Study of the Corrosion Inhibition Efficiency by Using New Azo Compounds from Thiazole Derivatives

Saja H. Abdalla*, Salih H. Abbas, and Zeki A. Al-Shamkhani

Department of Chemistry, College of Science, University of Basrah, Al Basrah, Iraq.

*Corresponding authors E-mail: sajaha@gmail.com

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ABSTRACT

Azo compounds were prepared by reacting thiazole derivatives with 2-naphthol, o-vanillin, 4-(dimethyl amino) pyridine, and 4-bromo aniline with thiazole derivatives. The molar conductivity of the azo compounds prepared in this study was measured using (ethanol) as a solvent and at room temperature, all compounds showed non-electrolytic behavior. The compounds were characterized by TGA/DTA, studying the thermal stability and calculating the activation energy of azo compounds, where the thermal analyses of the prepared compounds were measured to identify the temperature at which the compound disintegrates, as the compound has more than one degree of disintegration, and this variation in the degrees of disintegration and the remaining materials depends on the nature of the compound and the type of groups present in it, as well as the efficiency of their use as materials. Resistant to corrosion by weight loss method with different concentrations at constant temperature. The higher the concentration, the greater the inhibition efficiency.

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1. Introduction

Azo compounds are one of the most important colored organic compounds which were characterized by the presence of a group (-N=N-) in their composition. They are broad as they act as inhibitors of protein and RNA formation, they also have medicinal uses, they act as anti-bacterial, and they are used as cosmetics [1]. They are considered one of the important intermediates in the preparation of many other organic compounds, they have wide uses in the field of inorganic chemistry as an important ligand used in the preparation of many coordination complexes [2][3] and in the field of analytical chemistry as reagents in the search for metals. Recent studies have focused on azo dyes derived from benzo thiazoles and thiazole derivatives due to their applications in various fields, especially in the design of biologically active molecules. Azo compounds, including those containing an aryl azo group, have been used in the field of pharmacy. This type of compound has also been exploited in various industrial fields. In a recent study, a large number of polymeric dyes were prepared, including polyamide and nylon fibers [4]. The appearance of these colors is due to the presence of the double bond between the two nitrogen atoms, which can absorb light in the visible region between (400-750) nm [5]. We comparison of the thermal decomposition of compounds, the activation energy E_a and the thermodynamic parameters ΔS, ΔH, and ΔG of the activated compound were calculated by using the Freeman-Carroll equation [6]. Molar conductivity measurements of the prepared azo compounds were done in this study using (ethanol) as a solvent and at room temperature which demonstrated that all compounds have non-electrolytic behavior, and azo compounds were used as anticorrosion agents using the weight loss method [7].

2. Experimental procedure

2.1 Synthesis of thiazole derivatives

Thiazole derivatives (T1-T3) were prepared by adding iodine (15 mmol) to a mixture of acetophenone (15 mmol) and thiourea (30 mmol) substitutes, using absolute ethanol as a solvent. The mixture was then heated under a water bath in a glass beaker for (6-8) h, then added. Add hot distilled water to it until the solution becomes homogeneous and rises for 24 h. The course of the reaction was followed by using thin-layer chromatography (TLC), as shown in the Fig. (1).

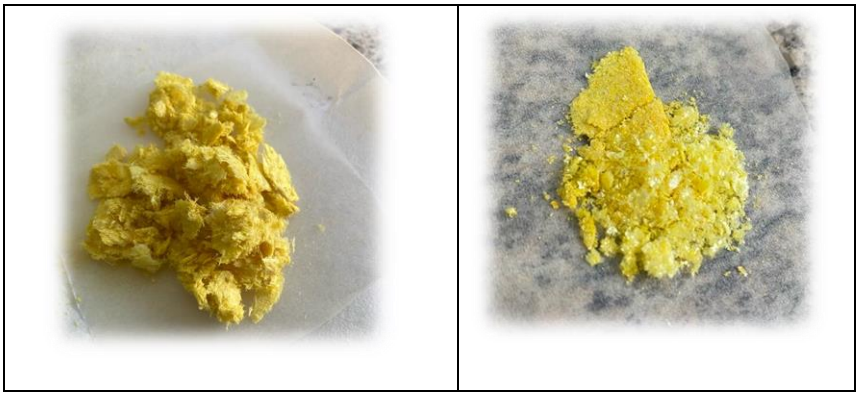


Figure 1: Image of the preparation of thiazole derivatives

2.2 Synthesis of azo compounds

Dissolving the amine (2 mmol) prepared from thiazole derivatives (T1-T3), in a mixture of concentrated hydrochloric acid (2 ml) and distilled water (8 ml), then place it in an ice bath until the temperature reaches (0-5) °C. Dissolve 7.5 mmol of sodium nitrite in (10 ml) of distilled water and place in an ice bath at a temperature of (0-5) °C, prepare diazonium salt by adding sodium nitrite solution in the form of drops to the solution with continuous stirring and maintaining the solution temperature at (0-5) °C, compounds (A1-A9) were prepared from coupling reaction between diazonium salts derived from 2-amino-4-(4- phenyl) thiazole, 2-amino-4-(4-methyl phenyl) thiazole, 2-amino-4-(4- methoxy) phenyl thiazole (T1-T3) with 2-naphthol, o-vanillin, 4-(dimethyl amino) pyridine. Also, azo compounds (A10-A12) were prepared from the coupling reaction between diazonium salts derived from 4-bromo aniline with 2-amino-4-(4-phenyl) thiazole, 2-amino-4-(4-methyl phenyl) thiazole, 2-amino-4-(4-methoxy) phenyl thiazole, as shown in the Fig. (2).

