

Synthesis and Spectral Properties of Novel Azo Dyes and Evaluation of Corrosion Inhibitors for Dissolution of C-Steel in Hydrochloric Acid Solution

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ABSTRACT

This research includes the synthesis new azo dyes compounds derived from (6R;7R)-7-[2-(2-Amino-4-thiazolyl)glyoxylaido]-8-oxo-3{[(1,2,5,6,-tetrahydro-2-methyl-5,6-dioxo-as-triazin-3-yl)-thio]methyl}-5-thia-1-azabicyclo [4.2.0] octa-2-ene-2-carboxylic acid, 7²(z)-(o-methyloxime),disodium salt with each one of the following phenols and naphthols such as:

2-Naphthol, 8-Hydroxy quinoline, 4-Chloro phenol, Resorsinol.The study involved solvent and pH effects on the azo dyes behavior by electronic absorption spectra, molar conductance and evaluated them as corrosion inhibitors for carbon steel in acid media (0.5N HCl).Furthermore study their by elemental analysis (CHN) and IR spectroscopy.

Keywords : azo dyes , acide base properties , corrosion inhibitors .

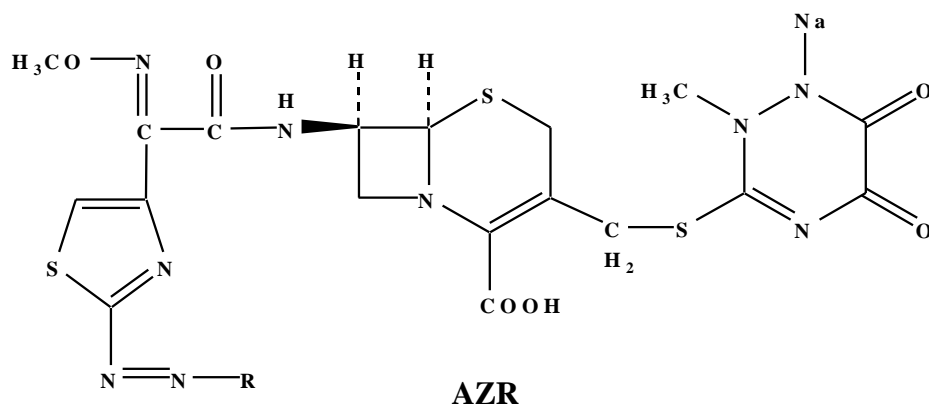
INTRODUCTION

The synthesis of azo dyes is very well established and each year new azo dyes are being developed [1] and are a very important class of chemical compounds receiving attention in scientific research. They are highly colored and have been used as dyes and pigments for a long time[2,3]. Furthermore, they have been studied widely because of their excellent thermal and optical properties in applications such as optical recording medium[4], toner[5], ink-jet printing[6], and oil-soluble lightfast dyes[7]. In the field of azo dyes, phenols and naphthols play major roles for most of the commercial dyes. Most of the dyes are marketed in the form of azo disperse, azo-vat, azo-acid dyes, *etc.* All have the naphthols moiety bearing hydroxyl group(OH) as an auxochrome group[8].Heterocycles are extensively used in disperse dye chemistry either for textile uses.Non-textil applications of heterocyclic dyes include their use in reprography, functional dye and non-linear optical systems, photodynamic therapy and leasers. Azo dyes containing heterocyclic rings lead to brighter and often deeper shades than other azo dyes. Heterocyclic dyes may be good candidates for printing and photographig-like applications on account of their brightness and tinctorial strength[9].

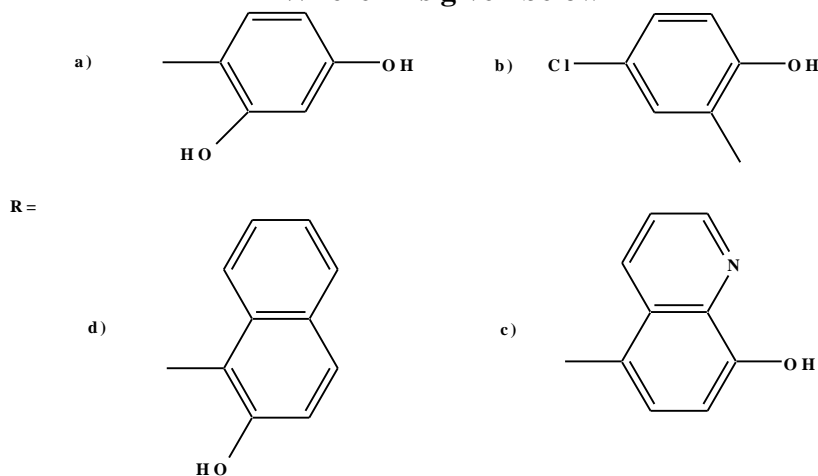
Corrosion problems have received considerable amount of attention because of their economic and safety consequences .the use of inhibitors is one of most practical methods for protection against corrosion[10,11]. A survey of literature

reveals that the applicability of organic compounds as corrosion inhibitors for mild steel in acidic media has been recognized for a long time. A large number of organic compounds, particularly those containing nitrogen, oxygen or sulphur in a conjugated system, are known to be applied as inhibitors to control acid corrosion of iron and steel. Carbon steel is used as essential part in the manufacturing of installations used in petroleum and other industries. It has been observed that the most of the organic inhibitors act by adsorption on the metal surface[12-15]. The adsorption of corrosion inhibitor depends mainly on physico-chemical properties of the molecules such as functional group, steric factor, molecular size, molecular weight, molecular structure, aromaticity, electron density at the donor atoms and π -orbital character of donating electrons and also, on the electronic structure of molecules[16-21].

In this study , we report some novel azo dyes compounds derived from (6R;7R)-7-[2-(2-Amino-4-thiazolyl)glyoxylaido]-8-oxo-3{[(1,2,5,6,-tetrahydro-2-methyl-5,6-dioxo-as-triazin-3-yl)-thio]methyl}-5-thia-1-azabicyclo[4.2.0]octa-2-ene-2-carboxylic acid, 7²(z)-(o-methyloxime),disodium salt with phenols and naphthols in order to study the spectral proprties and evalution of corrosion inhibitors for dissolution of carbon steel in hydrochloric acid Solution. These compounds have general structure AZR :



Where R is given below



compound	AZP ₁	AZP ₂	AZN ₁	AZN ₂
R	a	b	c	d

EXPERIMENTAL

(i) Synthesis and materials

All the compounds amine, substituted phenols and naphthols, boric acid, phosphoric acid, acetic acid, hydrochloric acid, sodium hydroxide and solvents were commercial products of analar grade and were used without further purification. The preparation of azo dyes compounds was carried out according to Schem 1.

