Study on Corrosion Inhibition of Brass in water using Horse Tail Herb as Green Inhibitor

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ABSTRACT

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Brass used in heat exchanger is corroded in industrial water. The used inhibitor is extracted from horse tail herb added in different concentrations and temperature to industrial water in which heat exchanger if exposed to these conditions. Brass is studied under the above conditions using potentiodynamic polarization, scanning electron microscopy (SEM) and X-ray diffraction. Potentiodynamic polarization technique was used to evaluate the corrosion rate data. Inhibition efficiency of 92.1 % at 298 k was achieved with the addition of 1350 ppm of horse tail herb from potentiodynamic polarization measurements. Potentiodynamic curves show that the presence of extracted horse tail herb in industrial water affects both the anodic and cathodic processes, decreases the corrosion current density and shifts the corrosion potential towards more noble values; the inhibitors are mixed type inhibitors.

Keywords: Brass; inhibitors; Industrial water; electrochemical studies: SEM

دراسة تثبيط تاكل الراصد المستخدم في المبادلات الحرارية باستخدام عينة ذنب الخيل

الخلاصة

يتاكل البراص المستخدم في المبادلات الحرارية في المياه الصناعية. تم اضافة المشبط المستخلص من عشبة ذنب الخيل عند تراكيز و درجات حرارة مختلفة مشابهة للظروف المتعرضة لها المبادلات الحرارية. تمت الدراسة ضمن الظروف اعلاه باستخدام تقنية المجهاد الساكن, المجهر الالكتروني الماسح و حيود الاشعة السينية. من نتائج التي تم الحصول عليها من منحنيات الاستقطاب تبين ان كفائة التثبيط 92.1% عند 298 كلفن عند اضافة 1350 جزء بالمليون من المثبط اعلاه.

INTRODUCTION

orrosion has been known to main since the earliest metallurgical times and has been a constant drain on his productive activities, as such, purposeful attention have been focused on the problem both by scientists and engineers [1]. It is the disintegration of an engineered material [metal alloy] into its constituent atoms due to chemical reactions within their surrounding environment [2].

Copper and its alloys are widely used in industry because of their excellent electrical and thermal conductivity and are often used in heating and cooling system

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alloy

0.00014

[3–5]. Brass has been widely used as tubing material for condensers and heat exchangers in various cooling water systems [6–11].

Brass is susceptible to a corrosion process known as dezincification and this tendency increases with increasing zinc content of the brass [12, 13]. During the past decade, many techniques have been used to minimize the dezincification and corrosion of brasses.

One of the most important and practical methods in the corrosion protection of metals is the use of organic inhibitors to protect the metal surface from the corrosion environment, especially in aggressive media [14, 15].

Most of the inhibitors functional in industries are the organic compounds which contain hetero atoms such as oxygen, sulfur, nitrogen etc. These hetero atoms play an important role in inhibition due to the availability of free electron pairs. Compounds that contain π - bonds generally exhibit good inhibitive effects via supplying the electrons through π -orbital. When both the effects combine, enhances the inhibition significantly by increasing the adsorption behavior [16-18].

Due to the currently imposed environmental requirements for eco-friendly corrosion inhibitors, there is a growing interest in the use of natural products such as leaves, seeds or bark extracts. Some papers have reported the use of natural products in the development of effective green corrosion inhibitors for different metals in various environments [19-23].

Aim of present

The purpose of the present work is to prepare and use new natural and locally available material as corrosion inhibitors, and study their inhibition effects on the corrosion rate of brass used in heat exchanger by using potentiostat technique to get Tafel extrapolation method at different temperatures and different inhibitors concentrations.

EXPERIMENTAL PROCEDURE

Material

26.07

72.8

The applied samples in this research are prepared from Brass which is used as heat exchanger with the following dimensions (20 mm*15mm*1 mm).

Analysis of these materials was carried out using (spark technique) by the Specialized Institute of Engineering Industries as shown in tables (1)

 Cu
 Zn
 Pb %
 Sn %
 Fe %
 Al %
 C %
 Ti %
 Cu

0.0085

 Table (1) Composition of the studied Brass samples.

Propertion	of Horsetail herb as (Creen inhibitor
Preparation	of Horselan nerd as u	treen innibilor

0.00022

Horsetail herb is prepared below to use as Green inhibitor:

0.0059

A. An amount of the dried horse tail herb was crushed in an electric mixer then sieved to have only powdered horse tail herb.