Figure 2: Image of the preparation of Azo Compounds

3. Results and Discussion

3.1 Molar Conductivity

Molar conductivity is considered one of the important diagnostic methods, as this data shows whether ionic or non-ionic and also helps in predicting the geometric shape of the prepared compounds. The purpose or goal of molar conductivity measurements is to deduce the ionic formulas of the compounds in their solutions [8]. The molar conductivity of azo compounds prepared in ethanol solvent was studied for each solution at room temperature and at a concentration of (1 x 10^{-3} M). Through the molar conductivity values of the compounds prepared in this study, it was found that they are considered non-electrical conductors because they showed very small values of conductivity. This indicates that all compounds are non-electrolytic (non-ionic) substances.

3.2 Thermogravimetric analysis (TGA)

The thermodynamic functions of the prepared were calculated for each stage of thermogravimetric decomposition using the Freeman-Carroll method graphically and according to the equation (1) [9][10], Table (1) show the obtained thermal stability and thermos dynamic functions, the thermal analysis TGA/DTA of the prepared compounds was measured within the temperature range (25-550) °C using a device type (shimadzu- TGA-50), type of cell is platinum, flow rate 30(ml/min).

$$\frac{\Delta \log (dW/dt)}{\Delta \log W_r} = \frac{-(E^*/2.303R)\,\Delta(T^{-1})}{\Delta \log W_r} + n \quad ----(1)$$

Table 1: Thermal stability and thermodynamic functions from TGA data of azo compounds (A1-A12)

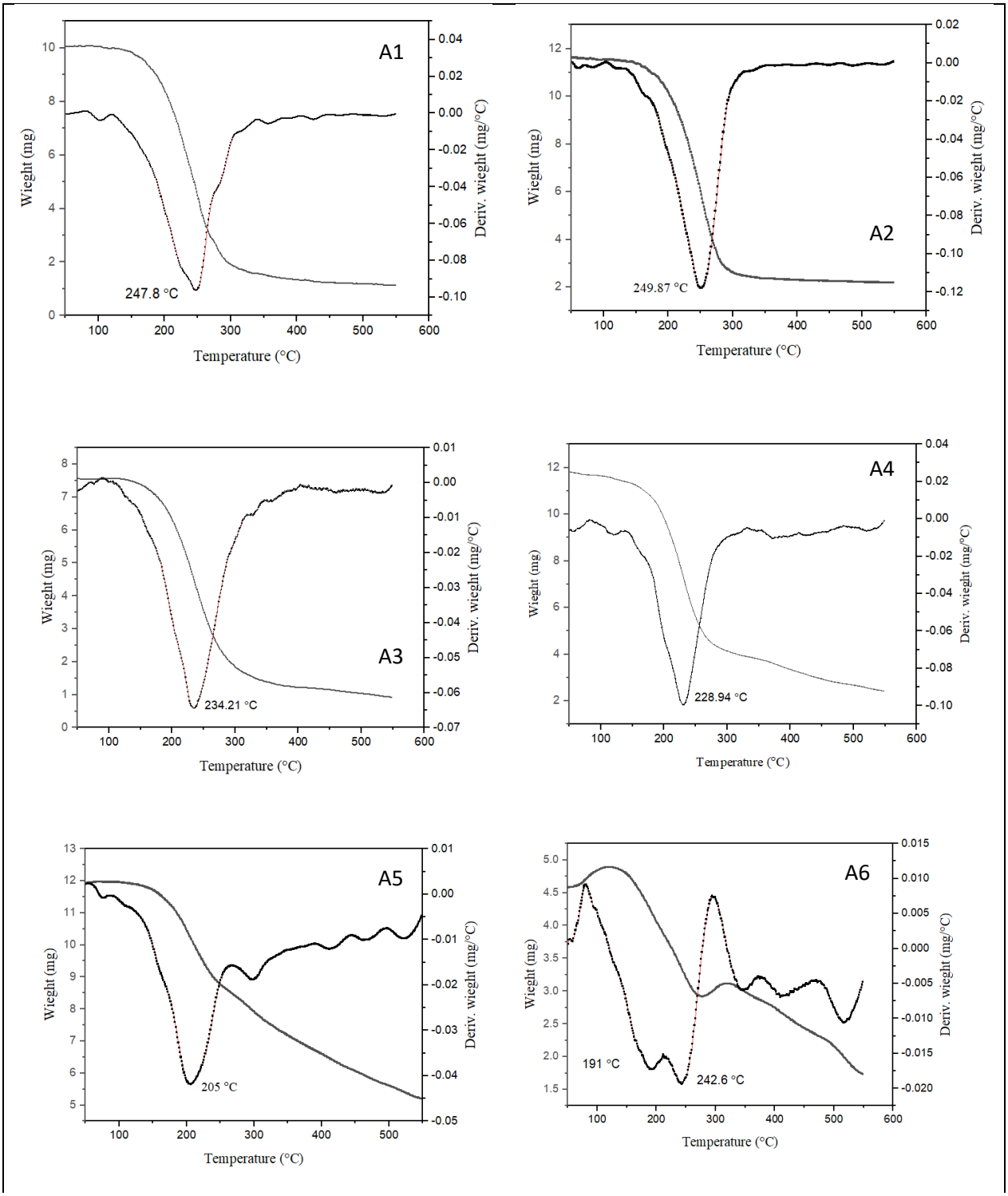
Sample	Z	T_s (K)	Ea KJ/mol	ΔH KJ/mol	ΔS KJ/mol	ΔG KJ/mol
A1	0.555	520.8	16.102	- 4.313	-254.49	129.27
A2	5.249	522.87	0.593	-4.347	-235.6	123.21
A3	0.454	516.21	52.214	-4.171	-255.95	130.02
A4	4.754	501.94	0.746	-4.172	-236.32	118.61
A5	0.714	478	11.105	-4.004	-251.68	120.29
A6	6.370	515.6	0.495	-4.264	-234.07	120.07
A7	1.247	549	57.441	-4.506	-248.19	136.25
A8	0.714	555.5	9.573	-4.608	-252.93	140.49
A9	5.586	608.7	0.534	-5.054	-236.58	143.83
A10	6.794	599.4	0.376	-4.979	-234.82	140.65
A11	6.097	514.7	0.496	-4.278	-234.46	120.67
A12	4.0	621.2	1.684	-5.161	-239.53	148.74

