is shown in structure **1** and that of Cu(EDA - Sal) is shown in structure **2**. The purpose of undertaking the study of the hydrolysis of the Schiff base and its complex is on one hand to verify whether there is any influence of alteration of the electronic wave function for the coordinating azomethine site and on the other to understand whether the presence of metal ion has any catalytic effect on the rate of hydrolysis of complex when compared to its corresponding Schiff base.




**EDA-Sal (1)** **Cu(EDA Sal ) (2)**

As per the reports available, EDA-Sal forms very stable mononuclear copper complex which in turn can lead to the generation of a binuclear complexes also[12]. It is expected to have the formation of a protonated azomethine site and a quinonoid phenolic moiety involve in the mechanism of hydrolysis of the Schiff base site of EDA-Sal depending on the *p*H. However, when this Schiff base serve as the ligand to form complexes, no such possibility exists. Hence, it is presumed that the hydrolysis of the Schiff base site in the complexes might either entertain a different mechanism or the complex must slowly yield the ligand for hydrolysis under similar mechanism.

The hydrolyses of EDA-Sal were carried out in 90: 10 aqueous / D.M.F (v/v ) and aqueous / MeOH (v/v). The complex hydrolyses were done in 90: 10 aqueous/D.M.F ( v/v ). All the reactions followed first order either in EDA-Sal or in or in Cu(EDA - Sal).

### Acid-base equilibria of EDA-Sal

Since EDA -Sal contains two basic nitrogen atoms and two acidic phenolic groups, it is expected to involve in acid - base equilibria as shown in Scheme 1[13]. $LH_4^{+2}$, $LH_3^{+}$, $LH_2$, $LH^{-}$, $L^{-2}$ are doubly protonated EDA-Sal, monoprotonated EDA-Sal; EDA-

Sal, mono deprotonated EDA-Sal and doubly deprotonated EDA-Sal respectively. Whether the deprotonation of $LH_4^{+2}$ to $LH_2$ and $LH_2$ to $L^{-2}$ take place in successive steps or in single steps would be decided by the extent of intra-molecular electronic exchange interactions[8]. Molecular minimization studies suggested that the highest stability exists for $LH_4^{+2}$ structural conformation, whereas either one or several of $LH_3^+$, $LH_2$, $LH^-$ are observed as predominant species at other *p*H values. The conformational analysis of the molecule against a rotation around the ethylenic C — C bond is shown in Fig. 1.

Surprisingly, it was observed that the molecule prefers *syn* conformation and the *anti* form is slightly more energetic. The four acid - base equilibria constants of EDA-Sal were evaluated by plotting the graphs against log $\{(A_a-A_{ab}) / (A_{ab}- A_b)\}$ and *p*H around the inflection points of *p*H *vs* absorbance plot where $A_a$, $A_b$ and $A_{ab}$ are the absorbance values of the conjugate acid, base and the mixture of the acid and base form respectively. These data along with the other associated thermodynamic data are presented in Table 1. The $pK_{a1}$ and $pK_{a2}$ values of simple ethylene diamine are 7.18 and 9.96[11]. As it is observed from Table 1, $pK_{a1}$ and $pK_{a2}$ of EDA-Sal values are small when compared to those of simple ethylene diamine and we propose that this is due to implication of greater conjugate acidity to $LH_4^{+2}$ and $LH_3^+$ species. Further, as Table 1 reveals, the $pK_{a1}$ and $pK_{a2}$ of EDA-Sal are close and we propose that there is no such possibility between the protonated azomethine groups through the bridging moiety as it keeps the two protonated salicylaldimine terminals apart due to repulsion. However, considerably large difference existed between $pK_{a3}$ and $pK_{a4}$ due to the availability of interaction through intramolecular hydrogen bonding in $LH_2$ species.