0.0082

0.0019

B. Ten grams of the powdered horse tail herb were added to 100 milliliter of ethanol in a reflux condenser for 3 hours then filtered for 30 minutes to obtain 50 milliliters of the extracted horsetail herb.

C. Different concentrations of the extracted horse tail herb have been used (150, 250, 350... 1350 ppm) at different temperatures for Brass.

The Electrolyte

The electrolyte was received from Al-Dura Thermal Electricity used in heat exchanger.

The specification of the solution that used in the research was carried out using {Conductivity meter type Hach Sension 7; pH meter type WTW and turbidity meter type Hach 20100 N} by Venous Laboratory Scientific as given in Table (2).

Type of test	Units	Result		
pН		7.81		
Conductivity	µs/Cm	750		
Total Dissolve salt (T.D.S)	ppm	470		
Total Hardens (T.H)	ppm	320		
Calcium	ppm	75.2		
Magnesium	ppm	31.68		
M. Alkalinity	ppm	98		
NaCl	ppm	152		

Table (2) water analysis.

Mechanical Pre-treatment

- **1.** Raw materials of brass were received in the form of tube. This tube was cut into small sample in dimensions of (20 mm*15mm*1 mm) and their sharp edges were then chamfered via grinding wheel and then the samples heat treated in furnace (at 523 k for 30 min, Italy) to relieving stress.
- **2.** Sample was abraded in sequence on 100, 220, 320, 500, 1000, grades of emery papers under running tap water on a hand grinder then washed with tap water followed by distilled water, dried with a clean tissue, degreased by immersing them in acetone then dried with tissue paper and kept in a dissector over a silica gel bed.
- **3.** The specimens were then polished (Dp-Heavy Diamond, Dp-Lubricant for metallographic grinding and polishing, Denmark).

Polarization Technique

A corrosion cell consists of the working electrode which is brass sample placed on a holder; this holder has a hole of 1cm² areas, [Figure (1)] to make sure that only a sample surface area is being exposed to the solution. The reference electrode used in this study was saturated calomel electrode (SCE) bridged by a Laggin-Haber probe; it was fixed about (1 mm) apart from the surface of the working electrode that was exposed to the solution to minimize the experimental error due to IR drop. The auxiliary electrode used in the electrochemical cell was platinum type seated directly opposite to the working electrode, the polarization was carried out in a 1-liter beaker consisting of working, counter and reference electrodes, placed on a mental heater (type Heidolph, MR Hei Standard \ W-Germany) in order to heat the solution to the required temperature. Constant potentials (anodic or cathodic) were imposed on the working electrode specimen, by using the potentiostat (LIDA INSTRUMENT- Germany). This potentiostat is able to induce constant potentials ranging from $(\pm 1V)$ which is the potential of the standard calomel electrode (SCE). The measurement potential range study is from -280 to 200 mv. The potential difference between the working and the reference electrodes (WE – RE) and any current passing in the circuit of working electrode and the auxiliary electrode can be measured by using the SCI computer software.



Figure (1) Schematic diagram of corrosion cell used in polarization

Results and Discussion

Polarization Measurement of Brass Using Horse Tail Herbs (HTH)

Figures (2 and 3 to 7) show the potentiodynamic polarization curves of Brass in industrial water in absence and presence of hours tail herb containing (1350, 450, 1050) ppm at (298, 308, 318) k. Corrosion current (icorr) was found to decrease with an increase in inhibitor concentration indicating, the increased inhibition efficiency with the increase in the concentration of the inhibitor. In acidic solution, the anodic process of corrosion is the passage of metal ions from the solid metal to the solution, and the principal cathodic process is the discharge of hydrogen gas or reduction of oxygen. The observed inhibitor action indicates that the inhibition efficiency increases with an increase in HTH concentration and exhibits both cathodic and anodic inhibition through adsorption on the copper alloys surface blocking active sites. There is no definite charge observed in the corrosion potential (E_{corr}) . According to Riggs [24] and others [25] if displacement in E curves (i) is > 85 mV with respect to E, the inhibitor can be seen as a cathodic or anodic type, (ii) if displacement in E is < 85 mV, the inhibitor can be seen as mixed type. In our study the maximum displacement is less than 85 mV, which indicates that HTH is a mixed type inhibitor.