Thermal studies are among the most common and used methods for evaluating the thermal properties of materials. These methods depend on monitoring the change in the behavior of the material as a function of temperature and using a fixed or variable heating rate depending on the type of study. On the other hand, this study is accompanied by physical and chemical changes in the studied materials. Or these changes depend on the nature of the material, the temperature, as well as the type of gas used (such as air, nitrogen, or argon). Table 2 is listed the thermal decomposition of the prepared compounds, as compounds (A1, A2, A3, A4) include one decomposition step, compounds (A5, A8) include two decomposition steps, compound (A11) three decomposition steps, and compound (A9) four decomposition steps. While compound (A10) decomposed six steps, this is evidence of thermal stability, as shown in the Fig. (3), this difference in the degrees of disintegration of the compounds (A1-A12) and the remaining materials depends on the nature of the compound, the type of groups

present in it, and the extent of their work. It also depends on the presence of moisture or the presence of the solvent.

Table 2: Thermal analytical results TG and DTA of azo compounds (A1-A12)

Sample	Maximum disintegration temperature ℃	Temperature of loss of 25% of the material ℃	Temperature of loss of 50% of the material ℃	Temperature of loss of 75% of the material ℃	Remaining at temperature of 550 ℃	Disintegration rate %/min
A1	247	216	240	268	11.26	5.72
A2	250	216	240	260	18.38	5.08
A3	238	216	244	296	11.57	5.69
A4	232	212	233	336	21.88	5.05
A5	204, 297	202	272	420	43.76	2.693
A6	191, 242, 348, 430, 532	236	472	> 550	37	4.65
A7	211, 276	225	278	321	18	6.68
A8	285, 398	215	365	302	24.34	4.54
A9	102, 238, 342, 417	329	442	> 600	40.76	4.62
A10	100, 102, 216, 263, 325, 512	296	483	> 600	44.66	3.77
A11	242, 275, 521	333	> 600	> 600	42.62	4.62
A12	226, 348, 418	372	510	> 550	48	4.87