$$LH_4^{+2} \rightleftharpoons LH_3^+ + H^+ \quad (K'_{a1})$$

$$LH_3^+ \rightleftharpoons LH_2 + H^+ \quad (K'_{a2})$$

$$LH_2 \rightleftharpoons LH^- + H^+ \quad (K'_{a3})$$

$$LH^- \rightleftharpoons L^{-2} + H^+ \quad (K'_{a4})$$

Scheme 1 — Acid-Base equilibria of EDA-Sal

## Kinetics of Hydrolysis of EDA-Sal

To monitor the kinetics of hydrolysis of EDA-Sal, we followed the change in absorbance with time. Appropriate wavelengths were chosen, where considerable variation in absorbance was observed. Incidentally, in all the buffers of study, 275 nm was found to be unique wavelength where a steady fall was

Table 1 — Acid-base thermodynamic data of EDA-Sal at 25°C

| $pK_a$ | $\Delta G°$ ( = 2.303 RT $pK_a$) * |
|---|---|
| 4.36 | 24.89 |
| 5.54 | 31.62 |
| 7.94 | 45.28 |
| 10.30 | 58.78 |

Protonation constant $K_1 = 1 / K_{a1}K_{a2} = 8\times10^9$ liter$^2$ mol$^{-2}$
Deprotonation constant $K_2 = K_{a3}K_{a4} = 5.79\times10^{-19}$ mol$^2$ liter$^{-2}$
Standard free energy of protonation = $-RT \ln K_1 = -56.52$
Standard free energy of deprotonation = $-RT \ln K_2 = 104.06$
* in kJ mol$^{-1}$

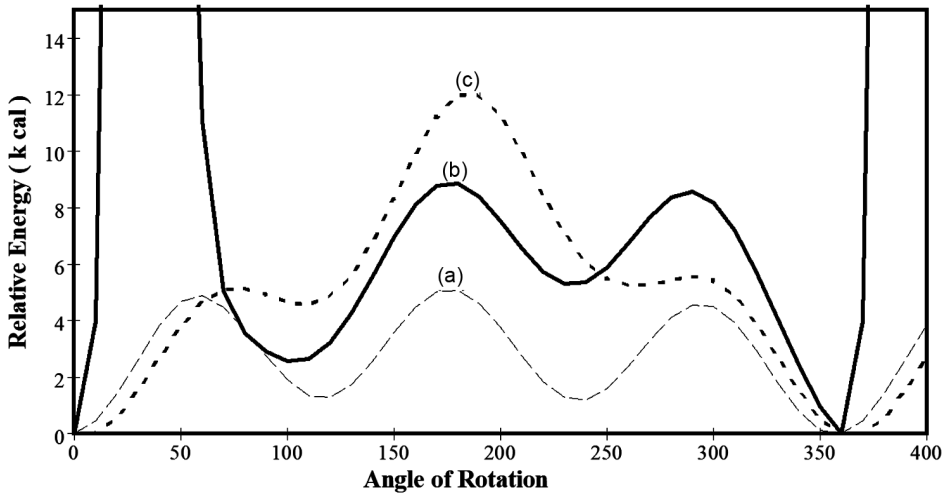



Fig. 1 — Rotational conformational analysis of EDA-Sal around C — C bond of the stablest conformer of; (a) $LH_2$, (b) $LH_3^+$ and (c) $LH_4^{+2}$

observed. Further, the repetitive spectral profiles of EDA-Sal in all the buffers studied shown isosbestic points, indicating the presence of only two absorbing species throughout the course of the reaction[13,14]. As isobesticity is observed in the entire zone of reaction studies, a uniform mechanism suggested throughout the course of the reaction. For the initial concentration of EDA-Sal, the infinite spectrum is found to resemble the spectrum of solution of double the concentration of salicylaldehyde under similar conditions. Moreover, an equimolar solution of ethylene diamine does not absorb in the wavelength range of study. It is also observed that the infinite spectrum of the reaction mixture also passes through the same isosbestic point, and hence it is confirmed our assumption that throughout the course of the reaction, EDA-Sal and Sal are the only two absorbing species[8]. So, the kinetic reaction for the hydrolysis of EDA-Sal is suggested as represented by the following equation.

$$\text{EDA-Sal} + 2\,H_2O \xrightarrow{k_o} \text{Ethylene Diamine} + 2\,\text{Sal} \quad (1)$$

Where $k_o$ is the overall rate constant.

