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**A Review of Inhibitors for the Corrosion of Transition Metals in Aqueous Acids**

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**Abstract**

The importance of the electrical double layer between a metal and an acid electrolyte and the interaction with organic and inorganic molecules to produce initially electrostatic adsorption are highlighted. In some cases, a chemical bond is formed involving charge transfer or charge sharing between the metal surface and inhibitor molecules forming a coordinate bond through lone-pair electrons on heteroatoms or electrons on inhibitors with multiple and aromatic bonds. The application of mathematical formulae to the variation in adsorbed inhibitor molecules at the metal surface is considered, with inhibitor concentration isotherms considering thermodynamic principles or the water displacement reaction where for an inhibitor molecule to adsorb at a metal surface several water molecules must be displaced first. The predominant ways in which molecules enable inhibition are formation of a *physical barrier* where a physical adsorbed barrier of molecules (usually polymeric or oxide promoting for this mode to predominant) impede movement near the metal surface or *reduction in metal reactivity* where chemisorbed inhibitor molecules adhere to active sites on the metals reducing the number of cathodic and anodic sites. Adsorption involving charged inhibitor species causes a *change in the double layer* and a change potential at the outer Helmholtz plane influencing the corrosion rates of both anodic and cathodic reactions. The first three modes are intimately with adsorption and the double layer the last involves *interaction* *of the inhibitor molecules* and the intermediate products formed during the *partial electrochemical reactions*, interaction of the adsorbed intermediates with organic molecules can either decrease (inhibit) or increase (stimulate) electrode reaction rate depending on the stability of the inhibitor-intermediate complex formed.

**Keywords**: Adsorption; hydrogen evolution, LPR; measurement; mechanism; metal dissolution; Tafel extrapolation.

**List of Symbols**

***Symbol Meaning Units***

*A* Electrode area cm2

*b* Tafel slope V decade-1

*c* Concentration mol dm-3

*Ecor* Corrosion potential V

*F* Faraday constant C mol-1

*I* Current A

*Icor* Corrosion current A

*jcor* Corrosion current density A cm-2

*Ecor* Corrosion potential V

*Eq* Equilibrium potential V

*φ* Surface charge C m-3

*γ* Electrosorption valency -

*λ* Stoichiometric electron number -

*SA* Adsorbate -

*M* Adsorbent -

*(δμs/δE)* Electrosorption equilibrium -

*(δqm/δΓad)* Charge flow C s-1

*Γad* Surface concentration of adsorbed species mol cm-2

*µs* Chemical potential V

*ZP* Area impedance of P Ω cm2

*Y0* Coefficient relating to the surface and electroactive species mol cm-2

*ω* Angular frequency rad s-1

*h*  A measure of the heterogeneity of the surface -

*τ* Inhibition efficiency -

*χ* Electronegativity -

 Gibbs free change of adsorption kJ mol-1

*K* Equilibrium constant for the adsorption process M-1

*R* Molar gas constant J mol-1 K-1

*T* Temperature K

*c*  Inhibitor concentration mol dm-3

*θ* Surface coverage -

*p* and *a* Constants characteristic of each system in Equation (8) -

*s* Temkin heterogeneity factor -

*f(θ,x)* Configuration factor that depends the physical model and assumptions underlying the derivation of the isotherm -

*Ψ* Change in potential at the outer Helmholtz plane V

*Γ* Theoretical inhibition coefficient -

**Abbreviations**

EHDI Electrohydrodynamic impedance

HSAB Hard and soft acid and bases

ZCP Zero charge potential

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*(Approx. 13,000 words, 110 refs, 9 tables and 21 figures).*

**1. Introduction**

This review summarises the fundamentals of corrosion inhibition (of transition metals) in acid electrolytes. Starting with a brief historical prospective of corrosion inhibition showing that the earliest corrosion inhibitors were based on natural products until the first synthesis of a compound for the inhibition corrosion. The development of corrosion inhibition is also considered together with the first recognition of adsorption as a corrosion inhibition process and the correlation of inhibitor molecular structure on corrosion inhibition.

The electrical double layer is described under positive and negative charge and at the zero charge potential. The variation in double layer capacitance with changes in inhibitor concentration and acid concentration are also shown.

Adsorption is recognized as the first step in inhibition. The factors affecting the behaviour of inhibitor molecules undergoing the two types of adsorption; physical adsorption and chemisorption are reviewed. Several examples of the various adsorption isotherms that are used to ascribe the manner of adsorption by a mathematical formula are considered. Molecular interaction of the adsorbed layer and heterogeneity factors or the water displacement reaction, are given. The four modes of inhibition; formation of a physical barrier, reduction of metal reactivity, changes in the electrical double layer and participation of the inhibitor molecule in partial electrochemical reactions are explained, examples being provided, to illustrate trends.

This review illustrates the major experimental techniques used to quantify corrosion inhibition and to study its mechanism. In addition to weight loss, solution analysis for metal concentration and off gases for rate of hydrogen evolution, electrochemical techniques have included Tafel analysis of polarisation curves, linear polarisation resistance, electrochemical impedance spectroscopy and electrochemical noise analysis.

## **2. An historical perspective of corrosion inhibition**

The term ‘inhibitor’ is taken from Latin, the ‘inhibere’ meaning ‘to check’ and corrodere meaning ‘to gnaw away’. Inhibitors impede corrosion processes and lower corrosion rates; in some cases, they almost completely prevent the dissolution of metals in corrosive media. Corrosion inhibitors for ferrous metals in acid conditions have been known as far back as the Middle Ages. Master-armourers added a variety of organic products, such as flour, yeast, bran, etc., to control the attack of bare metal and prevent the appearance known as pickling brittleness when removing scale by pickling, i.e., cleaning of metallic surfaces by removal of oxide(s) and soils (such as in the surface pretreatment or acid etching) from metal articles [1].

In 1900, two US patents were granted to Robinson and Sutherland, the first [2] involved the use of a liquor produced from bran middlings, wheat, or starchy materials for the pickling of metal bars, plates, sheets, etc. The second [3] also involved the use of a natural product namely wheat-bran middlings for the inhibition of sulphuric acid ("to cleanse the plate of all grease and enable the acid to remove oxide more readily"). In 1907, Laverty and Laverty [4] proposed the use of hydrocarbons such as tar sludge (a waste product from oil refining containing tars and oils, thus being preferable, due to cheapness) in a sulphuric acid pickling baths for the removal of scale or oxide. This produced articles ready for galvanising or tinning without appreciable attack of the metal surface [4]. Figure 1 shows the development of corrosion inhibitors.

Cushman [5] (1909) described the use of a train of bottles for the testing of dry pigments on steel sheet, with a constant air stream. Two test steel sheet specimens were placed in bottles containing distilled water and dry pigments and the corrosion rate measured by weight loss. A blank was used to show whether corrosion was inhibited, simulated or unaffected by the presence of pigment.

In 1928, work commenced in the Soviet Union on the creation of synthetic corrosion inhibitors. This led to the manufacture of ‘***Antra***’, a product of the sulphonation of anthracene or anthracene oil, developed by the Karpov institute [6]. One of the earliest advances in corrosion inhibition science was the recognition of adsorption in 1936. Mann *et al.* [7] demonstrated that the formation of a protecting layer of inhibitor is formed by adsorption by plotting the logarithm of the percentage of reduction in corrosion against the logarithm of inhibitor in terms of nitrogen for inhibition of mild steel in sulphuric acid by aliphatic amines.