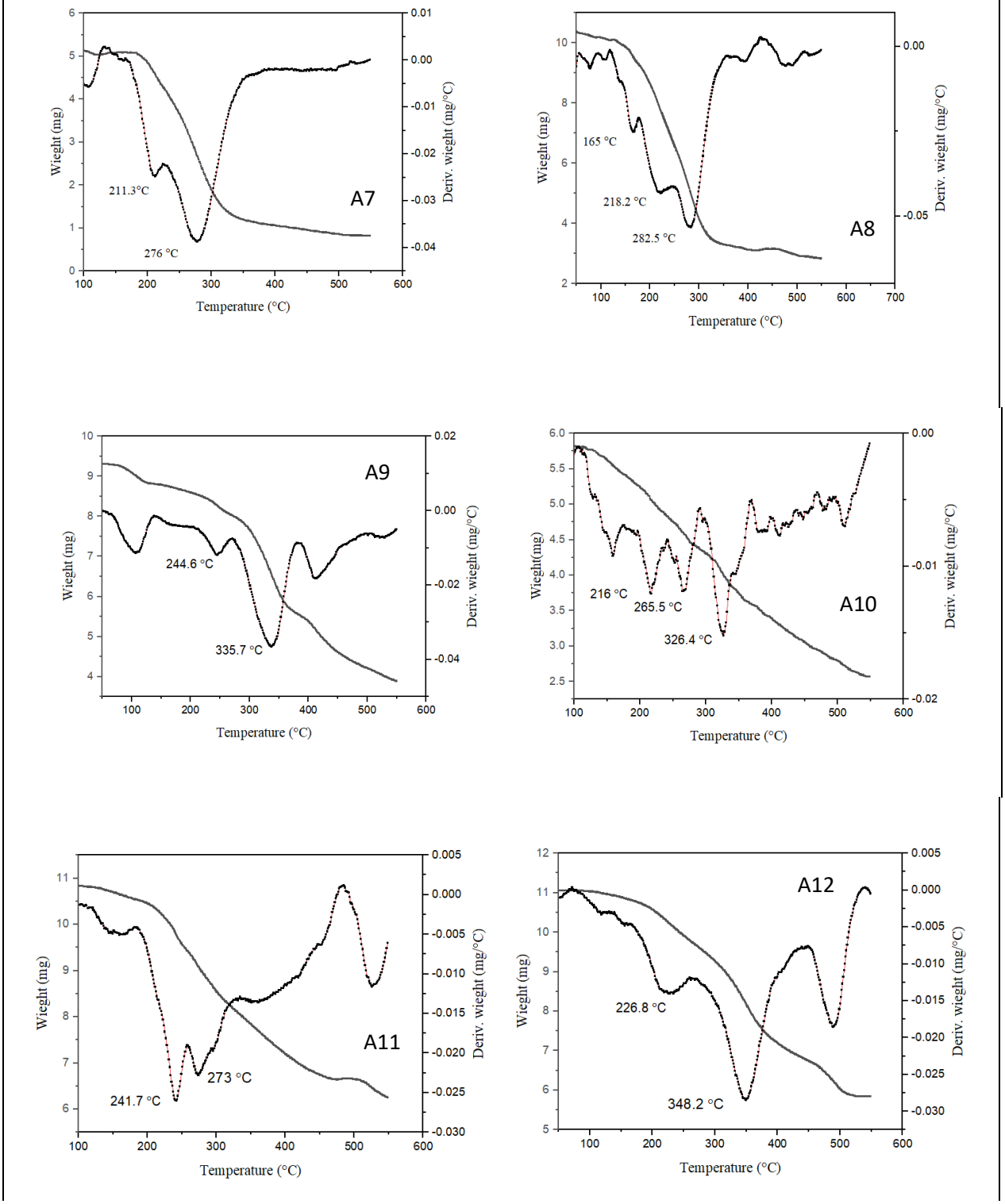


Figure 3: TG and DTA of (A1-A12)

4. Weight loss measurements

The weight loss method is one of the common methods in corrosion tests [11]. This method was used directly to measure the efficiency of corrosion inhibitors. It is considered one of the simplest methods, but at the same time, it gives high accuracy, as the lost weight is noticeably observed in this method. A study was conducted on the effectiveness of corrosion of carbon steel models in acidic media with 1 M hydrochloric acid as a corrosion medium. The weight loss method is used to calculate the speed of corrosion after immersing the models in the corrosive environment for a specific period at (24, 48, 72) h and at a room temperature of 27 °C. Use carbon steel models with dimensions of (0.3 x 1.8 x 2.5) cm so that the submerged area is (9.8) cm^2. Dimensions were measured using a vernier with a sensitivity of 1 mm. The results are attached to Table (3) inhibitor efficiency (ŋ%), corrosion ratio (C_R), and surface coverage (θ) [12].

Inhibition efficiency ŋ % is calculated[13]:

$$ŋ\% = \ (C_R\ (uninh) - C_R(inh))/C_R(uninh)\ \times 100 - - - -(2)$$

Surface coverage θ is calculated:

$$\theta = \ \frac{C_R\ (uninh) - C_R(inh)}{C_R(uninh)} - - - -(3)$$

$C_{R(uninh)}$ = Corrosion average absence inhibitor

$C_{R(inh)}$ = Corrosion average presence inhibitor

Table (3-7) show the effect of increasing the inhibition efficiency with increasing concentration. At a concentration of 10 ppm, the C_R is (0.00046), and it was decreased with increasing of the concentration of inhibitor, it has been (0.0002) at 50 ppm. This is due to the fact that increasing the concentration leads to an increase in the thickness of the protective layer on the surface of the alloy resists the corrosive environment, and then the inhibition efficiency increases on the surface. It works to move out corrosive particles represented by water molecules containing dilute hydrochloric acid and forms a layer on the surface of the alloy that covers the active sites on the surface of the alloy. Thus, the surface is more homogeneous, and then this layer works to reduce the corrosion rate [14], the mechanism of action of corrosion inhibitors can be explained based on molecular adsorption of the inhibitor on the surface of the carbon steel alloy. Weight loss values as a function of time with concentration (10, 20,30,40,50) ppm of a compound as inhibitor in the presence and absence of the inhibitor, as shown in the Fig. 4-8. The inhibitory role of (azo compounds) was studied in an acidic medium of hydrochloric acid at a concentration of (1 M) as an acidic medium for corrosion, and by adding different concentrations (10, 20, 30, 40, 50) ppm of inhibitors (A1, A2,

A3, A8, A10, A11) to the corrosive medium. This is due to the ability of azo dyes to form protective barriers, which contribute to reducing acid attack and preventing the formation of corrosion products. The inhibitory capacity of these dyes also varies from one dye to another due to the difference in the groups replaced by the azo dye, and this is clear when comparing the amount of loss before adding the inhibitor[15][16]. The efficiency of the inhibitor depends on some factors, including the number of adsorption sites, charge density, molecular size, temperature of hydrogenation, and pattern of interaction with the metal surface [17][18].