The above stoichiometry is confirmed by comparing the concentration profiles of EDA-Sal and Sal calculated with the aid of the molar extinction coefficients of pure species and Beer-Lambert's Law with time as exemplified in the Fig. 2.

As evident from Fig. 2, the equality [EDA-Sal] + 0.5 [ sal ] = $C_o$ is essentially holding good during the reaction path where EDA-Sal]$_t$ and [ sal]$_t$ are the respective concentrations of EDA-Sal and Sal at time t and $C_0$ is that of EDA-Sal when t = 0. At all those wavelengths, λ, where

$$\varepsilon\,\lambda\text{EDA-Sal} = 2\,\varepsilon\lambda\text{sal} \quad (2)$$

isosbestic points are expected under these stoichiometric conditions as ethylenediamine does not absorb in this wavelength region.

The rate equation applicable to Eqn. 1 under pseudo- order conditions can be expressed as

$$d\,[\,\text{EDA-Sal}\,]/dt = K\,[\,\text{EDA-Sal}\,]_t^{x} \quad (3)$$

$$K = k_o\,[\,H_2O\,]_t^{y}\,[\,H^+\,]_t^{z} \quad (4)$$

**Effect of *p*H on Hydrolysis**

The [$H^+$] entry in Eqn. (4) is provided to accommodate the effect of *p*H, if any on the reaction rate. Since [$H_2O$] is far greater than [EDA-Sal] and the reaction mixture is buffered, approximating $k_o[H_2O\,]_t^{y}\,[\,H^+]_t^{z}$ to the pseudo-rate constant, K, through Eqn. (4) is justified. The pseudo-first order rate constants obtained through pseudo-first order plots of EDA-Sal and that of Cu(EDA-Sal) are presented in Table 2. The general effect of *p*H on the pseudo-first order rate constant is shown in Fig. 3.

From the data in Table 2 and Fig. 3, it is found that the general dependence of hydrolyses of rate constants on the *p*H is on similar lines to our earlier reports[8]. At highly acidic *p*H's, the log k is very low and is independent of *p*H. It then increases with *p*H till *p*H 4. Further increase in *p*H initially reduces rate which is then nearly independent of *p*H. Again, the rate increases almost linearly with *p*H.

From the literature, it is clear that the Schiff base undergo hydrolysis in the acidic range due to the development of considerable carbonium ion in the conjugate acid through resonance as shown below[13,16]

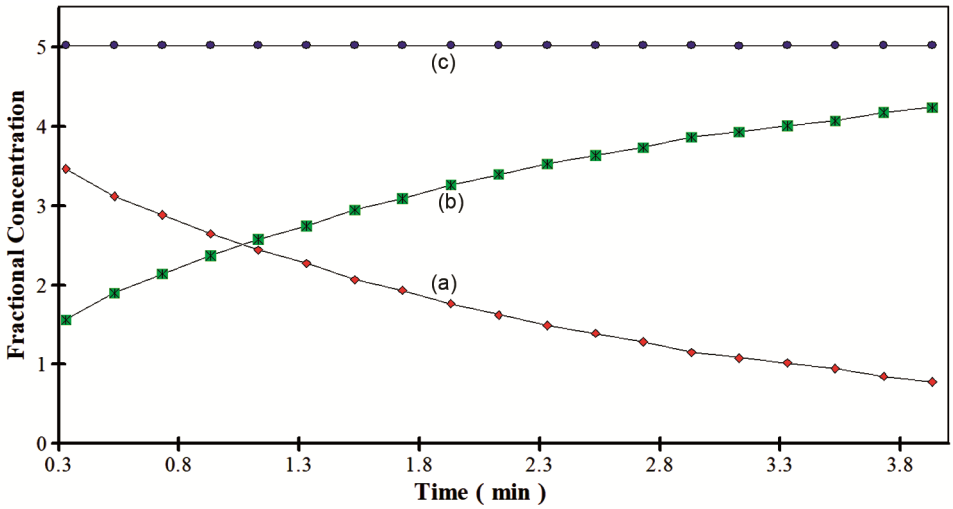



Fig. 2 — Multicomponent analysis profiles hydrolysis at 30°C: (a) [EDA-Sal], (b) [Sal] /2, (c) combined concentration, {[ EDA-Sal]$_t$ + 0.5 [Sal]$_t$