A 1958 paper by Kaesche and Hackerman [8] was one of the first to mention the effect of molecular structure on corrosion inhibition. These authors studied the inhibition of iron corrosion in 1 M HCl by aniline and several of its derivatives and some alkylamines, using cathodic polarisation measurements and colorimetric analysis (see Table 1). Kaesche and Hackerman [8] observed that an increase in chain length of the *N*-substituted anilines resulted in an increase in inhibition. It was assumed that the inhibitor molecule adsorbed perpendicular to the metal surface and an increase in the inhibition was attributed to an increase in projected area as the chain length increased. A large difference between the inhibition of the two toluidine compounds can also be noted.

One of the first literature citations of phosphonium salts as acid corrosion inhibitors was by Horner *et al.* [9] in 1963, who observed the connection between catalysis poisoning of Raney nickel in hydrogenation reactions and corrosion inhibition. Phosphorus-containing compounds showed good inhibition for iron corrosion, especially phosphonium salts, diphenyldithiophosphonic acids and, less effectively, phenylphosphonic acid. It was concluded that reduction products were responsible for inhibition by phosphonium salts. Inhibition was tested on several mineral acids and the protective action for hydrochloric and sulphuric acids was found to be similar but poor in phosphoric acid and almost non-existent in nitric acid. Organoarsenic compounds had similar behaviour but were not quite as effective. Sulphonium salts were found to be highly protective.

In 1970, Redmore applied to the USA patent office [10] for a patent on the use of ‘Phosphonium compounds and/or ethylene glycol-bis-(trialkylphosphonium acetates)’ in the prevention of corrosion of iron, steel and ferrous alloys as pipe and equipment material which comes in contact with corrosive oil-containing media (and any other system or application). Ten other possible applications such as demulsifying agents, biocide, flocculants and lube oil additives were mentioned showing the surfactant nature of the materials mentioned. This patent was followed by three more in the 1970's ‘Polyalkyleneethers having dangling phosphonium groups’ [11], ‘Use of phosphonium compounds as corrosion inhibitors’ [12] and ‘Phosphonium compounds’ [13]. These patents also followed the same theme for applications. Redmore [14] produced another in 1984, ‘Phosphonium salt corrosion inhibitors for high density brines’, describing the use of phosphonium salts for deep, high-pressure wells where zinc halides and high temperatures produce a highly corrosive mixture.

Corrosion inhibitors for transition metals in acidic media have been intensively studied. Imidazolium salts were used by several authors [15-19] as environmentally friendly, highly effective inhibitor compound that can provide a protective action in situations ranging from the marine environment to oil and gas pipelines. 1,​3-​thiazole and its amino derivatives such as 2-​amine-​1,​3-​thiazole​, and 4-​amine-​1,​3-​thiazole [20] showed good inhibition property. Other authors have used in their studies ammonium salts [21-30], pyrazolone derivative [31], thiophene methanol [32], poly(sodium 2-​acrylamido-​2-​methylpropane sulfonate-​co-​styrene)​/magnetite nanoparticle composites [33], 5-​chlorobenzotriazole and its mixture with sodium fluphenaminate [34], 1,​3-​diketone malonates [35], acenaphtho quinoxaline [36], bis(benzimidazol-​2-​yl) disulphide [37], α-​amino acids alkylamides [38], fatty acid oxadiazoles [39], poly(aminoquinone) [40], thiourea and its derivatives, such as phenylthiourea, o-​tolylphenyl thiourea, p-​chlorophenyl thiourea and o-​nitrophenyl thiourea [41], 2-​mercaptothiazoline and cetyl pyridinium chloride [42], polyvinylpyrrolidone and polyethylenimine [43].

## **3. Adsorption**

It is generally assumed that the first stage of the acid inhibition mechanism is adsorption. Adsorption is influenced by the nature and surface charge of the metal, by the chemical structure of the inhibitor molecule and by the type of aggressive media [44]. The adsorption by organic inhibitor molecules may be considered as a displacement of water molecules:

 **** (1)

where *x* is the number of water molecules displaced per inhibitor molecule adsorbed.

### 3.1 Electrostatic (physical) adsorption

Electrostatic adsorption is the result of electrostatic attraction between the inhibitor molecules and the electrical charge at the metal surface. The surface charge of the metal develops via the electric field at the outer Helmholtz plane of the electrical double layer existing at the metal-solution interface. The surface charge can be defined by the potential of the metal (*Ecor*) versus its zero charge potential (ZCP), *Eq* = 0 [1], see equation (2). When a metal immersed in an electrolyte, an excess of either cations or anions will form in the double layer, leading to a surface charge. ZCP is the potential of the metal when no excess charge of either species is present.

 (2)

When the difference between *Ecor* and *Eq* = 0 is negative, φ is negative and cationic adsorption is favoured (as illustrated by Figure 2a). Anionic adsorption occurs if *ϕ* is positive, as illustrated in Figure 2b). Electrostatic adsorption can also occur with molecules without formal positive and negative charge, by dipoles.

In hydrochloric acid, aniline forms anilinium and chloride ions. Banerjee and Malhotra [45], who studied corrosion of mild steel in hydrochloric acid at 30 °C inhibited by aniline, suggested that the inhibitor was adsorbed in three different ways depending on the value of surface charge.

When the surface is polarised positively (Figure 3), chloride ions will adsorb first then anilinium ions and protonated water. These adsorbed layers will inhibit the movement of metal ions into solution and an increase in adsorbed aniline observed (Figure 4). When the surface is polarised negatively (Figure 3b), anilinium ions and protonated water adsorbs directly on the metal surface with a subsequent increase in aniline adsorption. At the ZCP, no ions adsorb through ionic charge; a few aniline molecules may adsorb through pπ-dπ bonding of the vacant metal orbitals and aromatic inhibitor electrons. Adsorption of aniline will be smaller than in the two previous cases due to the lower density of packing of molecules planar to the surface [45]. Antropov [46] postulated that metals with equal values of *φ*, similar behaviour of a given inhibitor should be expected in the same environment. Schultze and Koppitz [47] studied partial charge by the concept of electrosorption valency, γ, where a charge transfer of *z* is possible between the adsorbate, *SA* and adsorbent, *M*. They proposed an electrosorption with the desorption of x water molecules.

x M—OH2 + SA(aq)  M—SAz+ + ze- + *x*H2O(aq) (3)

 (4)

Schultze and Koppitz [47] derived an equation for electrosorption valency in equation (4), showing that it is dependent on the electrosorption equilibrium, *(δμs/δE)* and charge flow, *(δqm/δΓad)*, where *Γad* is the surface concentration of adsorbed species (mol cm-2) and *μs* is the chemical potential.

Inhibited solutions contain anions such as halides can absorb on the metal surface by creating oriented dipoles and subsequently allow the adsorption of cations on the dipoles. This results in a synergistic effect. Frignani *et al.* [48] postulated that this could explain the differences in inhibition efficiency of various pyridinium organic inhibitors between hydrochloric and sulphuric acid solutions at 25, 50, and 75 °C.

Electrostatic adsorption is a rapid process but is also highly reversible [49] and has a low activation energy. Hackerman and Hurd [49] noted that electrostatic adsorption is relatively independent of temperature. However, it is common to find activation energies of < 40 kJ mol-1 attributed to diffusion processes of electrosorption valency. Electrostatic adsorption seems to be dependent on the electrical characteristics of the organic inhibitor, the surface charge and the type of adsorbate anions present in the aggressive media.

Foroulis [50] investigated the importance of molecular configuration on electrostatic adsorption and by the examination of adsorption of benzoic acid. They found that the resonance structures were important. This allows charged structures from an original neutral molecule (see Figure 5).