Diazotization of amine

A finely ground powder of the (10 mmol) amine dissolved in water was cooled to (0-5°C) and diazotized with 1M sodium nitrite solution (10 ml) at 0°C in 3M HCl (8 ml). The rate of addition was adjusted so that the temperature of the solutions remains below 10 °C. The solution was kept in an ice bath and used immediately in the next step [22].

Coupling with phenols

A solution of one of the substituted phenols or naphthols (10 mmol) in 1M NaOH (20 ml) was prepared and cooled in an ice bath. The diazonium salt from step 1 was then added slowly with stirring to the phenol or naphthol solution. The reaction mixture was then left standing in the ice bath for at least 15 minutes until the crystallization is complete (giving a colored solid). The pH of the solution was adjusted with dilute HCl or NaOH solutions (0.1M) in order to induce precipitation. The dark red azo dye was then collected and washed with cold water recrystallized from methanol yielded the pure azo dyes the yield was 64 – 85% [22].

(ii) Studies

(A) Solvent effect study

Stock solution (5.0×10^{-4} M) of the azo compounds was prepared by dissolving an accurately weighted amount of the compound in the required volume of methanol. More dilute solutions were obtained by accurate dilution with the appropriate solvent with concentration (5.0×10^{-5} M) and the absorptions were recorded within the wavelength range from 340 to 700 nm[23].

(B) pH effect study

The modified aqueous universal buffer series (pH 2.0-12.0) were prepared according to the method of Britton-Robinson by mixing (0.04M) boric acid, (0.04M) phosphoric acid and (0.04M) glacial acetic acid and adjusting the pH to required value with (0.2M) NaOH[24]. Stock solution (5.0×10^{-4} M) of the azo compounds was prepared by dissolving an accurately weighted amount of the compound in the required volume of methanol. More dilute solutions were obtained by accurate dilution with the appropriate buffer solution with concentration (5.0×10^{-5} M) and the absorptions were recorded within the wavelength range of 340 to 700 nm[23].

(C) Molar conductance study

Conductance measurements were determined (5.0×10^{-4} M) methanol at room temperature ($\sim 25^\circ\text{C}$) and the values are given in Table 1 which indicate that the azo dyes are electrolytes.

(D) Inhibitor Corrosion study

Inhibitor Corrosion study were carried out by weight the specimens (C-Steel) before and after immersion in 100 ml acid solution (0.5 M HCl) for (3 days) at room temperature in the absence and presence of inhibitors compounds[25].

(iii) Techniques

Elemental analysis were performed with a Euro vector EA 3000A / Italy. IR spectra were obtained using a FT-IR-8400s. Fourier Transform Infrared spectrophotometer Shimadzu Japan. U.V spectra were recorded on a PD-303 UV spectrophotometer – APEL. The pH values were checked using a Wissenschaftlich-Technische- Werk Statten model IA10-1112 pH meter accurate ± 0.02 pH units. Conductance were measured by BC3020 Benchtop Conductivity meter.

RESULTS AND DISCUSSION

(i) Characterization

The synthesized compounds were characterized by IR , UV and Elemental analysis.

The IR spectra of the prepared azo compounds were recorded as KBr disk and the spectral data of the products were gathered in Figure 1 and Table 2:

The azo dyes exhibited intense IR absorptions at $3340.48 - 3409.91\text{ cm}^{-1}$ [$\nu(\text{O-H})$] , $2935.64 - 2939.31\text{ cm}^{-1}$ [$\nu(\text{C-H aliphatic})$] , $1768.60 - 1772.64\text{ cm}^{-1}$ [$\nu(\text{C=O})$] , $1625.88 - 1668.31\text{ cm}^{-1}$ [$\nu(\text{C=O amide})$] , $1508.23 - 1633.59\text{ cm}^{-1}$ [$\nu(\text{C=C})$] , $1346.22 - 1407.94\text{ cm}^{-1}$ [$\nu(\text{N=N-})$] , $1037.63 - 1039.56\text{ cm}^{-1}$ [$\nu(\text{C-O})$].

(ii) Solvent effect on the absorption spectra of azo dyes

The absorption spectra of azo dyes have been studied in various organic solvents of different polarities such as, Dioxane, Chloroform, Methanol, Tetrahydrofuran, Dimethylformamide, Dimethyl sulfoxide and the Table 3 summarized the values of λ_{max} and ϵ_{max} of the characteristic absorption band have been displayed.

Spectra of the azo dyes comprise mainly one band in the visible region 425-520 nm is an intense one and much affected by molecular structure of azo dyes. Thus, it can be assigned to $\pi-\pi^*$ transition involving π electronic system of the azo dyes[26].

The dyes were found to exhibit a strong solvent dependence; this dependence shows more or less regular variation with the polarity of the solvents used. The visible bands suffer bathochromic shifts as the polarity of the solvent was increased, a behaviour which is characteristic of such local electronic transitions Figure 2[23].

The most interesting effects of changing the solvents polarities are observed with the main longer broad band. In general, this band exhibits an appreciable red shift in its λ_{max} on changing the solvent from MeOH to DMF or DMSO. This behavior indicates that the excited state of these compounds is more polar than their ground state and thus, this red shift can be ascribed to stabilization of the polar excited state as the polarity of the solvent increased, i.e., lower excitation energy is required in DMSO or DMF relative to MeOH[26,27].

(iii)pH effect on the absorption spectra of azo dyes

The absorptions spectra of the azo dyes were recorded within various buffer solutions range (2-12) and wavelength range (340-700)nm. Figure 3 shows spectra curves of azo dyes in different buffer solutions and Table 4 shows these absorptions bands and their values.

It is important to indicate that the coloured of azo dyes prepared changes shape on changing the medium from red colour in acidic media to violet in alkaline media, this means that these azo dyes can be used as an acid-base indicator. A detailed study will be performed in a separate article[23].

The electronic spectra of the studied azo dyes in the pH range (2-6) show mainly one band in the visible region (425-505) nm, representing the absorptions of the protonated form of these azo dyes. Increasing the pH values of the medium results in increasing its intensity or bathochromic shift of this band to azo dyes. In solutions of pH ranging from 8 to 12 the visible spectra of the azo dyes comprise bathochromic shift from (425-505) nm to (495-555) nm and new small band will be appearance in shorter wavelength in 380 nm in pH 12 due to the azo dye AZP₂ and a new band due to azo dye AZN₂ in range (390-400) nm in the pH range (8-12) with increasing in intensity[26].

The changing which happened in the results in absorption spectra to azo dyes in the pH range (8-12) from red shift to all azo dyes and appear new band in azo dyes AZP₂ and AZN₂ can be indicated that as the pH increasing, de-protonation occurs and the ionization of the dyes produces possible resonance forms[28].

(iii) Weight loss study [29-32]

Samples of carbon steel were used for weight loss measurement studies at (25°C) for three days. Figure 4 and 5 show the variation of weight loss of inhibitors amine and azo dyes.