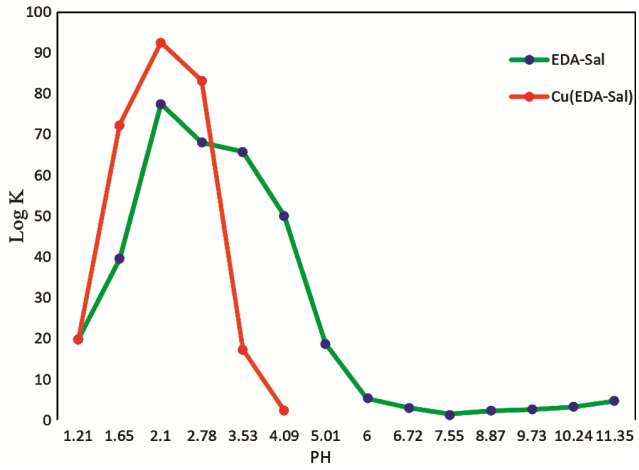
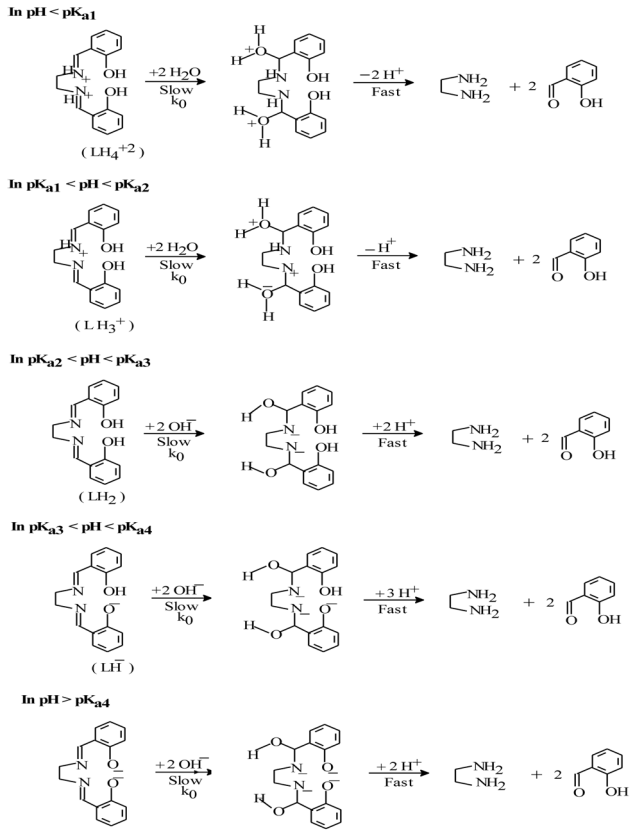



Fig. 3 — The Effect of *p*H on the pseudo-first order rate constants




Scheme 2 — Mechanism of hydrolysis of EDA-Sal

$$- CH = NH- \leftrightarrow - \overset{+}{C}H - NH -$$

This carbonium ion site is proposed to undergo slow nucleophilic attack by water molecules. At highly acidic *p*H's the rate is usually independent of *p*H due to Schiff base essentially exists in the conjugative acid form. As the *p*H increases, the reaction rate will decrease due to the decrease in the concentration of conjugate acid and hence that of the carbonium ion Schiff base. With further increase in *p*H, the reaction rate slowly increases with a different mechanism. In neutral *p*H range, the Schiff base exists in neutral status and is more resistant to nucleophilic attack by solvent molecules. In neutral and basic *p*H's, the $OH^-$, which is better nucleophile determines the rate of reaction rather than $H_2O$ through a different mechanism involving nucleophilic attack of $OH^-$. In these lines, possible mechanism for the hydrolysis in different *p*H ranges is suggested in Scheme 2.