Table 3: Inhibition efficiency ŋ % for carbon steel with 10 ppm of an inhibitor at room temperature 27 °C

Inhibitor	Time (h)	Weight loss (g)	Corrosion rate ($mg.cm^{-2}.h^{-1}$)	Surface coverage θ	Inhibition efficiency ŋ %
Blank 1 M HCl	24	0.2726			
	48	0.5086			
	72	0.9132			
1-((4-phenylthiazol-2-yl) diazenyl) naphthalene-2-o1 (A1)	24	0.1098	0.00046	0.5972	59.72
	48	0.1933	0.00041	0.6199	61.99
	72	0.3298	0.00046	0.6388	63.88
1-((4-(*p*-tolyl) thiazol-2-yl) diazenyl) naphthalene-2-o1 (A2)	24	0.0995	0.00042	0.6349	63.49
	48	0.1566	0.00033	0.6920	69.20
	72	0.2854	0.00040	0.6874	68.74
1-((4-(4-methoxyphenyl) thiazol-2-yl) diazenyl) naphthalene-2-o (A3)	24	0.1124	0.00047	0.5876	58.76
	48	0.1974	0.00041	0.6118	61.18
	72	0.3254	0.00046	0.6436	64.36
N,N-dimethyl-2-((4-*p*-tolyl) thiazol-2-y diazenyl) pyridine-4-amine (A8)	24	0.1062	0.00045	0.6104	61.04
	48	0.2197	0.00046	0.568	56.80
	72	0.3658	0.00051	0.5994	59.94

2-((4-bromophenyl) diazenyl) -4-phenyl thiazole (A10)	24	0.1129	0.00048	0.5858	58.58
	48	0.2327	0.00049	0.5424	54.24
	72	0.4008	0.00056	0.5611	56.11
2-((4-bromophenyl) diazenyl) -4-(*p*-tolyl) thiazole (A11)	24	0.1302	0.00055	0.5223	52.23
	48	0.2097	0.00044	0.5876	58.76
	72	0.3678	0.00052	0.5972	59.72

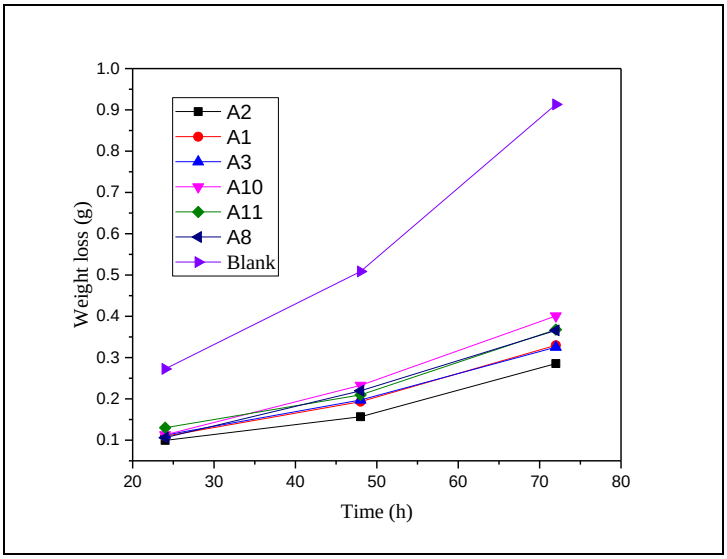


Figure 4: Weight loss values as a function of time with 10 ppm of a compound as an inhibitor

Table 4: Inhibition efficiency ŋ % for carbon steel with 20 ppm of an inhibitor

Inhibitors	Time (h)	Weight loss (g)	Corrosion rate ($mg.cm^{-2}.h^{-1}$)	Surface coverage θ	Inhibition efficiency ŋ %
Blank 1 M HCl	24	0.2726			
	48	0.5086			
	72	0.9132			
	24	0.1176	0.0005	0.5685	56.85

1-((4-phenylthiazol-2-yl) diazenyl) naphthalene-2-o1 (A1)	48	0.1944	0.00041	0.6177	61.77
	72	0.3411	0.00048	0.6264	62.64
1-((4-(*p*-tolyl) thiazol-2-yl) diazenyl) naphthalene-2-o1 (A2)	24	0.1123	0.00047	0.5880	58.80
	48	0.1744	0.00037	0.6570	65.70
	72	0.2811	0.00039	0.6921	69.21
1-((4-(4-methoxyphenyl) thiazol-2-yl) diazenyl) naphthalene-2-o1 (A3)	24	0.1154	0.00049	0.5766	57.66
	48	0.2118	0.00045	0.5835	58.35
	72	0.3211	0.00046	0.6483	64.83
N,N-dimethyl-2-((4-*p*-tolyl) thiazol-2-yl) diazenyl) pyridine-4-amine (A8)	24	0.0943	0.00041	0.6540	65.40
	48	0.2097	0.00044	0.5876	58.76
	72	0.3054	0.00043	0.6655	66.55
2-((4-bromophenyl) diazenyl)-4-phenyl thiazole (A10)	24	0.1093	0.00046	0.5990	59.90
	48	0.2177	0.00046	0.5719	57.19
	72	0.3054	0.00043	0.6655	66.55
2-((4-bromophenyl) diazenyl)-4-(*p*-tolyl) thiazole (A11)	24	0.1143	0.00048	0.5807	58.07
	48	0.2077	0.00044	0.5916	59.16
	72	0.3154	0.00045	0.6546	65.46