Growcock [51] studying corrosion inhibition of steel in hydrochloric acid (15 and 28%) by 1-octyn-3-ol, CH3(CH2)4CH(OH)C≡H, by impedance spectroscopy described the impedance of *P*, *ZP* has the form:

 (5)

*Y0* is a coefficient relating to the surface and electroactive species, *ω* is the angular frequency (in rad s-1), and *h* is a measure of the heterogeneity of the surface, *h* = 1 corresponding to a homogeneous surface.

### 3.2 Chemisorption

Chemisorption is a process involving charge transfer or charge sharing between the metal surface and inhibitor molecules forming a coordinate bond. Chemisorption is slower than electrostatic adsorption and has a higher activation energy [49]. A greater degree of adsorption should be expected at evaluated temperatures. Chemisorption is not completely reversible. Electron transfer is typical for transition metals with vacant low energy-electron orbitals and inhibitors with loosely bound electrons such as those present in inhibitors with multiple and aromatic bonds, with π electrons. Heteroatoms with lone-pair electrons such as N, P, S, Se, etc., facilitate electron transfer. The strength of adsorption for a heteroatom is related to the electron density and polarisability of the functional group.

Hackerman and Hurd [49] postulated that chemisorption between surface atoms and inhibitor molecules can be considered in terms of Lewis acid-base theory, involving primarily electron availability and orbital character. In most cases, the inhibitor acts as the electron donor and the metal as the electron acceptor. Matsen *et al.* [52] applied charge-transfer complex theory to chemisorption; the ground state of the complex is described by a linear combination of a wave function for a no-bond state and that for a dative state in which an electron has been transferred from Lewis acid to a Lewis base. For adsorption on a metal, the metal is the acid, the electron affinity of the acid is set to equal to the work function and the coulomb energy to the image energy.

Aramaki and Nishihara [53] applied the HSAB (hard and soft acids and bases principle) to the adsorption of some 5B and 6B (R2O, R2S, R2Se, R2Te, R3N, R3P and R3Sb) n-propyl derivatives on anodically polarized nickel in 3M HClO4, using AC impedance. The hard and soft acids and bases principle is a term used in coordination chemistry involving the transfer of electrons from a ligand (or lone-pair and π electrons in an inhibitor molecule), known as the *"base"*, to a metal ion (or metal lattice atom), known as the *"acid"*. Small, compact and not very polarizable bases prefer small, compact and not very polarizable acids and are termed *"hard"*. Large and more polarizable bases prefer large and more polarizable acids and are referred to as *"soft"*. Figure 6 illustrates the variation of log [*τ*/(1-*τ*)] with electronegativity, *χ*; the plots of the Te compound deviate from the line due to limited solubility. The double layer capacity measurements showed that the compounds classified as a soft base was more readily adsorbed on the nickel surface acting as a soft acid than that of a hard base, in line with the HSAB principle.

Structural characteristics can influence the electron density on the heteroatom and consequently the strength of the chemisorption bond. Ayers and Hackerman [54] investigated the effect of methyl substitution on the pyridine ring (using 2-, 3-, and 4-picoline inhibition of iron in 6.08 M hydrochloric acid at 35 oC) (Figure 7). The order of inhibition was found to be 4-picoline = 2-picoline > 3-picoline > pyridine.

They postulated [54] that this is attributable to the electron density of the nitrogen atom involved in chemisorption. Substitution at the 2- and 4- positions would donate electron charge through the inductive effect as in a covalent single bond between two dissimilar atoms, the electron pair forming the bond is never shared absolutely equally between the two atoms, it tends to polarise to the more electronegative atom of the two. Most groups attached to carbon exert such *inductive effects*, with the exception of alkyl groups which are electron-donating. Substitution at the 2- and 4- positions would also donate electron charge hyperconjugation because when alkyl groups are attached to unsaturated systems, e.g. double bonded or aromatic, it appears that the alkyl groups are capable of electron release via a mechanism different from the inductive effect; while substitution at the 3 position would cause donation by induction only.

The introduction of a nucleophilic or an electrophilic group at the expense of a hydrogen atom to an inhibitor molecule, would affect electron density and thus inhibitor efficiency. Smialowsky and Kaminsky [55] studied the inhibition of thiophene derivatives and noted an increase in inhibition with both nucleophilic and electrophilic substitutions. This result was interpreted as follows: the introduction of a nucleophilic substituent promotes electron density and enhances chemisorption and the presence of an electrophilic group decreases electron density. A strong polarising group increases the dipole moment of the molecule that also leads to increased adsorption.

Other structural parameters influencing inhibition efficiency include projected molecular area [54], relative molecular mass and molecular configuration [56]. For example, Ayers and Hackerman [54] calculated the projected molecular areas for pyridine and mono, di- and trimethylpyridine in three positions (using Stuart and Briegleb atom models), the ring system vertical (A1), the ring system parallel, either with the smallest possible circle (A2), the cation oriented the same way (A2') or allowing free rotation about the centre of the ring (A3). The results are shown in Table 2‎0. The results for projected molecular area were compared to their inhibitive value and concluded that the parallel orientation gave the best correlation.

Trabanelli and Zucchi [56] studied the influence of relative molecular mass by examining inhibitor efficiency variations in alkyl chain length of some mercaptans and sulphides. They found that inhibition efficiency increased with chain length for the mercaptans. This is due to the increasing inductive effect of the alkyl group producing stronger chemisorption through the sulphur atom. However, no such trend was found with the sulphides, inhibition increased to a maximum at di-*n*-hexyl sulphide and then decreased at longer chain lengths. This was attributed to the screening action of the hydrocarbon chain. Trabanelli and Zucchi [56] also worked on the effect of molecular configuration by comparison of normal, secondary and tertiary butylsulphides and butylmercaptans. In both cases the inhibition decreases with increased branching despite electron density increasing on the sulphur. This was attributed to the steric hindrance effect of the methyl groups screening the sulphur from the metal surface. Tertiary butylsulphide exhibited no inhibition showing complete screening as the sulphur's electrons could not come into close enough proximity to the surface atom to form a chemisorption bond due to complete encompassment of the attached methyl-groups.

The effect of molecular configuration is also shown by the work of Fiaud *et al.* [57, 58] on diphosphines inhibition of zinc and iron in sulphuric acid. Corrosion inhibition was found to follow the order:



 *n* = 3 > 1 > 4 [57]

 *n* = 3 > 2 > 1 > 4 > 6 [58]

Figure 8 shows results obtained by Fiaud, *et al.* [57], illustrating the order shown above. For iron, it was found that no such order existed. However, a slight increase as *n* increased was observed. For zinc, this was contributed to the two different types of adsorption, flat molecular configuration and a chelate (bridge) configuration. At low *n* values, the former configuration is adopted but at higher values, the second type is adopted. It appears that the best diphosphine inhibitor studied is phosphinopropane (*n* = 3) which had the greatest increase in overpotential for the hydrogen evolution reaction. This was attributed to the formation of the most favourable/stable chelating structure. For higher values of *n*, the chelate structure is less favourable. For iron, all *n* values yield a flat configuration explaining the slight increase in τ as *n* increased.

As mentioned previously, π orbitals can interact with metal surface atoms to form chemisorption bonds. The *π* bond emanates from s-p hybridisation, as illustrated in Figure 9a), for aromatic π bond and, Figure 9b, for unsaturated aliphatic π bond. The π bond forms parallel to the plane of *σ* bond.

Foroulis [50] showed an example of *π* interaction with metal atoms in non-aromatic structures by studying the inhibition variation between single, double and triple bonded compounds for a series of alcohols (see Table 3).