Table 2 — Acid-base thermodynamic data of EDA-Sal at 25°C

| *p*H | Pseudo- first order rate constant at 30°C $K\times10^3$ $s^{-1}$ | |
|---|---|---|
| | EDA-Sal | Cu EDA-Sal |
| 0.97 | 22.30 | 19.80 |
| 1.26 | 28.10 | 72.42 |
| 2.53 | 41.66 | 92.50 |
| 3.14 | 48.31 | 83.15 |
| 3.99 | 59.82 | 17.37 |
| 4.95 | 29.30 | 02.25 |
| 5.42 | 16.00 | – |
| 5.65 | 12.00 | – |
| 7.27 | 06.10 | – |
| 8.10 | 04.57 | – |
| 9.05 | 04.16 | – |
| 9.96 | 03.94 | – |
| 10.40 | 05.10 | – |
| 11.52 | 06.74 | – |

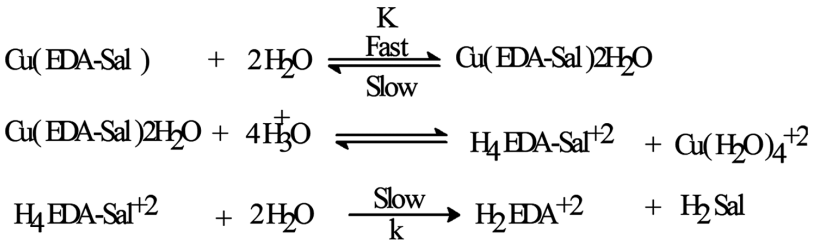
Table 3 — Thermodynamic data [p] of the hydrolysis of EDA-Sal

| *p*H | $E_a$ | $\Delta H^{\#}$ | $-\Delta S^{\#}$ [q] | $\Delta G^{\#}$ |
|---|---|---|---|---|
| 0.97 | 11.63 | 9.11 | 0.21 | 73.60 |
| 3.99 | 7.75 | 5.30 | 0.22 | 7174 |
| 7.27 | 18.50 | 16.00 | 0.20 | 76.84 |
| 11.52 | 15.82 | 13.30 | 0.21 | 76.60 |

[p] kJ $mol^{-1}$ , [q] kJ $mol^{-1}$ $K^{-1}$

## Effect of Temperature on Hydrolysis

The influence of temperature on the reaction rates of hydrolyses of EDA-Sal have also been investigated. From the Arrhenius linear plots between log k and 1/T, the activation energy values, $E_a$, were calculated[17]. Eyring Equation has been used for the evaluation of other thermodynamic parameters. All the evaluated values are furnished in Table 3. As data reveals, the $\Delta S^{\#}$ values are so close to each other in all the buffers of study supports our assumption of existence of short-lived transition state with almost similar structure in the entire zone of study as shown in Scheme 2 [18,20].




Scheme 3 — Mechanism of hydrolysis of Cu(EDA-Sal

**Hydrolysis of Cu(EDA - Sal)**

In order to compare the rates of hydrolysis pattern of Cu(EDA-Sal) with that of EDA - Sal, the hydrolyses reactions of Cu(EDA-Sal) have also been done under the similar conditions as conducted for (**1**). From the repetitive spectral profiles and other photometric measurements of the complex, it is found that the complex undergoes hydrolysis only in acidic medium. In neutral and basic media. the complex remains stable. The most possible expected reason for the stability of complex in the neutral and basic media regions is in Schiff base complex, the imine nitrogen atoms and phenolic oxygen atoms are linked to copper metal and hence the azomethine carbon is expected to carry high positive charge relative to the corresponding pure Schiff base (**1**). This results in a higher nucleophilic attack by water molecule on this carbon atom. So, in highly acidic *p*H's the rates of hydrolysis reactions of Cu(EDA-Sal) are a little bit more than that of EDA-Sal. However, in all the buffers where studies were possible, isosbestic points are observed in the repetitive spectral runs suggesting the presence of only two absorbing components in the reaction mixture as in the case of (**1**). Careful observation of the spectra recorded at infinite time for both the ligand EDA-Sal and its copper complex Cu(EDA-Sal) shows similarity in profiles. This feature indicates near identical final products from the ligand frame work of the complex suggesting that the metal coordination might not have altered the reaction mechanism of hydrolysis. However, the initial spectral profiles of the complex and the ligand are obviously different.