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Figure (2) Polarization curve of Brass in uninhibited solution at 298k.



Figure (3) Polarization curve of BRASS in uninhibited solution at 308k.



Figure (4) Polarization curve of BRASS in uninhibited solution at 318k.

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Figure (5) Polarization curve of Brass in solution containing 1350ppm at 298k.



Figure (6) Polarization curve of Brassin solution containing 450ppm at 308k.



Figure (7) Polarization curve of Brassin solution containing 1050ppm at 308k.

Tables (3) and (4) below show corrosion potential and corrosion current density for uninhibited and all inhibited solutions; these values have been obtained from polarization curves under different conditions.

These tables show that the corrosion current density (icorr) values increase with increasing temperature, while the corrosion potential (Ecorr) values also increase with temperature. The increase of Ecorr and icorr seems to be related with the increase in both, cathodic and anodic current densities with temperature. The increase in cathodic branch has been attributed to the enhancement of the cathodic reaction with temperature and the effect of temperature in this solution is greater due to the presence of aggressive ions, as a result, these ions affect the anodic branch.

Nurul Izni Kairi [27] studied the effected of the Curcuma longa extract on mild steel surface in 1 M HCl at different temperatures (30-55 °C) by gravimetric measurements.

Concentration of inhibitor ppm	Temp. k	Corrosion Potential Ecorr (mV)	Corrosion Current Density icorr (μΑ/cm ²)	βc (mV/Dec)	βa (mV/Dec)
		0.7.0	1.60		0.11
Without inhibitor		-95.8	4.68	-118.5	3.41
150		-93.1	3.02	-87.1	2.85
250		-90.3	2.19	-79.1	2.07
350		-95.5	1.21	-62.9	1.127
450		-80.5	0.95	-91.4	0.895
550		-88.1	0.62	-82.4	0.579
650	298	-83.7	0.73	-99.8	0.69
750	270	-84.8	0.64	-97.1	0.606
850		-72.6	0.61	-99.3	0.5729
950		-80.5	0.51	-92.3	0.4777
1050		-76.3	0.50	-93.1	0.471
1150		-73.4	0.374	-88.5	0.3456
1250		-73.6	0.372	-90.4	0.3437
1350		-64.9	0.371	-93.5	0.3428
Without inhibitor		-112.3	6.07	-152.7	70.5
150		-91.4	3.59	-95.5	87.8
250	200	-90	2.38	-74.9	60.3
350	308	-76.5	1.64	-121.1	82.1
450		-78.4	0.93	-35.5	49.8
550		-79.3	2.4	-118.6	86.6
Without inhibitor		-124.4	17.42	-183.2	104.2
150		-111.7	9.17	83.8	76.4
250		-112.3	7.50	-81.8	77.7
350	318	-110.3	5.72	-80.4	87.3
450		-108.5	3.72	-84	87.4
550		-114	3.33	-70.3	86.7
650		-98.8	2.94	-83.1	90.1
750		-104.7	2.19	-80.2	97.9
850		-117.8	2.29	-105.3	98
950		-98.8	1.73	-77.2	90.7
1050		-109.3	1.53	-71.9	96.7
1150		-122	1.64	-77.9	108.3

Table (3) Corrosion current density and Potential of Brass.

Inhibitor Efficiency

The inhibition efficiency was calculated, using the following equation:

IE (%) = $[(i_{corr} - i_{corr(inh)}) / i_{corr}] * 100$ (1)

where i_{corr} and $i_{corr (inh)}$ are the corrosion current density values in the absence and presence of extracted HTH, respectively.

The results of the inhibitor efficiency obtained by calculations are presented in Table (4). The best result obtained for Brass was provided by extracted HTH at 1150 ppm concentration with efficiency at 92.1% at 298k, and 85% at 308k, and 91.21% at 318k.

In general, the effective corrosion inhibition performance of extracted horse tail herb could be associated with textured silica and silicates, which account for about 2-3% of the elements silicon, potassium, aluminum, and manganese. Extracted HTH constituents such as 15 different composite of bioflavonoid compounds could be, or act as inhibiting passive film formers on the copper alloy substrate surface. The formed film would act as a barrier between the copper alloy and corrosive environment interface thus preventing and/or stifling corrosion reactions of anodic oxidation/ dissolution and cathodic reduction processes.