at room temperature 27 °C

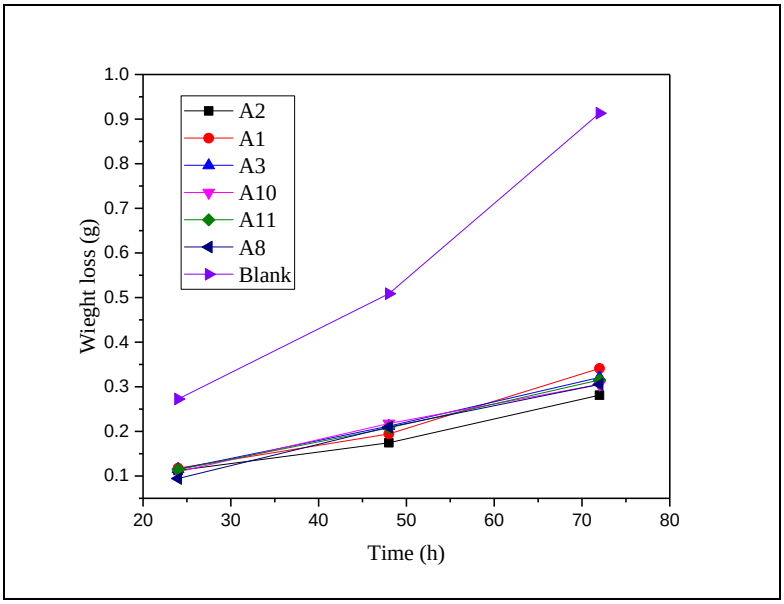


Figure 5: Weight loss values as a function of time with 20 ppm of a compound as inhibitor

Table 5: Inhibition efficiency ŋ % for carbon steel with 30 ppm of an inhibitor at room temperature 27 °C

Inhibitors	Time (h)	Weight loss (g)	Corrosion rate ($mg.cm^{-2}.h^{-1}$)	Surface coverage θ	Inhibition efficiency ŋ %
Blank 1 M HCl	24	0.2726			
	48	0.5086			
	72	0.9132			
1-((4-phenylthiazol-2-yl)diazenyl) naphthalene-2-o1 (A1)	24	0.0796	0.00033	0.7079	70.79
	48	0.1823	0.00038	0.6415	64.15
	72	0.2569	0.00036	0.7186	71.86
1-((4-(*p*-tolyl) thiazol-2-yl)diazenyl) naphthalene-2-o1 (A2)	24	0.0776	0.00032	0.7153	71.53
	48	0.0743	0.00015	0.8539	85.39
	72	0.2533	0.00035	0.7226	72.26

1-((4-(4-methoxyphenyl) thiazol-2-yl) diazenyl) naphthalene-2-o1 (A3)	24	0.0696	0.00029	0.7446	74.46
	48	0.1923	0.00041	0.6219	62.19
	72	0.2489	0.00035	0.7274	72.74
N,N-dimethyl-2-((4-*p*-tolyl) thiazol-2-yl)diazenyl) pyridine-4-amine (A8)	24	0.0949	0.0004	0.6518	65.18
	48	0.1313	0.00027	0.7418	74.18
	72	0.2169	0.00031	0.7624	76.24
2-((4-bromophenyl) diazenyl)-4-phenyl thiazole (A10)	24	0.0876	0.00037	0.6786	67.86
	48	0.1913	0.0004	0.6238	62.38
	72	0.2669	0.00037	0.7077	70.77
2-((4-bromophenyl) diazenyl)-4-(*p*-tolyl) thiazole (A11)	24	0.0919	0.00039	0.6628	66.28
	48	0.1613	0.00034	0.6828	68.28
	72	0.2069	0.00029	0.7734	77.34

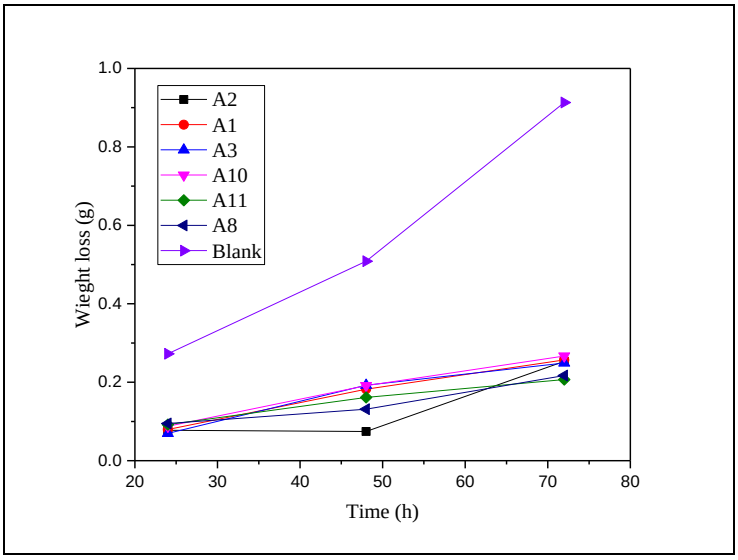


Figure 6: Weight loss values as a function of time with 30 ppm of a compound as an inhibitor