Table 3 shows that introducing unsaturation decreases corrosion dramatically, particularly in the case of the introduction of acetylenic groups. The decrease in inhibition by the substitution of two α-hydrogens by alkyl groups in compound 4, was attributed to steric hindrance.

Another example of the effect of unsaturation in corrosion inhibition is given by the work of Fiaud *et al.* [49] on diphosphines inhibition of zinc in 0.05 M sulphuric acid at 25 °C by diphenylphosphinoethylene (Ph3PCH2CH2PPh3), diphenylphosphinoethylene (Ph3PCH=CH-PPh3) and bis-1,2-diphenylphosphinoacetylene (Ph3PC≡CPPh3). The introduction of a double bond had little effect on inhibition efficiency compared to the saturated phosphine (see Table 4) and this was attributed to the adsorption configuration existing with the phenyl ring parallel to the surface and the double bond normal, resulting in minimal interaction between the π electrons and the surface atoms. The introduction of an acetylenic group at low concentration accelerated corrosion although, at higher concentrations and longer immersion times, inhibition was observed. This was attributed to depolarisation of hydrogen evolution by hydrogenation of the triple bond by reduced protons; reduction leading to the more protective double bond species.

### 3.3 Surface Interactions between Inhibitor Molecules

Lateral interactions may arise between inhibitor molecules, surface coverage of the metal increases, thus influencing the inhibition efficiency. Attraction between inhibitor molecules usually generates stronger adsorption and high inhibition efficiency. Repulsive interactions may occur between molecules or ions containing dipoles leading to weaker adsorption and lower inhibition efficiency.

One example of stronger adsorption is shown by Hackerman *et al.* [59] whose work on homopiperazine adsorption on iron in 6 M HCl at 25 °C suggested that a sharp decrease in corrosion current density at an inhibitor concentration of 0.016 mol dm-3 was the result of a change in adsorption (see Figure 10).

At low inhibitor concentrations, homopiperazine was considered to adsorb perpendicular to the surface via chemisorption of one imine group, the other remaining protonated and facing the solution. At high inhibitor concentrations, adsorption was thought to occur via both imine groups (see Figure 10) and adsorption is subsequently stronger. Electrostatic repulsion forces the adsorbed molecules at the higher inhibitor concentrations, to change from the *trans* configuration in vertical adsorption to the less favoured *cis* enabling the second imine group to become close enough to the surface for chemisorption.

### 4. Adsorption isotherms

Interpretation of inhibitor adsorbent behaviour can be enhanced by 'fitting' experimental data to an adsorption isotherm. The first four isotherms (Langmuir, Freundlich, Temkin and Frumkin) considered molecular interaction in the adsorption layer and heterogeneity factors. The last isotherms (Flory-Huggins, Dhar-Flory-Huggins and Bockris-Swindels) considered the water displacement reaction shown in equation (1) and introduced a method for measurement of the number of water molecules displaced by each inhibitor molecule, *x*, and the Gibbs free change of adsorption, via calculation of the equilibrium constant for the adsorption process, *K*.

 (6)

#### 4.1 Langmuir isotherm

Use of the Langmuir adsorption isotherm is the most simplistic approach and in the assumption that every site is equivalent and the ability of a molecule to adsorb there is independent of whether or not the nearby sites are occupied. The Langmuir adsorption isotherm can be ascribed by the following equations:

 (7)

 or (8)

Where *c* = inhibitor concentration, *θ* = surface coverage, *p* and a are constants characteristic of each system.

An invariable test for Langmuir isotherm adsorption is a rectilinear plot of log (*θ/1- θ*) against log *c*.

Meakins [60] observed that some quaternary ammonium compounds adsorbed on steel in sulphuric acid at 20, 40, and 70 °C according to the Langmuir isotherm. Figure 11 illustrates the results obtained at 20 °C. Other authors have reported similar behaviour. For example, thiourea and its derivatives inhibiting steel in sulphuric acid at 40 °C [61], pyridiylhydrazone derivatives inhibiting aluminium in hydrochloric acid at 25 °C [62] and benzotriazole inhibiting iron in sulphuric acid [63].

#### 4.2 Freundlich isotherm

The Freundlich adsorption isotherm makes one different assumption from the Langmuir isotherm in that all the sites are not equivalent and adsorption occurs at the most energetically favourable ones. It is assumed that the adsorption enthalpy changes logarithmically with inhibitor concentration. The Freundlich adsorption isotherm can be written as:

 (9)

Where *c* = inhibitor concentration, *T* is the thermodynamic temperature (K) and *R* is the gas constant (8.314 J K-1 mol-1); '*p*' and '*a*' are constants characteristic of each system. An invariable test for Freundlich isotherm adsorption is a rectilinear plot of log *θ* against log *c*.

Szklarska-Smialowska and Dus [64] observed that several phosphororganic compounds adsorbed on steel in 2 M HCl at 24 °C according to the Freundlich isotherm as shown in Figure 12. Sanyal and Srivastava [65] also reported Freundlich isotherm behaviour for a corrosion inhibition, of steel in hydrochloric by acid benzyltriphenylphosphonium bromide.

#### 4.3 Temkin isotherm

The Temkin adsorption isotherm also presumes that adsorption occurs at the most energetically favourable sites. It is assumed that the adsorption enthalpy changes linearly with inhibitor concentration. The Temkin adsorption isotherm can be ascribed by the following equations:

 (10)

A plot of *θ* against log *c* is a test for the Temkin isotherm.

Gad Allah *et al.* [66] noted that adsorption of some pyrazole derivatives on zinc in 0.06 M hydrochloric acid obeyed Temkin isotherm behaviour. The example given in Figure 13 is for phenylpyrazole. Other examples of Temkin isotherm behaviour are by Abou-Romia *et al.* [67] for pyrazole derivatives inhibition of aluminium in 0.5 M sulphuric acid at 25 °C, Abdel-Aal *et al.* [68] for quinoline inhibition of zinc in 0.1 M hydrochloric acid at 30 °C and Moretti *et al.* [69] for indole inhibition of steel in 0.5 M sulphuric acid at 25, 40, 55, and 70 °C.

#### 4.4 Frumkin isotherm

The Frumkin adsorption isotherm can be described by the following equations:

 (11)

and (12)

A rectilinear plot of ln[*θ* /*c*(1- *θ*)] against *θ* is an invariable test for the Frumkin isotherm. The parameter '*a*' is the Frumkin interaction constant, describing molecular interaction in the adsorption layer and the heterogeneity of the surface. It can be positive or negative and is a measure of the steepness of the adsorption isotherm, the more positive '*a*' is the greater the interaction at higher surface coverages [70]. '*a*' can be related to the Temkin heterogeneity factor, *f* by the following equation:

 (13)

An example of Frumkin isotherm behaviour is shown in Figure 14 for the adsorption of triethanolamine in 0.5 M NaCl at pH 1.3 and 25 °C on an aluminium electrode [71]. Metikoš-Hukovic *et al.* [71] also calculated a (1.74) and K (18.41 mol.dm-3) from the Frumkin isotherm. Other authors [72-74] have also reported Frumkin isotherm behaviour. Fouda and Al-Naimi [73] calculated the values of *f* and shown in Table 5 for adsorption on copper in 3 M H2SO4 at 25 °C.

**4.5 Flory-Huggins isotherm**

The Flory-Huggins adsorption isotherm is the first of the isotherms in this section to consider the water displacement reaction shown in equation (1). This isotherm can be described by the following equations:

 (14)

and (15)

From equation (15), a rectilinear plot of log (*θ/c*) against log (1-*θ*) is an invariable test of the Flory-Huggins adsorption isotherm. The gradient will be equivalent to the value of *x* and from the intercept, log *xK* can be obtained.