Concentration of inhibitor ppm	Temp. k	Corrosion Current Density icorr $($	Inhibitor efficiency %	CR mpy
Without inhibitor	_	μΑ/cm) 4.68		4.5
150	-	3.02	35.47	2.9
250		2.19	53.32	2.1
350	_	1.21	74.14	1.17
450		0.95	79.7	0.92
550		0.62	86.75	0.6
650		0.73	84.4	0.7
750	298	0.64	86.32	0.62
850		0.61	87	0.59
950		0.51	89.1	0.49
1050		0.50	89.31	0.48
1150		0.374	92	0.363
1250		0.372	92.05	0.361
1350		0.371	92.07	0.36
Without inhibitor		6.07		5.89
150		3.59	41	3.48
250	- 308	2.38	61	2.31
350		1.64	73	1.59
450		0.93	85	0.9
550		2.4	60	2.33
Without inhibitor		17.42		16.8
150		9.17	47.359	8.9
250		7.50	56.95	7.28
350		5.72	67.169	5.55
450		3.72	78.649	3.61
550	318	3.33	80.88	3.23
650		2.94	83.122	2.85
750	1	2.19	87.433	2.12
850		2.29	86.855	2.22
950		1.73	90	1.68
1050		1.53	91.21	1.48
1150		1.64	90.5	1.59

Table (4) Corrosion current density and potential of Brass at different temperature.

Effect of Concentration on Inhibitor Efficiency

Table (4) depicts the variation in inhibition efficiency (IE) with respect to concentration of the inhibitor. It can be inferred that as the dosage of inhibitor increased, the IE was found to increase. This behavior can be attributed to the increase in surface area covered by the adsorbed molecules of the extract with increased concentration. A maximum of 92.1% was obtained at 1350 ppm concentration of the extract of inhibitor for brass at 298k.



Figure (8) The relationship between inhibitor concentration and inhibitor efficiency at different temperature with addition of inhibitor to brass.

Effect of Concentration on Corrosion Rate

Corrosion Rate (CR) was calculating from the following equation:

$$CR (mpy) = \frac{0.13 \times EW \times I_{corr}}{d} \qquad \dots \dots (2)$$

Figure (9) show the variation in corrosion rate versus inhibitor concentration. The effect of addition of HTH at different concentrations on the corrosion of copper alloy in industrial water was investigated using polarization technique at (298, 308, 318) k. The corrosion rates of the Brass in industrial water with and without different concentrations of inhibitor were determined. The results obtained are presented in Figure (9), the corrosion rate decreases as the concentration of extracted HTH increases (up to approximately 1350 ppm). This behavior is attributed to higher adsorption level of active inhibitor molecules from the extract on the metal surface which acts as corrosion protection.

Figure (9) shows the corrosion rate for Brass specimens immersed in industrial water at different temperatures. The results show that the corrosion rate decreases as the concentration of extracted HTH increases (up to approximately 1350 ppm at 298 k, 450ppm at 308 k and 1050 ppm at 318 k). This behavior is attributed to higher adsorption level of active inhibitor molecules from the extract on the Brass surface. For extracted HTH concentrations higher than 1150 ppm at 298k, the corrosion rate remained approximately constant. This could be an indication that the 1350 ppm at 298 k concentration is the best inhibitor concentration. Higher extract

10 9 8 **Corrosion Rate mpy** 7 6 298k 5 4 308k 3 318k 2 1 0 0 500 1000 1500 concentration of inhibitor ppm

concentrations did not show any additional significant protection under these particular laboratory test conditions.

Figure (9) The variation in corrosion rate versus inhibitor concentration for Brass.

Effect of Temperature on Corrosion Rate and Inhibition Efficiency IE (%)

Table (4) illustrates the variation in IE (%) and corrosion rate (CR) with HTF concentration at different temperatures in industrial water. The obtained data in Table (4) reveal that in industrial water, the corrosion rate increases rapidly in absence of HTH, on addition of HTH and the corrosion rate rapidly decreases. This suggests that the inhibitor species are adsorbed on the Brass/solution interface where the adsorbed species mechanically screen the coated part of the metal surface from the action of the corrosive medium.