In highly acidic range, the ligand's initial $\lambda_{max}$ is at 350 nm and its $\lambda_{max}$ at infinite time is around 320 nm whereas, the complex initial $\lambda_{max}$ is at 330 nm with a shoulder at 360 nm. This means that the complex is not immediately offering the ligand for subsequent hydrolysis and that it is able to retain its coordinated structure. Hence, the attack of water molecule to the azomethine site is on the complex and not after the complex jettisoned the ligand. Accordingly, we propose that the water molecule is first coordinating to the copper centre and then the aquo-complex is undergoing hydrolysis[17]. In these lines, mechanism is suggested for the hydrolysis of Cu(EDA-Sal) and is shown in Scheme 3. The pseudo rate constants obtained for the hydrolysis of Cu(EDA-Sal) furnished along with that of EDA-Sal in Table 2.

CuEDA-Sal.2$H_2O$ in Scheme 3 is related to the hydration constant, K. Since the concentration of the substrate, $H_4$EDA-Sal$^{+2}$, itself is indirectly governed by [$H^+$], the pseudo rate constant of hydrolysis is expected to be higher at lower *p*H's than that expected for the pure ligand. However, at *p*H > 3.5, the trend reverses as shown in Fig. 3.

**Possible governing factors of hydrolysis mechanism in Cu(EDA-Sal)**

The mechanism as we suggested in Scheme 3 for the hydrolysis of the Copper (II) complex is expected to govern by two opposing effects as explained under as A and B.

**A. Effects Opposing Hydrolysis**

(a) The complexation stabilizes the ligand by impoverishing the nitrogen for protonation.
(b) The nascent ligand frame work peeled off from the metal complexes in acid media is in *syn* arrangement with respect to the salicylidine groups. This allows intramolecular hydrogen bonding across the two salicylidine groups making the nitrogen less basic for proton attack. The pure ligand, however, exists in *anti*-conformation.

**B. Effects Favoring Hydrolysis**

(a) The complexation brings in labialization effect making the azomethine site vulnerable for enhanced nucleophilic attack.
(b) The metal center with a proven square planar geometry can coordinate to water molecules to result in an octahedral geometry. These water molecules are readily available to take part in the hydrolysis.

These two opposing forces weigh differently with *p*H alteration. At *p*H < 3.5, effect (B) dominates and at *p*H > 3.5, (A) does. While at *p*H = 3.5, the two effects balance each other as evident from Fig. 3 the effect of *p*H on EDA-Sal and Cu(EDA-Sal).

## Conclusions

The present investigation involved the verification of variation in the pathways of kinetics of hydrolysis of isochemical azomethine sites present in a Schiff base, 1,2-bis(salicylidinimino)ethelene, abbreviated as EDA -Sal and one of its novel mononuclear copper (II) complex abbreviated as Cu(EDA - Sal).

The present studies conducted in the varied *p*H and temperature ranges to check their influence. The investigations indicated that the Schiff base hydrolysis highly influenced by these parameters and exists in several acid - base forms.

The acid-base equilibrium and resonace effects have had significant hydrlytic cleavage of azomethine sites present in the Schiff base and its complex. Presence of isosbestic points evidenced that only two absorbing species in the entire reaction path. The hydrolysis follows pseudo - first order kinetics in both Schiff base and its corresponding complex.

Except in acidic range, the complex was found stable. Typical reasons for the variation in rate of hydrolysis of both the compounds were illucidated based on several influencing factors and suitable mechanisms for the hydrolysis was suggested. Psuedo first order rate constants, activation energy $E_a$ and other thermodynamic parameters such as $\Delta H^{\#}$, $\Delta G^{\#}$ and $\Delta S^{\#}$ were evaluated.

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