Figure 15 shows the adsorption of 4-amino-3-thio-1,2,4-triazolidine (HATT) and 2-amino-5-thio-1,3,4-thiadiazole (HATTD) adsorption on steel in sulphuric acid at 30 °C [74]. The standard free energy of adsorption was calculated to be -29.5 and -31.0 kJ mol-1, for HATT and HATTD respectively. Osman *et al.* [75] also noted that the Flory-Huggins adsorption isotherm suggests that the relative adsorbed areas for water molecules and organic compound are in a ratio of 1:2.1. Khamis and Atea [76] studied the adsorption of several triazoline derivatives on aluminium in hydrochloric acid using the Flory-Huggins adsorption isotherm. Values for *x*, *K* and the standard free energy of adsorption were calculated for each of the six compounds tested and the results are shown in Table 6.

Khamis *et al.* [77] studied water displacement reaction of adsorption of 2-(triphenylphosphoranylidene) succininc anhydride (2TPSA) on nickel in 0.1 M sulphuric acid at 27 °C using the Flory-Huggins and Dhar-Flory-Huggins (see equation (16)) adsorption isotherms.

 (16)

Khamis *et al.* [77] summarised both isotherms as having the form:

 (17)

Where *f(θ,x)* is the configuration factor that depends the physical model and assumptions underlying the derivation of the isotherm. Both isotherms were plotted as described by equation (17), as log *f(θ,x)* versus log *c* for values of *x* up to five. Figure 16 shows the fitting of the Dhar-Flory-Huggins adsorption isotherm. A gradient of unity was obtained for *x* = 3, indicating a 'fit' of experimental data to the Dhar-Flory-Huggins adsorption isotherm; a similar result was obtained for fitting of Flory-Huggins adsorption isotherm.

#### 4.6 Bockris-Swinkels isotherm

The Bockris-Swinkels isotherm [78] can be described by the following equations:

 (18)

 (19)

A plot of *θ*/(1- *θ*)*x*.[*θ* +*x*(1- *θ*)/*x*x]*x*-1 against *c* is a typical Bockris-Swinkels adsorption isotherm.

Figure 17 illustrates Savithri and Mayanna [79] categorisation of tetrabutylammonium iodide adsorption on mild steel in 0.1 M HCl at 30 °C by the Bockris-Swinkels isotherm for values of 4, 5, and 6 for *x*.

Rudresh and Mayanna [80] used equation (11) to calculated values of the free energy of adsorption for assumed values of *x* at various concentrations of triphenylphosphine and tri-*p*-tolylphosphine inhibition of zinc in 0.1 M HClO4 at 30 °C (see Table 7 and Table 8).

It may be noted that adsorption isotherms require several points (probably a minimum of five) to show any kind of validity. Despite this, the literature shows several authors willing to state that an inhibitor adsorbs in a particular manner despite only manipulating two or three data points. Some examples of this are given by the work of Drazic [81] stating that adsorption of 2-hydrazino-6-methylbenzothiazole adsorbs on steel according to the Langmuir isotherm, despite only showing two points for hydrochloric acid (always a straight line) and three points for sulphuric acid, which exhibits curvature. Another example is given by the work of Singh and Singh [82] who used three experimental data points to state that an organotin ((C6H5)2SnCl2) compound adsorbs on nickel in methanoic acid according to the Langmuir isotherm (log (*θ*/1-*θ)* against log *c*). It is also shown by the same authors [82] that a straight line can be fitted to a plot of *τ* against log *c* which is the equivalent of a Temkin isotherm plot (as *τ* ≈ *θ ×* 100) for the same system.

## **5. Mechanism of inhibitor action**

If an organic molecule adsorbs on a metal surface and causes a retardation of either the anodic dissolution of metal or cathodic hydrogen evolution reaction, or both, the molecule is regarded as an inhibitor of corrosion. In this section it hoped to indicate the predominant manner in which various inhibitors work to retard the corrosion process. Trabanelli [44] identified four different mechanisms:

 Formation of a physical barrier.

 Reduction of metal reactivity.

 Changes in the electrical double layer.

 Participation of the inhibitor molecule in partial electrochemical reactions.

According to Foroulis [50], the mechanisms above are predominant not exclusive modes as that in almost all cases the adsorbed species function as a physical barrier.

### 5.1 Formation of a physical barrier

The physical barrier mechanism involves the hindrance of reactants reaching the metal surface and corrosion products leaving where adsorbed inhibitor molecules impede movement near the metal surface. This mechanism requires the adsorbed layer to have impermeable coverage for effective inhibition. Foroulis [50] suggested that inhibitors that are chemisorbed would form an impervious layer with rather weak lateral forces between adsorbed molecules. However, when inhibitor molecules are electrostatic adsorbed there are strong lateral repulsive forces between inhibitor molecules. Consequently, an impervious barrier cannot be maintained. Poling [83] investigating inhibition of iron in hydrochloric acid by acetylenic compounds (proparyl alcohol, ethnylcyclohexanol and acetylene) observed surface-catalyzed polymerisation involving hydrogenation and dehydration, creating an impervious polymer film that impeded corrosion (Figure 18). Initiation was thought to be attributed to rapid chemisorption of the acetylenic compound via the triple bond. It was also noted that protection increased markedly with film thickness.

The attainment of limiting anodic and or cathodic currents (i.e., concentration polarisation) at moderate polarisation are indicative of the presence of a barrier type mechanism.

Pandit Rao *et al.* [84] reported that piperidine dithiocarbamate (PDC) inhibited of steel in sulphuric acid via the formation of complex film, possibly Fe(PDC)n, acting as a physical barrier.

Xue *et al.* [85] using SERS (Surface-Enhanced Raman Scattering), XPS (X-ray photoelectron spectroscopy) and CV (cyclic voltammetry) to identify the presence of a polymeric inhibiting layer of (benzotriazolato)copper(1+) for benzotriazole inhibition of copper in 2 M nitric acid. Benzotriazolium anions act as bridging ligands for the polymer chain. The primary step is the chemisorption of the nitrogen in the benzotriazole indicated by the absence of the N-H stretching bond in the infrared spectra and the presence of a band at 245 cm-1 in the SERS spectra, suggesting N-Cu stretching. The adsorbed benzotriazole molecule is believed to deprotonate in the presence of adsorbed water to form a complex of (benzotriazolato)copper(1+) and water. The final stage is the co-ordination of cuprous cations (produced during corrosion) via the two identical nitrogen atoms in the benzotriazolium anion as shown in Figure 19.

The presence of a (benzotriazolato)copper(1+) complex was also noted in the inhibition of brass (85/15 and 67/33 copper and zinc respectively) corrosion in 0.5 M sulphuric acid by da Costa *et al.* [86]. Solid (benzotriazolato)copper(1+) and (benzotriazolato)zinc(II) complexes were used to identify the adsorbed surface material by performing comparative studies. The mechanism described above for benzotriazole inhibition can also fall into the section of participation of the inhibitor molecule in partial electrochemical reactions as the inhibitor compound forms a complex with adsorbed cuprous ions.

### 5.2 Reduction of metal reactivity

The reduction in metal reactivity by alteration of the nature of the metal surface by the adsorption of inhibitor molecules does not necessarily involve complete coverage of the metal surface. This mechanism involves adsorption of the inhibitor molecules on active sites with respect to partial electrochemical reactions. A reduction in reaction rate via the decrease in the number of reactive cathodic or anodic or both sites on the metal does not alter the reaction mechanism. Hoar and Holliday [87] described the surface on a metal dissolving in acidic solutions as anodic sites at specific crystallographic locations in a sea of cathodic sites where hydrogen evolution occurring anywhere on the metal surface. The anodic sites would form at the ends of incomplete rows and edges of incomplete layers (i.e. crystallographic defects). Hoar and Holliday [87] postulated that an adsorbed molecule at an anodically active site could influence neighbouring cathodic sites and vice versa.