The effect of temperature on the inhibition efficiency of the studied inhibitor at all concentrations and temperatures shows two behavior aspects depending on the type of the studied acid solution as follows:

- 1. In industrial solutions, an increase or decrease in the inhibitor efficiency depending on its concentration was detected with increasing temperature indicating that adsorption of inhibitor species on copper alloy surface at these conditions is not merely physical or chemical adsorption but obeying a comprehensive adsorption (physical and chemical adsorption).
- 2. In industrial solutions, remarkable increase in the inhibitor efficiency was observed with increasing temperature up to 318 k. This increasing efficiency with increase in temperature suggests chemical adsorption mechanism which is effectively enhanced with rising temperature.

The effect of temperature on the inhibited acid-metal reaction is highly complex, because many changes occur on the metal surface such as rapid etching and desorption of inhibitor and the inhibitor itself may undergo decomposition and/or rearrangement.

Activation Energy

The apparent activation energy (Ea) of metal corrosion in acid media can be calculated from the Arrhenius equation.

 $\ln C_R = Ea/RT + A$

.....(3)

where Ea is the apparent activation energy for the corrosion of copper alloys, R is the universal gas constant, A is Arrhenius pre exponential factor and T is the absolute temperature. The values of Ea are obtained from the slope of the log C_R vs 1/T plot shown in Figures (10). The calculated activation energies, (Ea) at different concentrations of the inhibitor are presented in Table (5).

These values indicate that the presence of the inhibitor decreases the activation energy for the corrosion process. The decrease in activation energy indicating a strong adsorption of the inhibitor molecules on the copper alloys surface signifies that HTH is a powerful inhibitor. Smaller value of Ea in presence of HTH is an indication of 'chemisorption' mode of adsorption

 Table (5) Activation Energy.

Inhibitor Concentration (PPM)	Slope	Ea (KJ/mol)
No Inhibitor	0.775	14.839
150	0.560	10.722
250	0.525	10.1
350	0.682	13.1
450	0.474	9
550	0.996	19.1



Figure (10) Arrhenius plot for Brass in solution containing different concentration of HTH.

The relationship between the temperature, percentage inhibition efficiency (IE %) of an inhibitor and the activation energy in the presence of an inhibitor is given as follows [28].

- For inhibitor whose IE (%) decreases with temperature increase; the value of activation energy (Ea) found is greater than that in the inhibited solution.
- For inhibitor whose IE (%) does not change with temperature variation; keeps the activation energy (Ea) unchanged in the presence or absence of inhibitors.
- For inhibitors whose IE (%) increases with temperature increase; the value of activation energy (Ea) found is less than that in the uninhibited solution.

The experimental fact that the inhibitor efficiency increases with temperature and the activation energy is lower in the presence of inhibitor is explained in different ways in literature. According to Putilova [29] at higher temperature, the surface covered by inhibitor increases and rate determining step of the metal dissolution becomes the diffusion through the film of corrosion products and inhibitor.

Adsorption and Thermodynamic Considerations

The inhibitive action of organic components in plant extracts is attributed to their adsorption on the metal surface. The adsorbed layer acts as a barrier for mass and charge transfer leading to a reduction in the rate of corrosion of the metal. The fraction of the surface covered by the adsorbed molecules θ is an indication of the effectiveness of the adsorbed species and is directly proportional to the inhibition efficiency. The surface coverage θ of the metal surface by the adsorbed inhibitor is calculated [30]

$$\theta = \left[\left(i_{\text{corr}} - i_{\text{corr}(\text{inh})} \right) / i_{\text{corr}} \right] \qquad \dots \dots \dots (4)$$

Adsorption isotherms that describe the behavior of adsorbed species examine the variation in θ with concentration of the organic species. The obtained θ values were applied to different adsorption isotherm equations and the Langmuir adsorption isotherm fitted best. The Langmuir adsorption isotherm is given as:

 $C/\theta = 1/K + C$ (5) where C is the inhibitor concentration, K is the equilibrium constant and θ as earlier defined above.

The variations in surface coverage with concentration of extracted HTH are shown in Figures (11). These curves represent adsorption isotherm that are characterized by sharp rising part, indicating formation of an adsorbed layer on the copper alloy surface.