Table 6: Inhibition efficiency ŋ % for carbon steel with 40 ppm of an inhibitor at room temperature 27 °C

Inhibitors	Time (h)	Weight loss (g)	Corrosion rate ($mg.cm^{-2}.h^{-1}$)	Surface coverage θ	Inhibition efficiency ŋ %
Blank 1 M HCl	24	0.2726			
	48	0.5086			
	72	0.9132			
1-((4-phenylthiazol-2-yl)diazenyl) naphthalene-2-o1 (A1)	24	0.0543	0.00023	0.8008	80.08
	48	0.1211	0.00025	0.7618	76.18
	72	0.2287	0.00032	0.7495	74.95
1-((4-(*p*-tolyl) thiazol-2-yl)diazenyl) naphthalene-2-o1 (A2)	24	0.0554	0.00023	0.7967	79.67
	48	0.0675	0.00014	0.8672	86.72
	72	0.2211	0.00031	0.7578	75.78
1-((4-(4-methoxyphenyl) thiazol-2-yl) diazenyl) naphthalene-2-o1 (A3)	24	0.0587	0.00024	0.7846	78.46
	48	0.1508	0.00032	0.7034	70.34
	72	0.2277	0.00032	0.7506	75.06
N,N-dimethyl-2-((4-*p*-tolyl) thiazol-2-yl) diazenyl) pyridine-4-amine (A8)	24	0.0572	0.00024	0.7901	79.01
	48	0.1338	0.00028	0.7369	73.69
	72	0.2467	0.00034	0.7298	72.98
2-((4-bromophenyl) diazenyl)-4-phenyl thiazole (A10)	24	0.0572	0.00024	0.7901	79.01
	48	0.1328	0.00028	0.7388	73.88
	72	0.2267	0.00032	0.7517	75.17
2-((4-bromophenyl) diazenyl)-4-(*p*-tolyl) thiazole (A11)	24	0.0562	0.00023	0.7938	79.38
	48	0.1228	0.00026	0.7585	75.85
	72	0.2567	0.00036	0.7189	71.89

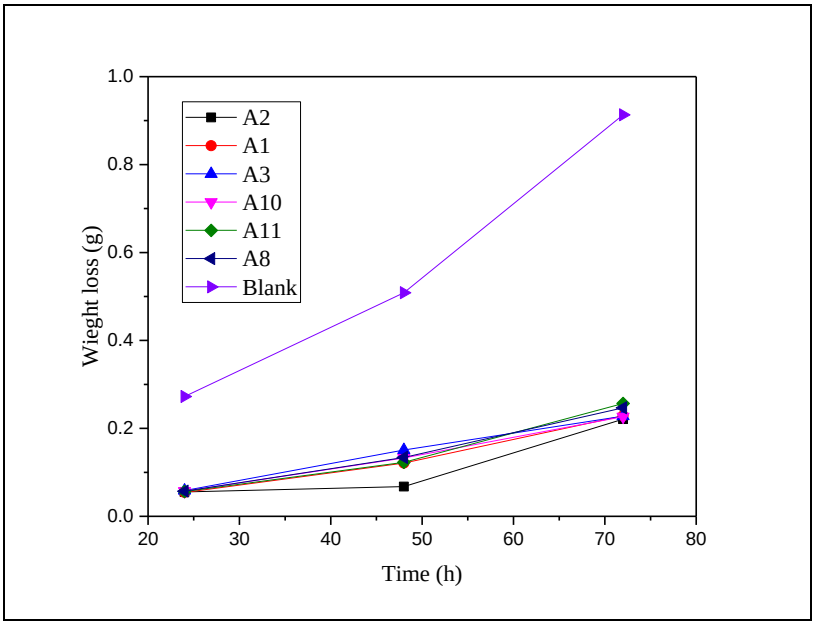


Figure 7: Weight loss values as a function of time with 40 ppm of a compound as an inhibitor

Table 7: Inhibition efficiency ŋ % for carbon steel with 50 ppm of an inhibitor at room temperature 27 °C

Inhibitor	Time (h)	Weight loss (g)	Corrosion rate ($mg.cm^{-2}.h^{-1}$)	Surface coverage θ	Inhibition efficiency ŋ %
Blank 1 M HCl	24	0.2726			
	48	0.5086			
	72	0.9132			
1-((4-phenylthiazol-2-yl) diazenyl) naphthalene-2-o1 (A1)	24	0.0476	0.0002	0.8253	82.53
	48	0.0612	0.00013	0.8796	87.96
	72	0.1433	0.0002	0.8430	84.30
1-((4-(*p*-tolyl) thiazol-2-yl) diazenyl) naphthalene-2-o1 (A2)	24	0.0576	0.00024	0.7887	78.87
	48	0.0546	0.00011	0.8926	89.26
	72	0.1633	0.00023	0.8211	82.11
1-((4-(4-methoxyphenyl)	24	0.0696	0.00029	0.7446	74.46