Hoar and Holliday [87] also postulated that at high surface coverages (near monolayer), any gaps due to irregular packing, will be small and due to the relative size of the hydrated hydrogen and metal ions, anodic inhibition will always predominate at high surface coverages (high inhibitor concentrations).

The reduction in reactivity due to adsorption is a function of metal-inhibitor interaction. A strong interaction between inhibitor and the metal surface, as encounter in chemisorption, is likely to lead to a pronounced decreased in metal reactivity.

Fiaud *et al.* [88] using electrohydrodynamical impedance (EHDI) and current density *vs.* rotation speed plots showed evidence of a blocking effect of triphenylphosphine on the copper surface as illustrated in Figure 20. Electrohydrodynamical impedance is frequency response analysis of a system to a sinusoidal speed modulation at a rotating disc electrode (RDE).

In Figure 20, the absence of triphenylphosphine (open circles) allows the plot to intercept the origin, indicating uniform accessibility to the metal surface. At low rotation speeds in the presence of triphenylphosphine (solid circles), the plot runs parallel to that obtained in the absence of triphenylphosphine and intercepts at X which may be indicative of a blocking effect. At high rotation speeds, the plot intercepts the origin. EHDI results confirmed evidence of a blocking effect. Rudresh and Mayanna [80] also reported a parallel shift in Tafel slopes indicative of blocking of anodic and cathodic sites for triphenylphosphine inhibition of zinc in perchloric acid at 30 °C.

The straight lines APA' and CPC' in Figure 21 represent schematically the polarisation curves of steel in uninhibited acid. A purely cathodic inhibitor would give a curve along CP and a purely anodic inhibitor would give a curve along AP. At low inhibitor concentrations (high corrosion rates), *m*-tolylthiourea gives slight inhibition and acts almost pure cathodically but as concentration (and inhibition) increases, the anodic reaction is inhibited. However, 2,6-dimethylquinoline give almost purely anodic inhibition over a considerable concentration range. These results mean that the inhibitors are not adsorbed equally at anodic and cathodic sites.

West [89] reviewed Hoar and Holliday's [87] work and their conclusions were in general agreement except they attempted a quantitative estimate of the degree of adsorption on anodic and cathodic sites.

### 5.3 Changes in the electrical double layer

The change in the electrical double layer due to adsorption of organic inhibitor molecules is probably one of the most important mechanisms of corrosion inhibition. Electrostatic adsorption occurs via an interaction of the double layer charge with the charge (formal or dipole-induced) of the inhibitor molecule. The result of the interaction is adsorption and a change in potential at the outer Helmholtz plane, this change is known as ψ. From equations (20) and (21), it is shown that a modification in ψ will influence the corrosion rates of both anodic and cathodic reactions.

 (20)

 (21)

When the ψ potential is negative, e.g., during iron corrosion in acidic solutions, the electrostatic adsorption of positively charged inhibitor molecules (i.e. protonated amines, ammonium salts, phosphonium salts) causes ψ to decrease and thus lowers the corrosion rate. One example of this type of mechanism is given by the work of Antropov [90] on pyridinium compounds.

 (22)

 (23)

For the inhibition of pyridine and its derivatives, Antropov [90] calculated the theoretical inhibition coefficients, *γ*, and *ψ* for the cathodic hydrogen ion discharge reaction in the presence of pyridinium ions, using equations (22) and (23). Correlation between experimental and theoretical values was good (see Table 9) indicating that inhibition was predominantly via electrostatic interaction of the inhibitor molecule and the electrical double and that the blocking effect was insignificant.

Foroulis [50] suggested that this mechanism explain the synergistic effect of halogen ions to increase the effectiveness of nitrogen-containing cationic organic compounds, e.g. amines, to inhibit the corrosion of iron in acidic solutions. If the adsorption of an organic molecule causes *ψ* to increases the compound will stimulate corrosion. This effect has been observed for benzoate ion adsorption on iron [91].

### 5.4 Participation of the inhibitor molecule in partial electrochemical reactions

Both the anodic dissolution reaction and the cathodic hydrogen evolution reaction involve the formation of adsorbed intermediates. Interaction of the adsorbed intermediates with organic molecules can either decrease (inhibition) or increase (stimulation) electrode reaction rate depending on the stability of the complex formed. The adsorbed intermediate for iron corrosion is presumed to be FeOH [92-95]. Kelly [95] postulated the mechanism for iron dissolution in acid solutions as:

 Fe + H2O  Fe(H2O)ads (24)

 Fe(H2O)ads  Fe(OH-)ads + H+ (25)

 Fe(OH-)ads  Fe(OH)ads + e- (26)

 Fe(OH)ads → Fe(OH)+ + e- RDS (27)

 Fe(OH)+ + H+ → Fe2+ H2O (28)

Donahue, Akiyama and Hobe [92, 93] indicated that inhibition occurs via the formation of a complex, such as [Fe(OH).Inhn], where Inh is the inhibitor. For the organic molecule to act as inhibitor, the surface complex must be more stable to oxidation as shown in step (28) than the intermediate, Fe(OH)ads. Work by Vaidyanathan and Hackerman [96] on inhibition of steel in 1.0 M perchloric acid at 25 °C by furan derivatives assumed a similar mechanism of inhibition. Donahue and Hobe [93] suggested that a molecule with a great affinity for a lattice ion but a negligible tendency for long-lived adsorption, would function as an inhibitor.

Inhibitors may also retard the cathodic reaction as indicated by a change in cathodic Tafel slope. This effect was observed by Grigoryev and Osipov [97] by aniline derivatives, benzaldehyde derivatives and pyrilium derivatives inhibiting iron in hydrochloric acid at 20 °C. According to Grigoryev and Osipov [97], the rate determining step for hydrogen evolution on iron in acid solutions (pH < 2) is the recombination of hydrogen to form adsorbed hydrogen molecules. However, Grigoryev and Osipov [97] observed that aniline derivatives and pyridinium derivatives retarded the cathodic reaction by controlling the rate of hydrogen discharge to form hydrogen adatoms.

**6. Recent developments**

1. Improved molecular modelling, including faster computations, menu driven algorithms and graphical displays were applied by several authors. Dohare *et al.* [98] and Benbouguerra *et al.* [99] have performed experimental theoretical investigations on Schiff base derivatives as corrosion inhibitors; while computational investigations were carried out on quinoline derivatives [100] and synthesized pyrimidine derivatives for corrosion inhibition [101]. Monte Carlo simulation [102], molecular dynamics simulation [103], and quantum chemical studies [104] were applied by other authors.

2. Several studies moved towards more sustainable, environmentally compatible inhibitors, such as the use of natural products grown as trees or crops which included oil of Foeniculumvulgare [105], Glycyrrhiza glabra leaves [106], Nettle leaves [107], Glycyrrhiza glabra [108], Sunflower seed [109], and Rollinia occidentalis [110].

**7. Summary**

1. The first stage of inhibition is the adsorption of inhibitor molecules on the metallic surface, there are two type of adsorption: electrostatic and chemical (chemisorption).

2. Electrostatic adsorption is the result of electrostatic attraction between the inhibitor molecules and the electrical charge at the metal surface.

3. Electrostatic adsorption is a rapid process, but is also highly reversible and has a low activation energy.

4. Electrostatic adsorption is dependent on the surface charge, the electrical characteristics of the organic inhibitor molecule and the type of adsorbate anions present in the aggressive media, e.g. chloride, sulphate ions.