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Figure (11) The variations in surface coverage, obtained from polarization data, with concentration of extracted HTH for Brass.

Table (6) The adsorbed molecules,	inhibitor efficiency an	d degree of surface
converge of Brass		

Inhibitor efficiency %	Temp. k	Coverage 0	С	С/ Ө
35.47		0.35	150	428.57
53.32		0.53	250	471.6
74.14		0.74	350	472.9
79.7		0.79	450	569.6
86.75		0.86	550	639.5
84.4		0.84	650	773.8
86.32	298	0.86	750	872.1
87		0.87	850	977
89.1		0.89	950	1067
89.31		0.893	1050	1176
92.1		0.92	1150	1250
92.1		0.92	1250	1358.6
92.1		0.92	1350	1467.4
41		0.41	150	365
61		0.61	250	409
73	308	0.73	350	479
85		0.83	450	542
60		0.6	550	916
47.359		0.47	150	319
56.95		0.56	250	446
67.169		0.67	350	522
78.649	318	0.78	450	576
80.88		0.8	550	687.5
83.122		0.83	650	783
87.433		0.87	750	862
86.855		0.86	850	988
90	1	0.9	950	1055
91.21		0.91	1050	1153
90.5		0.9	1150	1277

The plots of C_{inh}/θ vs. C_{inh} yield a straight line in Figure (12) this confirms that these inhibitors (plant extracts) obeys Langmuir adsorption isotherm at corrosive medium. It indicates that the adsorbing HTH species occupies typical adsorption site at the metal/solution interface.



Figure (12) Langmuir adsorption plots and different concentrations of extracted HTH for Brass

Scanning Electron Microscopic (SEM) Analysis

The SEM micrographs of the corroded and protected copper alloy specimen are shown in Figures (13 and 14). It is clear from the micrograph shown in Figure (13) that the surface of the copper alloy is extremely corroded in industrial water whereas in Figure 3-13 there is a disappearance of corroded and protected area owing to the formation of thick layer of adsorbed inhibitor on the copper alloy.



Figure (13) Scanning electron microscope image of Brass in industrial water without inhibitor.



Figure (14) Scanning electron microscope image with magnification 4.97 kx of Brass in industrial water after addition inhibitor

Energy Dispersive X-ray Analysis (EDX)

The composition of protective film formed on copper alloy surface was analyzed using EDX as shown in Figure (15). The EDX spectrum of polished copper alloy specimen shows an original copper surface. By adding HTH the decrease in copper band and appearance of carbon, oxygen and nitrogen band were observed due to the formation of a strong protective film of the inhibitor molecules on the surface of copper alloy specimen. The action of inhibitor is related to adsorption and formation of a barrier film on the electrode surface. The formation of such a barrier film is confirmed by EDX examinations of the electrode surface. Figure (16) indicates the EDX spectra show an additional line characteristic of the existence of C (due to the carbon atoms of HTH). In addition, the O signal is significantly enhanced due to the oxygen atoms of the head group. These data show that a carbonaceous material containing oxygen atoms has covered the electrode surface. This layer is undoubtedly due to the inhibitor, because the carbon signal and all the previous results plead on the favors that the corrosion inhibition effect of the HTH is due to their adsorption onto the electrode surface blocking the corrosion process. It is well known that the adsorption of organic compounds can be described by two main types of interaction: physical adsorption and chemisorption. In general, the proceeding of physical adsorption requires the presence of both electrically charged surface of the metal and charged species in the bulk of the solution. Chemisorption process involves charge sharing or charge-transfer from the inhibitor molecules to the metal surface to form a coordinate type of bond. This is possible in case of a positive as well as a negative charge of the surface.

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Figure (16) Copper alloy surface by EDX after add HTH.

CONCLUSION

- 1. The corrosion of Brass in industrial water increases with an increase in temperature, and decreases with an increase in inhibitor concentration.
- 2. Potentiodynamic polarization techniques demonstrate that inhibition efficiency of Brass in industrial water increases as the concentration of extracted HTH increases to reach best concentration. Inhibition efficiency is 92.1 % in added 1350 ppm.
- 3. Adsorption of inhibitor molecules on the Brass surface is found to follow the Langmuir adsorption isotherm at all test temperatures.
- 4. The inhibition efficiency of HTH is dependent on temperature, increases with temperature increases.

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