The Corrosion rate of all inhibitors were calculated by the following equation 1:-

$$\text{CR} = W \cdot K / A \cdot d \cdot t \dots\dots\dots (1)$$

Where CR: Corrosion rate, W: amount of weight loss (gm.), K : constant (534),
A : area of carbon steel (inch²), d: density of sample (7.86), t: time (hrs).

Figure 4 and 6 show the variation of Corrosion rate of amine and azo dyes (inhibitors).

The Inhibitors Efficiency (IE%) and surface coverage (θ) of all inhibitors amine and azo dyes were calculated by the following equations (2,3):-

$$IE(\%) = (CR_0 - CR_1 / CR_0) \times 100 \dots\dots\dots (2)$$

$$(\theta) = CR_0 - CR_1 / CR_0 \dots\dots\dots (3)$$

Where CR_0 = Corrosion rate with out inhibitors (blank).

CR_1 = Corrosion rate with inhibitors.

IE% = Inhibitors Efficiency.

(θ) = surface coverage.

Figure 4 and 7 show the variation of Inhibitors Efficiency of of amine and azo dyes (inhibitors) .

values of weight loss ,corrosion rate ,inhibitor efficiency and surface coverage (θ) for amine and azo dyes are give in Table 5 .

The results indicate that weight of carbon steel sample was decreased in comparison with absence of amine and azo dyes (inhibitors). Also the results indicate that azo dyes provide efficiency inhibition corroison more than amine, although the availability of the the lone pair of electrons on nitrogen and the protonation property of aza and thiol groups are responsible for the formation of protective on the metal surface and hence control the corrosion because these azo dyes were introduced prevented layer on the metal surface as a result as presence azo group (-N=N-) and aromatic cycles in the azo dyes [33] .

Table 1 : physical properties, molar conductance and elemental analysis of the Azo dyes

Azo dyes	Molecular Formula	M.W (g/mol)	Colour	M.P. °C	Yield %	Elemental Analysis found (Calc.)%			Molar Conductance $\mu\text{s.cm}^{-1}$
						C	H	N	
Amine	C₁₈H₁₆N₈O₇S₃Na₂	598.549	Pale yellow	–	–	–	–	–	60.2
AZP₁	C₂₄H₂₀N₉O₉S₃Na	697.663	red	> 250	65	41.02 (41.31)	2.68 (2.88)	17.91 (18.06)	74.8
AZP₂	C₂₄H₁₉N₉O₈S₃ClNa	716.109	dark red	> 250	85	40.04 (40.25)	2.49 (2.67)	17.47 (17.60)	65.4
AZN₁	C₂₈H₂₂N₉O₈S₃Na	731.724	dark red	> 250	64	45.66 (45.96)	2.87 (3.03)	17.09 (17.22)	44.4
AZN₂	C₂₇H₂₁N₁₀O₈S₃Na	732.71	dark red	> 250	70	44.06 (44.26)	2.71 (2.88)	18.98 (19.11)	40.4

Table 2 : Major IR absorption bands (cm⁻¹) of the Azo dyes

Azo dyes	V(O-H) cm ⁻¹	V(C-H) _{aliphatic} cm ⁻¹	V(C=O) cm ⁻¹	V(C=O) _{amide} cm ⁻¹	V(C=C) cm ⁻¹	V(-N=N-) cm ⁻¹	V(C-O) cm ⁻¹
AZP₁	3340.48	2939.31	1772.46	1643.24	1633.95	1406.01	1039.56
AZP₂	3407.98	2939.1	1770.53	1668.31	1508.23	1407.94	1039.56
AZN₁	3352.05	2935.46	1768.60	1629.74	1519.8	1396.37	1039.56
AZN₂	3409.91	2935.46	1772.64	1625.88	1510.16	1346.22	1037.63

**Table 3 : Electronic spectral characteristics of the azo dyes
in different pure organic solvents**

Azo dyes	Dioxane $\lambda_{\max}(\text{nm}), [\epsilon_{\max}]$ (L.cm ² .mol ⁻¹)	Chloroform $\lambda_{\max}(\text{nm}), [\epsilon_{\max}]$ (L.cm ² .mol ⁻¹)	THF $\lambda_{\max}(\text{nm}), [\epsilon_{\max}]$ (L.cm ² .mol ⁻¹)	Methanol $\lambda_{\max}(\text{nm}), [\epsilon_{\max}]$ (L.cm ² .mol ⁻¹)	DMSO $\lambda_{\max}(\text{nm}), [\epsilon_{\max}]$ (L.cm ² .mol ⁻¹)	DMF $\lambda_{\max}(\text{nm}), [\epsilon_{\max}]$ (L.cm ² .mol ⁻¹)
AZP ₁	425 [4900]	465 [4460]	465 [4540]	467 [5300]	490 [4720]	495 [5520]
AZP ₂	495 [5420]	500 [5800]	500 [5700]	500 [6960]	518 [8500]	510 [3920]
AZN ₁	495 [9340]	500 [9540]	500 [9480]	500 [9400]	510 [9900]	520 [7860]
AZN ₂	485 [8760]	488 [9320]	490 [8880]	500 [6460]	518 [13200]	520 [11000]

**Table 3 : Electronic spectral characteristics of the azo dyes
in different universal buffer solutions**

Azo dyes	pH2 $\lambda_{\max}(\text{nm}),$ Abs(cm ⁻¹)	pH4 $\lambda_{\max}(\text{nm}),$ Abs(cm ⁻¹)	pH6 $\lambda_{\max}(\text{nm}),$ Abs(cm ⁻¹)	pH8 $\lambda_{\max}(\text{nm}),$ Abs(cm ⁻¹)	pH10 $\lambda_{\max}(\text{nm}),$ Abs(cm ⁻¹)	pH12 $\lambda_{\max}(\text{nm}),$ Abs(cm ⁻¹)
AZP ₁	465 (0.218)	465 (0.214)	485 (0.23)	495 (0.294)	510 (0.307)	525 (0.341)
AZP ₂	500 (0.236)	500 (0.299)	505 (0.306)	505 (0.332)	512 (0.307)	380 (0.114) 542 (0.173)
AZN ₁	505 (0.377)	500 (0.411)	500 (0.437)	500 (0.456)	515 (0.439)	550 (0.435)
AZN ₂	490 (0.452)	505 (0.51)	513 (0.596)	400 (0.581) 528 (0.749)	400 (0.704) 535 (0.713)	390 (0.749) 555 (0.753)