5. Chemisorption is slower than electrostatic adsorption, has a higher activation energy and is not completely reversible.

6. Adsorbed molecules can interact either repulsively or attractively effecting the strength of the adsorption bond, as shown by a change in configuration due to repulsive forces, leading to a second heteroatom chemisorption, increasing the overall adsorption strength.

7. Adsorption isotherms can assist in the description of the adsorption and provide information on the adsorption equilibrium and thus give a value to the free energy of adsorption, *Gads*.

8. The reduction in metal reactivity involves adsorption of the inhibitor molecules on active sites with respect to partial electrochemical reactions.

9. Electrostatic adsorption occurs via an interaction of the double layer charge with the charge of the inhibitor molecule.

10. Both the anodic dissolution reaction and the cathodic hydrogen evolution reaction involve the formation of adsorbed intermediates.

**8. Recommendations for further R & D**

1. The study of the metallic surface under corrosion and its inhibition condition to examine the nature of any substances formed or present at the metal's surface,

2. Use in-situ techniques; so that the corrosion processes can be monitored as it occurs, thus providing measurements in the natural environment, without the need to alter the sample to suit the technique.

3. Early application of classical electrochemical techniques that could provide valuable information on a corroding system include: cyclic voltammetry (CV), which shows oxidation and reduction cycle on the specimens surface or electrochemical impedance spectroscopy (EIS) - which can measures corrosion rates and provide mechanistic information.

4. Application of computer simulation, where computer can be used to create molecular configuration of the metallic surface and inhibitor molecules (or whatever is equivalent to other fields) and manually dock inhibitor molecules to surfaces or allow the computer to calculate from known variables of interaction, etc. the best position adopted by an inhibitor molecule at a specific metallic surface.

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|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Inhibitor** | **%*τ*** | **%*τc*** | **Δ*bu* / V** | **%*τa*** |
| Aniline | 36 | 15 | -0.001 | 55 |
| *m*-Toluidine | 31 | 13 | +0.001 | 46 |
| *o*-Toluidine | 43 | 14 | +0.005 | 65 |
| *N*-Methylaniline | 25 | 10 | -0.003 | 41 |
| *N*-Ethylaniline | 30 | 13 | +0.006 | 47 |
| *N*-*n*-Propylaniline | 36 | 26 | +0.006 | 53 |
| *N*-Dimethylaniline | 41 | 11 | 0.000 | 66 |
| *N*-Diethylaniline | 54 | 37 | +0.010 | 68 |
| *N*-Di-*n*-propylaniline | 57 | 40 | +0.008 | 71 |
| Methylamine | 13 | 18 | 0.000 | 0 |
| Ethylamine | 17 | 20 | +0.002 | 20 |
| Propylamine | 23 | 20 | +0.006 | 29 |
| Error limit | 4 | 4 | 0.003-0.004 | 4 |

Table 1. Corrosion inhibitor efficiencies for aniline and amine inhibitors, showing total (%τ), both anodic and cathodic partial efficiencies (%τa and %τc); the change in cathodic Tafel slope compared to the uninhibited condition (**Δ**bu) is also shown [8].

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Inhibitor | %*τ* | A1 | A2 | A2' | A3 |
| Pyridine | 36 | 30 | 37 | 37 | 37 |
| 3-Picoline | 40 | 60 | 49 | 49 | 68 |
| 2-Picoline | 44 | 60 | 49 | 49 | 68 |
| 4-Picoline | 44 | 30 | 41 | 49 | 68 |
| 3,5-Lutidine | 44 | 60 | 58 | 58 | 68 |
| 2,6-Lutidine | 46 | 60 | 58 | 58 | 68 |
| 2,4-Lutidine | 46 | 60 | 58 | 58 | 68 |
| 2,4,6-Collidine | 48 | 60 | 68 | 68 | 68 |

Table 2. Calculated molecular areas in Å2 for pyridine and mono, di- and trimethylpyridine in three positions: the ring system vertical (A1), the ring system parallel, either with the smallest possible circle (A2), the cation oriented the same way (A2') or allowing free rotation about the center of the ring (A3) the inhibitor efficiency at an inhibitor concentration of 0.15 mol dm-3 is also shown [53].

|  |  |  |  |
| --- | --- | --- | --- |
| Inhibitor |  | Corrosion rate/ mg dm-2 day-1 | % Inhibition efficiency, %*τ* |
| Blank |  | >48900 | 0.00 |
|  | (1) | <48900 | 0.00 |
|  | (2) | 13200 | 73.0 |
|  | (3) | 146 | 99.7 |
|  | (4) | 1956 | 96.0 |

Table 3. Corrosion inhibition data for 1020 C-steel in 2.8 M HCl at 65 °C [50].

|  |  |  |
| --- | --- | --- |
| Inhibitor | *c*/ mmol dm-3 | % Inhibition efficiency |
| Ph3PCH2CH2PPh3 | 0.01 | 44 |
| 0.1 | 89 |
| 1.0 | 95 |
| Ph3PCH=CHPPh3 | 0.3 | 89 |
| 1.0 | 91 |
| 2.5 | 95 |
| Ph3PC≡CPPh3 | 0.05 | -71 |
| 0.3 | -31 |
| 0.6 | -18 |
| 1.0 | 70 |

Table 4. The effect of unsaturation on the inhibition efficiency of diphosphines as a function of zinc concentration in 0.05 M sulphuric acid at 25 °C [57].

|  |  |  |
| --- | --- | --- |
| Compound | *-* | *f* |
| Acrylonitrile | 34.3 | 13.8 |
| Phenylacetonitrile | 33.4 | 16.5 |
| Acetonitrile | 33.0 | 19.9 |
| Trichloroacetonitrile | 32.2 | 24.7 |

Table 5. Calculated values in the Frumkin adsorption isotherm for several nitrile compounds on 3 M H2SO4 at 25 °C [72].

|  |  |  |  |
| --- | --- | --- | --- |
| Compound | - / kJ mol-1 | *K* | *x* |
| 4-amino-3-H-MT | 30.7 | 3500 | 4.6 |
| 4-amino-3-methyl-MT | 37.2 | 45700 | 4.7 |
| 4-amino-3-*n*-propyl-MT | 37.9 | 61300 | 3.7 |
| 4-amino-3-isopropyl-MT | 39.2 | 99900 | 3.0 |
| 4-amino-3-phenyl-MT | 43.8 | 631900 | 3.3 |
| 4-amino-3-*p*-methoxyphenyl-MT | 45.2 | 1111400 | 3.3 |

Table 6. Calculated values of standard free energy of adsorption (-), equilibrium constant (K), and number of water molecules (x) replaced by each organic molecule for 5-mercapto-1,2,4-triazoline (MT) derivatives adsorbed on aluminum in 2 M HCl at 30 °C [76].

|  |  |  |
| --- | --- | --- |
| Concentration | *θ* | - / kJ mol-1 |
| / mol dm-3 | x = 15 | x = 10 |
| 10-5 | 0.10 | 23.51 | 23.56 |
| 5 × 10-5 | 0.25 | 25.06 | 27.23 |
| 10-4 | 0.40 | 27.07 | 27.95 |
| 1.5 × 10-4 | 0.48 | 27.40 | 28.45 |
| 2 × 10-4 | 0.54 | 27.82 | 29.62 |

Table 7. Surface coverage and free energy of adsorption (calculated using the Bockris-Swinkels isotherm for x = 10 and 15) for adsorption of triphenylphosphine, at various concentrations, on zinc in 0.1 M HClO4 at 30 °C [80]

|  |  |  |
| --- | --- | --- |
| Concentration | *𝛳* | -/ kJ mol-1 |
| / mol dm-3 | x = 17 | x = 12 |
| 1 × 10-5 | 0.12 | 23.51 | 28.74 |
| 5 × 10-5 | 0.29 | 25.56 | 28.03 |
| 1 × 10-4 | 0.45 | 26.86 | 28.95 |
| 1.5 × 10-4 | 0.55 | 28.45 | 29.92 |
| 2 × 10-4 | 0.62 | 25.65 | 30.67 |

Table 8. Surface coverage and free energy of adsorption (Calculated using the Bockris-Swinkels Isotherm for x = 12 and 17) for adsorption of tri-p-tolylphosphine, at various concentrations, on zinc in 0.1 M HClO4 at 30 °C [44].