thiazol-2-yl) diazenyl) naphthalene-2-o1 (A3)	48	0.0512	0.0001	0.8993	89.93
	72	0.1233	0.00017	0.8649	86.49
N,N-dimethyl-2-((4-*p*-tolyl) thiazol-2-yl) diazenyl) pyridine-4-amine (A8)	24	0.0546	0.00023	0.7997	79.97
	48	0.0692	0.00014	0.8639	86.39
	72	0.1313	0.00018	0.8562	85.62
2-((4-bromophenyl) diazenyl) -4- phenyl thiazole (A10)	24	0.0496	0.00021	0.8180	81.80
	48	0.0587	0.00012	0.8845	88.45
	72	0.1223	0.00017	0.8660	86.60
2-((4-bromophenyl) diazenyl)-4- (*p*-tolyl) thiazole (A11)	24	0.0446	0.00018	0.8363	83.63
	48	0.0592	0.00012	0.8836	88.36
	72	0.1203	0.00017	0.8682	86.82

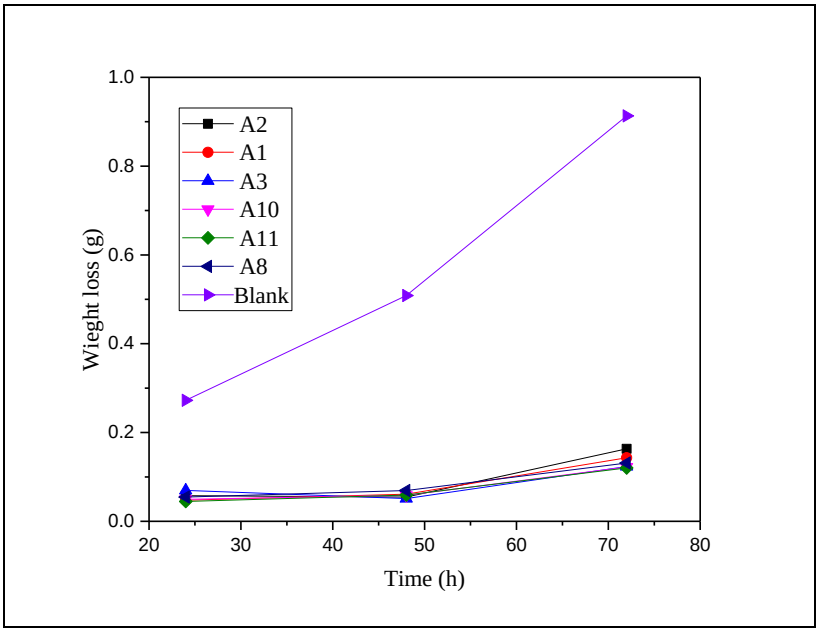


Figure 8: Weight loss values as a function of time with 50 ppm of a compound as an inhibitor

Conclusions

Azo compounds were prepared from thiazole derivatives, as demonstrated by molar conductivity measurements of the azo compounds prepared in this study using (ethanol) as a solvent and at room temperature. All compounds have non-electrical behavior. The prepared compounds were studied thermally and the thermodynamic functions (ΔG, ΔS, ΔH) were calculated for each stage of thermal decomposition, and the activation energy was calculated. The efficiency of the prepared compounds (A1, A2, A3, A8, A10, A11) as corrosion inhibitors in hydrochloric acid (1 M) for carbon steel was studied and evaluated using the weight loss method at concentrations (10, 20, 30, 40, 50) ppm. We conclude from this study that a high inhibition efficiency was obtained, as the higher the concentration, the greater the inhibition efficiency, these compounds can be used as corrosion inhibitors at different temperatures.

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دراسة كفاءة تثبيط التآكل باستخدام مركبات الآزو الجديدة من مشتقات الثيازول

سجى حيدر عبدالله * , صالح حمزة عباس , زكي عصمان الشمخاني

قسم الكيمياء , كلية العلوم , جامعة البصرة , البصرة , العراق

المستخلص

تم تحضير مركبات الآزو الجديدة من تفاعل مشتقات الثيازول مع 4-(dimethyl amino)pyridine , o-vanillin, 2-naphthol و4-bromo aniline مع مشتقات الثيازول.

تم قياس التوصيلية المولية لمركبات الآزو المحضرة في هذه الدراسة باستخدام (الإيثانول) كمذيب وعند درجة حرارة الغرفة أظهرت جميع المركبات سلوكاً غير إلكتروليتياً. تم تشخيص المركبات بتقنية TGA/DTA ودراسة الثبات الحراري وحساب طاقة التنشيط لمركبات الآزو، حيث تم قياس التحاليل الحرارية للمركبات المحضرة للتعرف على درجة الحرارة التي يتفكك عندها المركب حيث أن المركب له أكثر من درجة من التفكك، ويعتمد هذا الاختلاف في درجات التفكك والمواد المتبقية على طبيعة المركب ونوع المجموعات الموجودة فيه، وكذلك كفاءة استخدامها كمواد مقاومة للتآكل بطريقة فقدان الوزن بتراكيز مختلفة عند درجة حرارة ثابتة. كلما زاد التركيز، زادت كفاءة التثبيط.