Table 4 : Shows values of weight loss ,corrosion rate ,inhibitor efficiency and surface area (θ) of the amine and azo dyes

day Compounds	Day1				Day2				Day3			
	Wt	CR	IE%	θ	wt	CR	IE%	θ	wt	CR	IE%	θ
HCl	0.086	90.07 1	/	/	0.1310	91.44 1	/	/	0.164 0	76.00 .	/	/
amine	0.0182	25.408	73.27	0.7327	0.0308	21.499	76.48	0.7648	0.0645	30.015	60.79	0.6079
AZP ₁	0.001 7	2.373	97.0. 3	0.970 .	0.004	3.071	96.6 4	0.966 4	0.007 0	3.490	90.4 4	0.904 4
AZP ₂	0.0039	5.444	94.27	0.9427	0.0098	6.840	92.51	0.9251	0.0176	8.190	89.30	0.8930
AZN ₁	0.0045	6.282	98.94	0.9894	0.0087	6.770	92.59	0.9259	0.0151	7.026	90.82	0.9082
AZN ₂	0.0022	3.071	96.76	0.9676	0.0053	3.699	95.95	0.9595	0.0095	4.420	94.22	0.9422

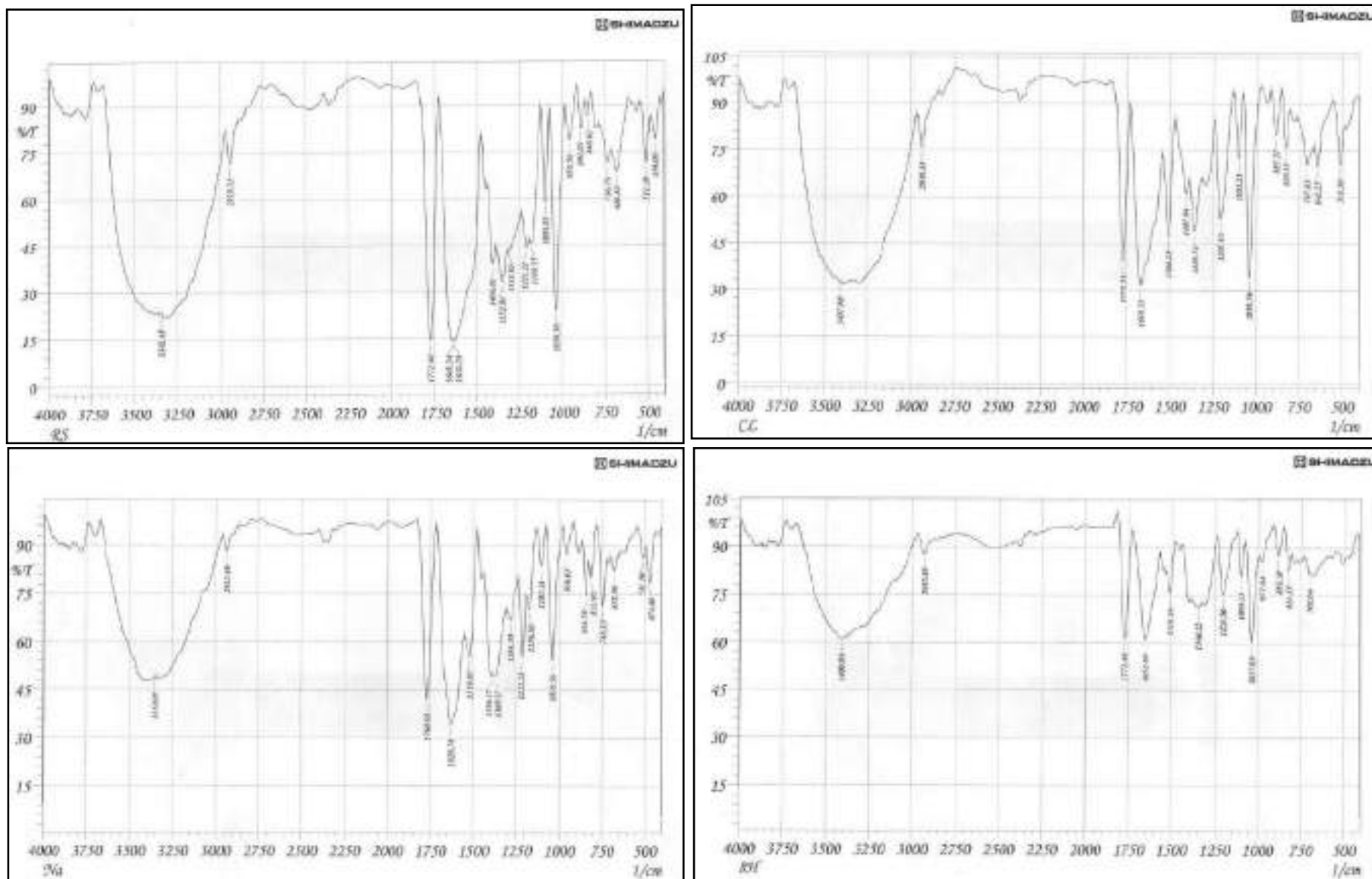


Figure 1 : The IR spectra of azo dyes

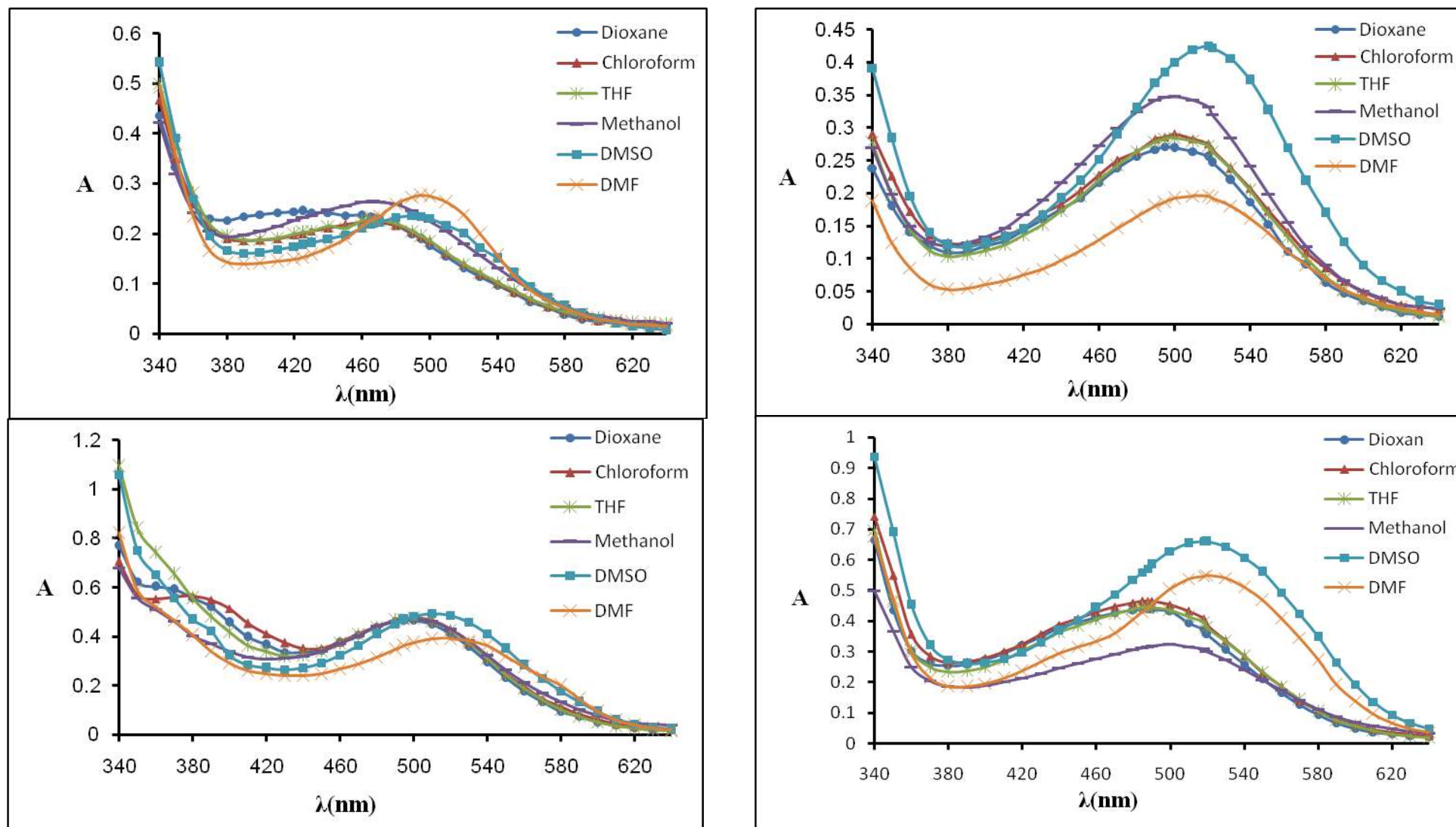


Figure 2 : Absorption spectra of azo dyes in various organic solvents

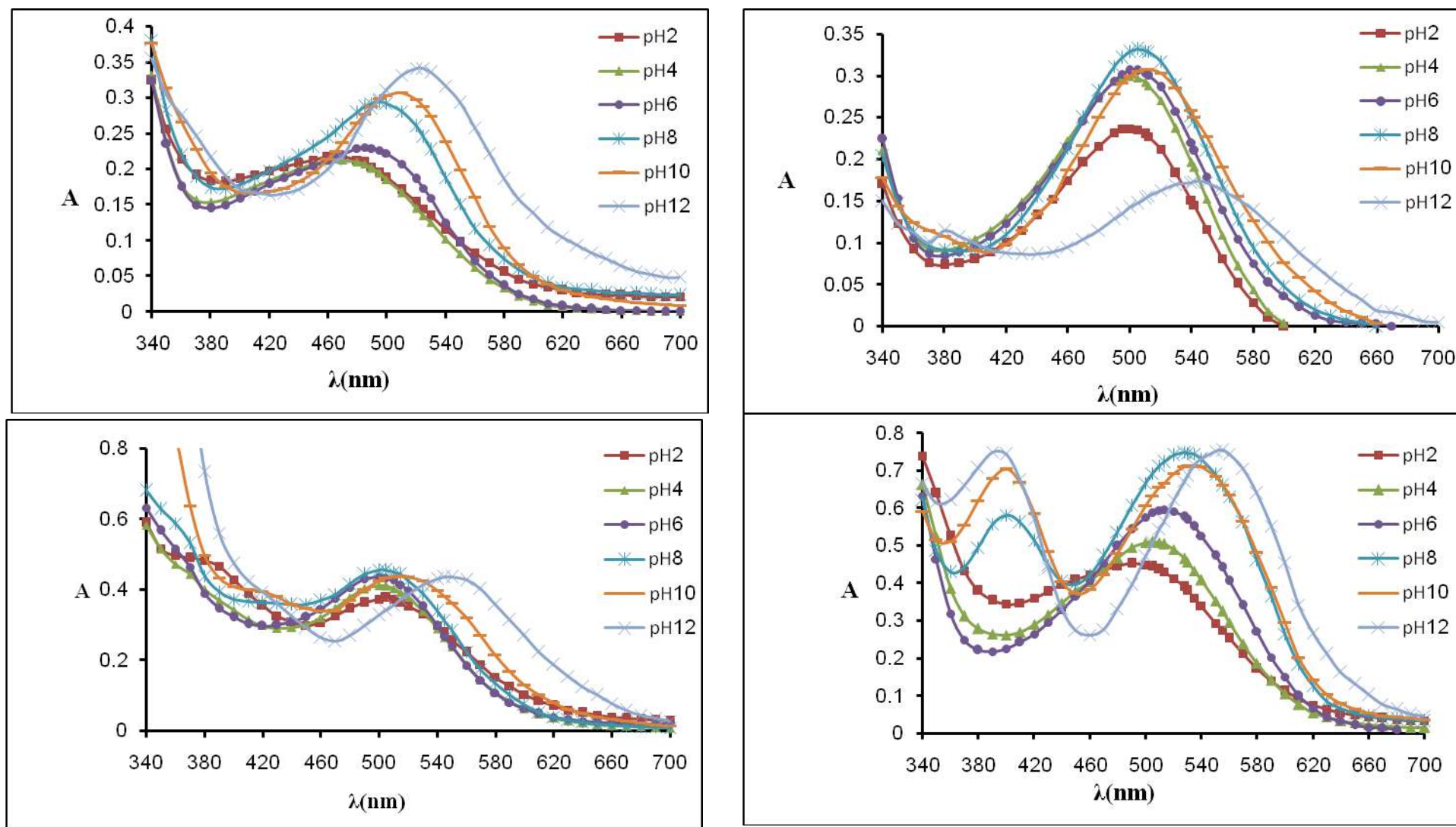