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Compound | Concentration / mol dm-3 | Δ*Ecor* (exp) / mV | *ψ* (calc) / mV | *γ* (exp) | (calc) |
| Pyridine in 0.5 M H2SO4 [65] | 0.01 | 5 | 10 | 1.2 | 1.3 |
| 0.02 | 7 | 14 | 1.3 | 1.5 |
| 0.05 | 9 | 18 | 1.4 | 1.6 |
| 0.10 | 14 | 28 | 1.7 | 2.2 |
| 0.20 | 16 | 32 | 2.3 | 2.5 |
| 0.50 | 19 | 38 | 3.0 | 2.9 |
| Pyridine in 6 M HCl | 0.01 | 1 | 2.8 | 1.05 | 1.07 |
| 0.05 | 2 | 5.6 | 1.20 | 1.14 |
| 0.10 | 5 | 14 | 1.40 | 1.42 |
| 0.15 | 6 | 16.8 | 1.60 | 1.52 |
| Chloride N *n*-decyl-3-oxypyridine in 3 M H2SO4 |  0.000256 | 36 | 72 | 7 | 7 |
| 0.00064 | 53 | 106 | 21 | 19 |
| 0.0016 | 67 | 134 | 39 | 41 |
| 0.004 | 82 | 164 | 100 | 93 |
| 0.01 | 92 | 184 | 170 | 161 |

Table 9. Calculated change of potential at the outer Helmholtz plane (ψ calc) and theoretical inhibition coefficients for cathodic hydrogen discharge (γH2 calc) and experimental values for the theoretical inhibition coefficients and changes in corrosion potential (ΔEcor) for pydridine and a derivative [90].

1907

Use of hydrocarbon such as tar sludge (waste product from oil refining).

1936

Recognition of inhibitor on metal surfaces.

1963

Connection between catalysis poisoning of Raney nickel in hydrogenation reactions and corrosion inhibition.

1984

Patent on Phosphonium salt corrosion inhibitors for high Density Brines.

1900

* Patent on liquor produced from wheat.
* Patent on the use of natural products.

1928

Creation of synthetic corrosion inhibitor; leading to manufacture of ***"Antra"***.

1958

Effect of molecular structure on corrosion inhibition.

1970

Patent on the use of "Phosphonium compounds and/or Ethylene Glycol – Bis-(trialkylphosphonium Acetates) as corrosion inhibitors.

1909

Testing dry pigments on steel sheet.

1900

1910

1920

1930

1940

1950

1960

1970

1980

1990

1970

2 Patents for the use of Phosphonium compounds as corrosion inhibitors.

1970

Patent on polyalkyleneethers having dangling phosphonium groups.

Figure 1. Developments of corrosion inhibitors. Patent on liquor produced from wheat, and Patent on the use of natural products (1900) [2]; Use of hydrocarbon such as tar sludge (waste product from oil refining) (1907) [3]; Testing dry pigments on steel sheet (1909) [5]; Creation of synthetic corrosion inhibitor; leading to manufacture of *"Antra"* (1928) [6]; Recognition of inhibitor on metal surfaces (1936) [7]; Effect of molecular structure on corrosion inhibition (1958) [8]; Connection between catalysis poisoning of Raney nickel in hydrogenation reactions and corrosion inhibition (1963) [9]; Patent on the use of ‘Phosphonium compounds and/or ethylene glycol-bis-(trialkylphosphonium acetates) as corrosion inhibitors (1970) [10]; Patent on ‘polyalkyleneethers having dangling phosphonium groups’ (1970) [11]; Patent on ‘the use of phosphonium compounds as corrosion inhibitors’ (1970) [12,13]; Patent on ‘Phosphonium salt corrosion inhibitors for high density brines’ (1984) [14].

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Figure 2. Schematic showing adsorption variations with surface charge [44].





Figure 3. Schematic representation of adsorption behaviour of aniline on mild steel in dilute HCl, at 30 oC, at different applied potentials [45].



Figure 4. Plot of concentration of adsorbed aniline on mild steel in dilute HCl, at 30 °C, at various applied potentials [45].



Figure 5. Resonance structures of benzoic acid [50].



Figure 6. Relationship between log [t/(1-t)] and electronegativity for the 5B and 6B group inhibitors at the corrosion potential and at -150 mV vs. SCE for nickel in 3 M HClO4 at 30 oC [53].



Figure 7. Resonance structures via hyperconjugation of a) m-picoline, b) p-picoline, and c) o-picoline [54].



Figure 8. Inhibition efficiency of some diphosphines of formula Ph2P(CH2)nPPh2 for zinc corrosion in 0.05 M sulphuric acid at 25 °C [57].



Figure 9. π-bonds in (a) aromatic, and (b) unsaturated aliphatic molecules [50].

|  |
| --- |
|  |

Figure 10. Adsorption via one or both imine groups in homopiperazine [59].



Figure 11. Langmuir adsorption isotherm for quaternary ammonium compounds inhibiting corrosion of steel in sulphuric acid at 20 oC [60].



Figure 12. A plot of the logarithm of inhibitor concentration against logarithm of surface coverage for tributyl-selenium-phosphate of low carbon steel in 1 M HCl at 50, 60, and 70 oC showing Freundlich isotherm behaviour [64].



Figure 13. Temkin adsorption isotherm of phenylpyrazole inhibiting zinc in 0.06 M hydrochloric acid at 25 °C measured by capacitance and polarisation measurement [66].



Figure 14. Frumkin adsorption isotherm for an aluminium electrode in a solution of triethanolamine in 0.5 M NaCl at pH 1.30 and 25 °C [71].



Figure 15. Application of the Flory-Huggins adsorption isotherm to 4-amino-3-thio-1,2,4-triazolidine (HATT) and 2-amino-5-thio-1,3,4-thiadiazole (HATTD) adsorbed on steel in sulphuric acid at 30 °C [75].



Figure 16. Test of Dhar-Flory-Huggins adsorption isotherm for values of x up to five for inhibition of nickel in 0.1 M H2SO4 at 27 °C by 2-(triphenylphosphoranylidene) succinic anhydride [77].



Figure 17. Bockris-Swinkels adsorption isotherm for adsorption of tetrabutylammonium iodide on mild steel in 0.1 M HCl at 30 °C [79].



Figure 18. Proposed structure of protective film formed by acetylenic compounds [83].





Figure 19. Proposed mechanism of benzotriazole inhibition of copper in nitric acid [85].



Figure 20. A plot of current density vs. rotation speed of a copper RDE in 0.05 M sulphuric acid with and without triphenylphosphine [88].



Figure 21. Relationship between corrosion potential and corrosion current for steel in 5% sulphuric acid at 70 °C inhibited by thiourea derivatives and quinoline derivatives [87].

**Figure captions**

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