Figure 3 : Absorption spectra of azo dyes in various universal buffer solutions

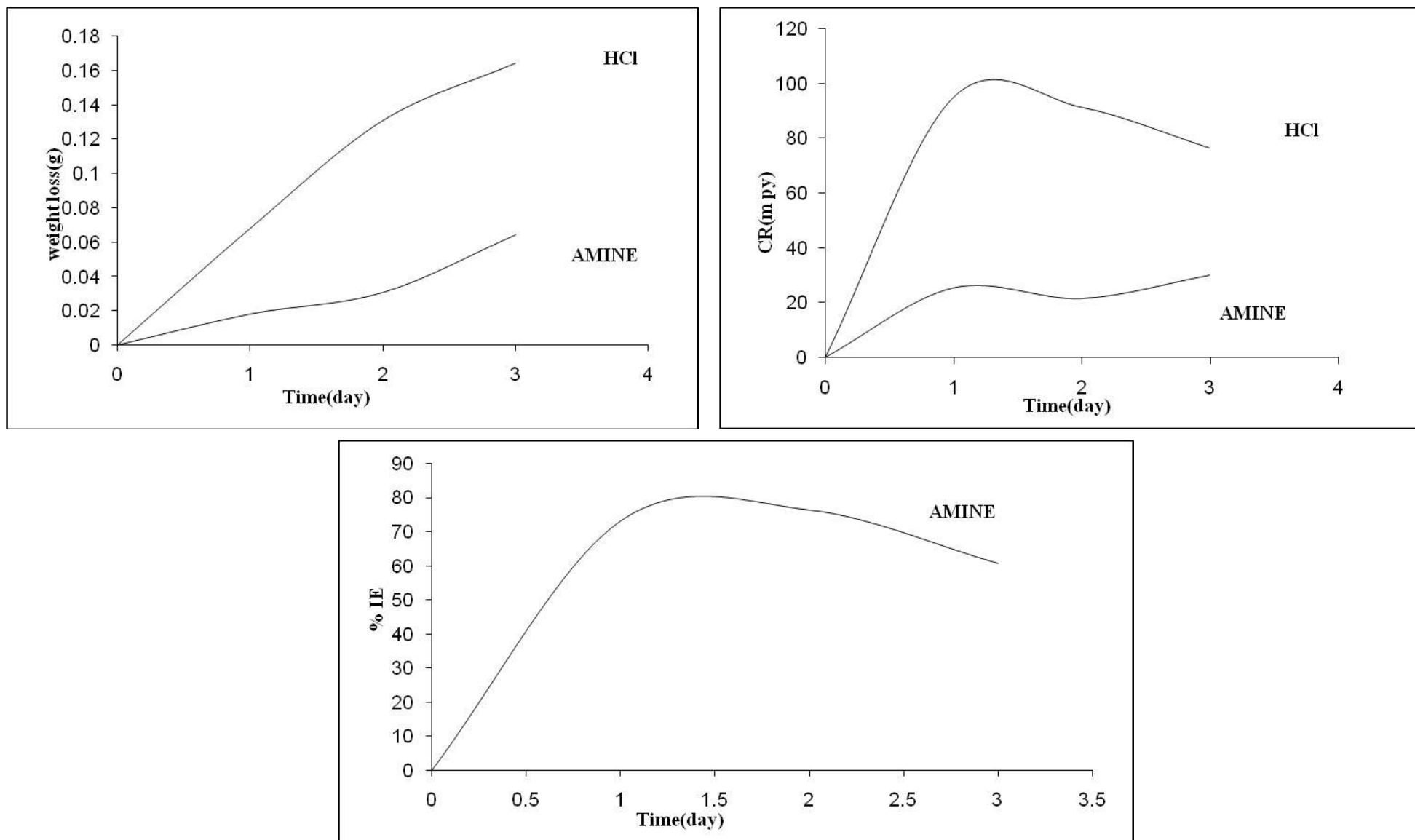


Figure 4 : Weight loss-time, Corrosion rate-time and Inhibitor efficiency-time curves for the corrosion of C-steel in 0.5M HCl in absence, and presence of amine

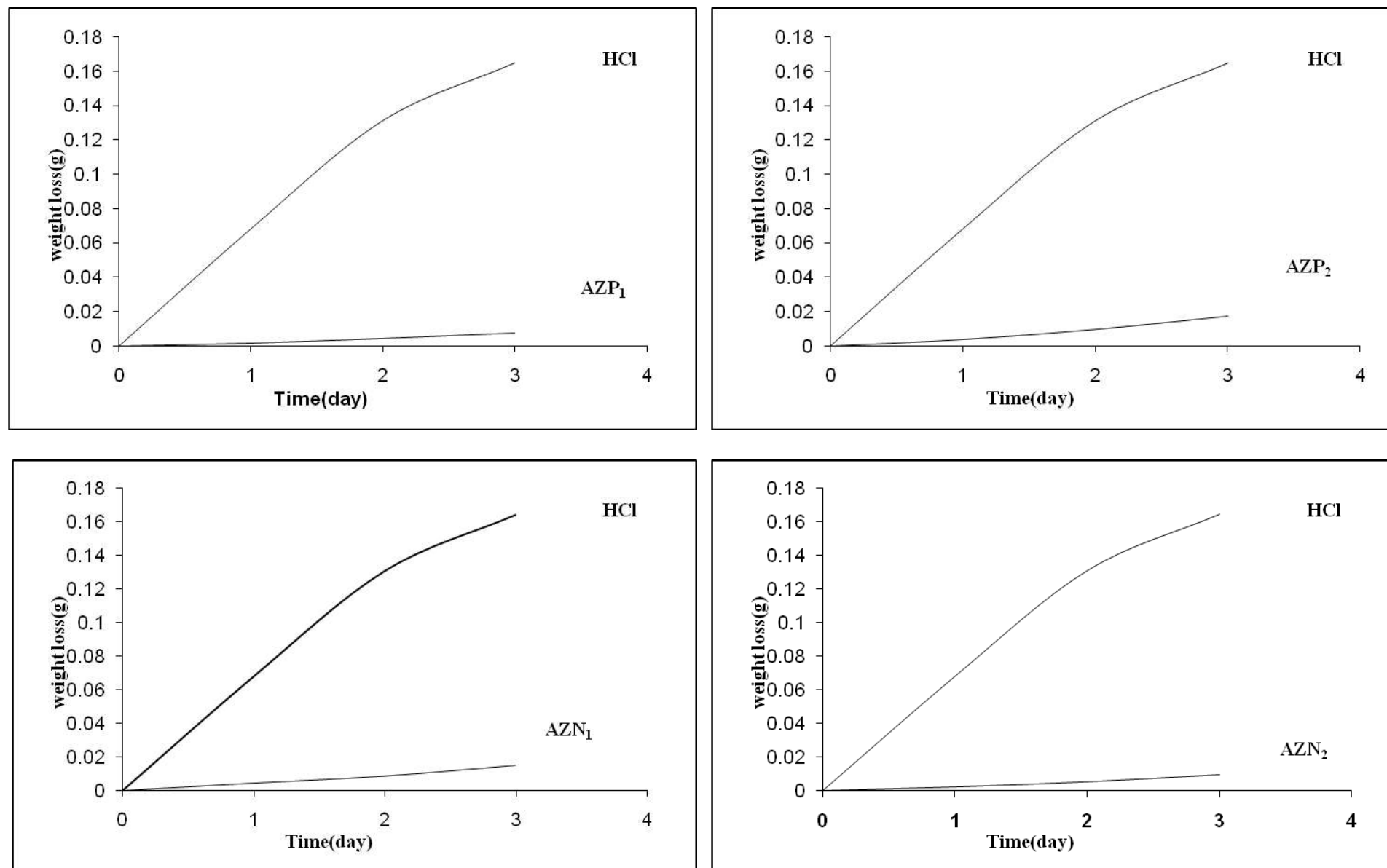


Figure 5 : Weight loss-time curves for the corrosion of C-steel in 0.5M HCl in absence, and presence of azo dyes

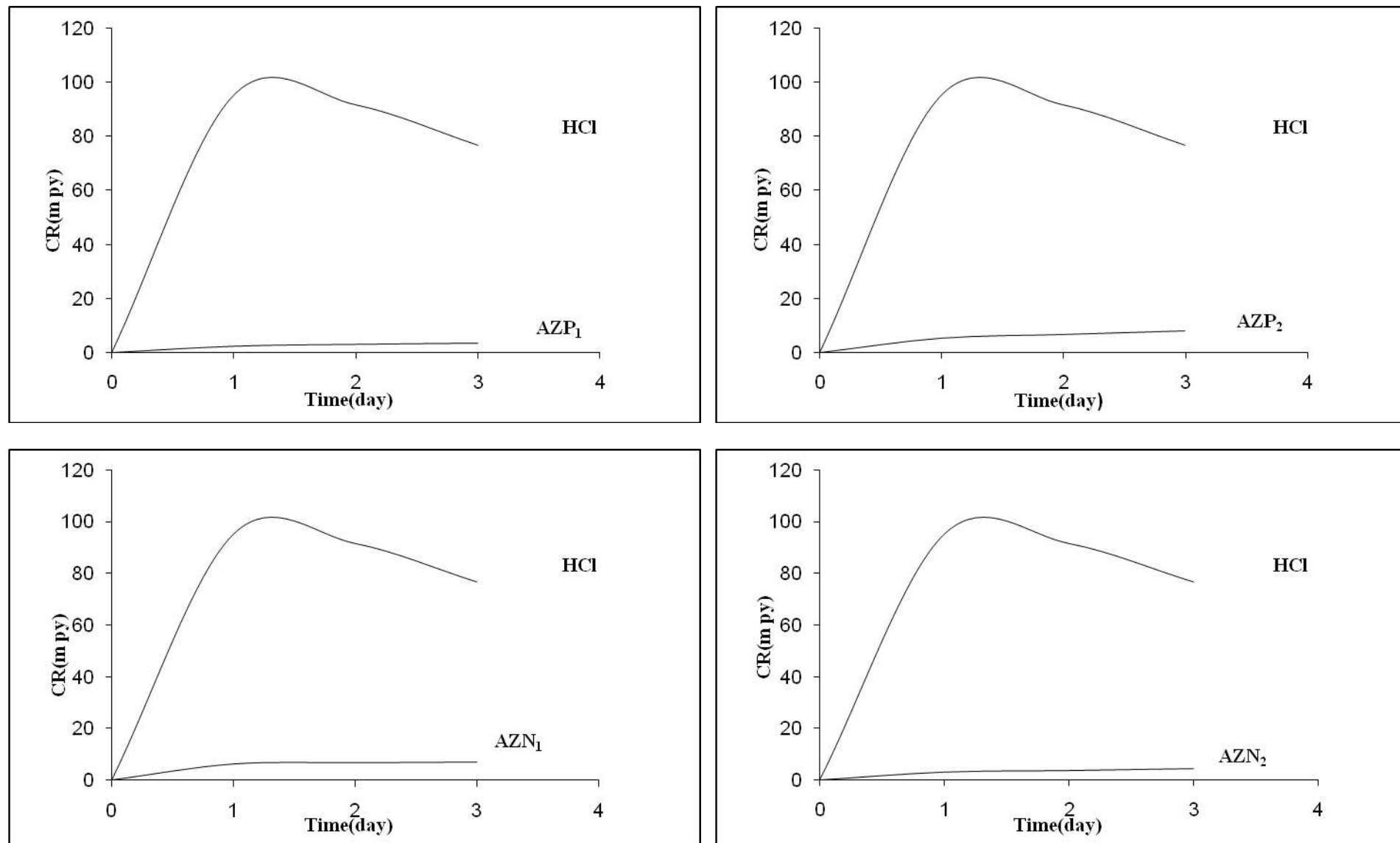


Figure 6 : Corrosion rate-time curves for the corrosion of C-steel in 0.5M HCl in absence, and presence of azo dyes

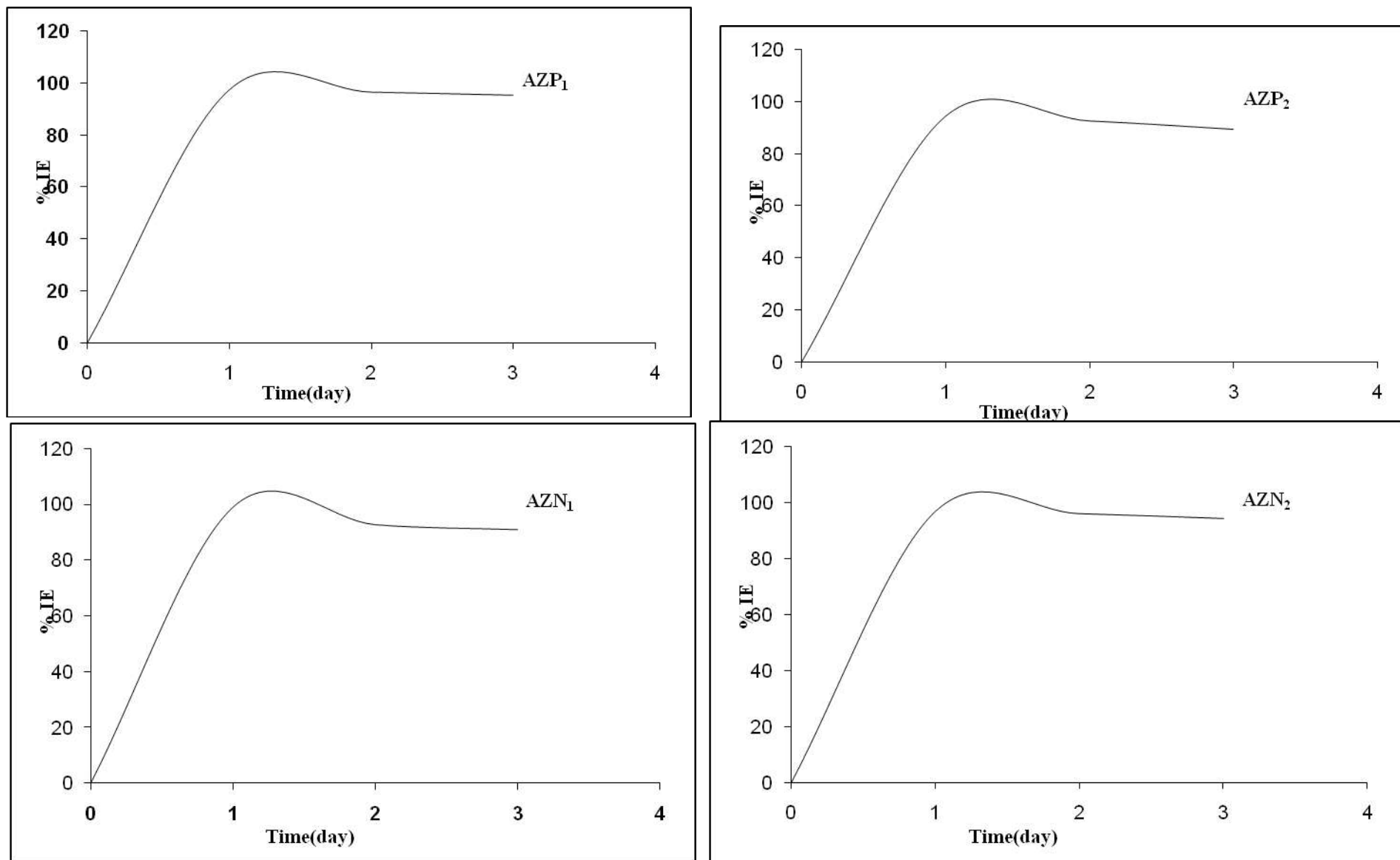
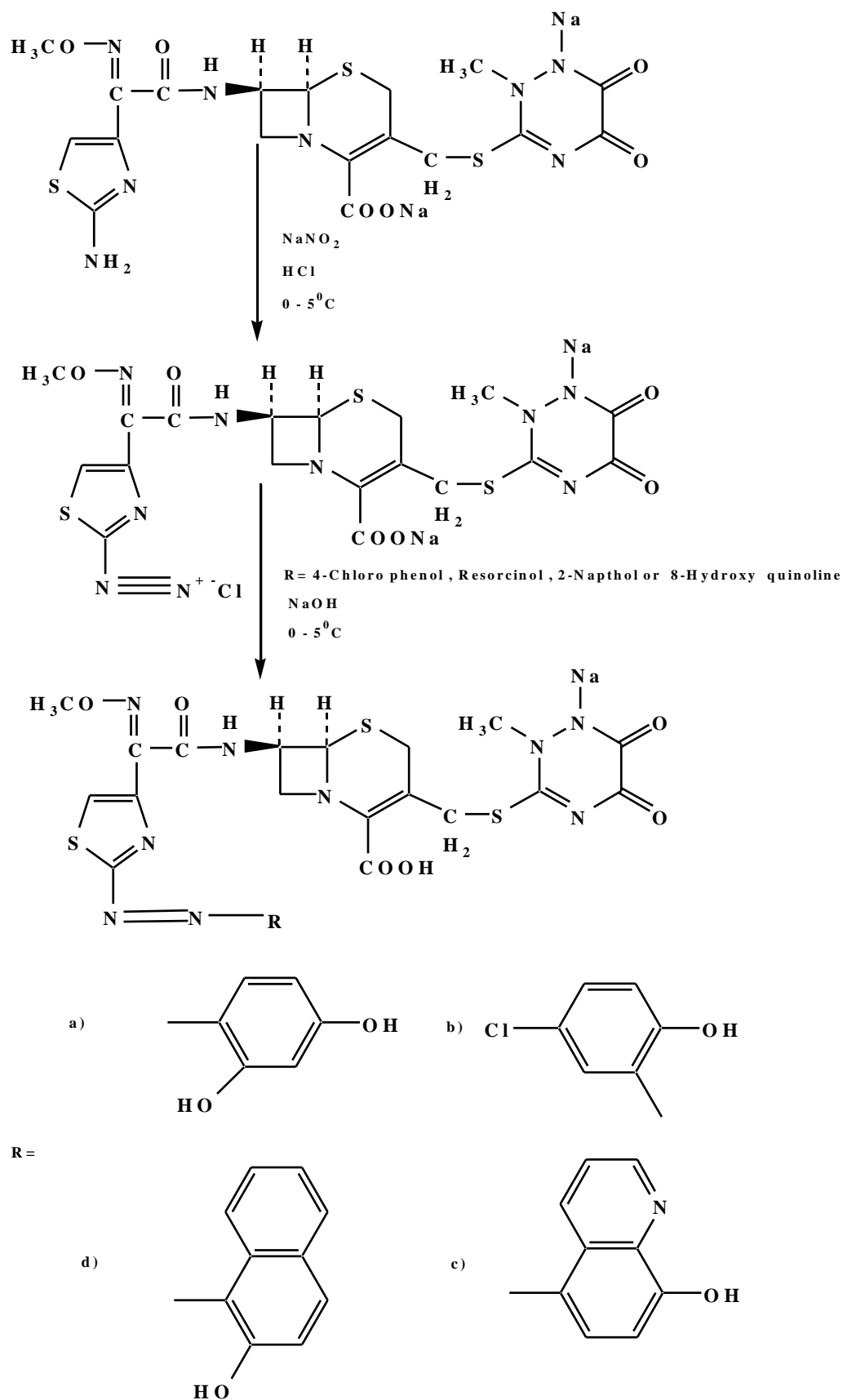


Figure 7 : Inhibitor efficiency-time curves for the corrosion of azo dyes of C-steel in 0.5M HCl



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تحضير ودراسة الخصائص الطيفية لصبغات آزوية جديدة وتقييمها كمثبطات تآكل للكربون ستيل في محلول حمض الهيدروكلوريك

رائد عوض عبيد ، أحمد مجيد جاسم ، محمد قاسم محمد
جامعة البصرة ، كلية التربية ، قسم الكيمياء

الخلاصة

لقد تضمن موضوع البحث تحضير صنف جديد من مركبات الازو الغير متجانسة مشتقة من الامين (6R;7R)-7-(2-أمينو-4-ثيازول)كلايوكسيلايدو]-8-أوكسو-3-[[3-1,2,5,6-رباعي هيدرو-2-مثيل-5,6-داي أوكسو-أس-ترايازين-3-يل)-ثايو[مثيل]-5-ثايا-1-آزابايسايكلو [4,2,0,1,2,4,0,2,4]أوكتا-2-بين-2-حامض الكاربوكسليك، $^{72}(Z)$ -0-مثيل أوكزيم، ملح ثنائي الصوديوم مع كل من الفينولات والنافثولات التالية:- 2-نافثول ، 8-هيدروكسي كوينولين ، 4-كلورو فينول ، ريسورسينول. وقد تمت دراسة تأثير المذيب والذالة الحامضية على سلوك تلك الصبغات بواسطة مطيافية الاشعة فوق البنفسجية - المرئية ثم قيست التوصيلية الكهربائية لتلك الصبغات وتقييمها كمثبطات تآكل للكربون - ستيل في محلول حمض الهيدروكلوريك (0.5N). بالإضافة الى دراستها بواسطة جهاز التحليل الدقيق للعناصر (CHN) ومطيافية الاشعة تحت الحمراء .