Synthetic Polymeric Corrosion Inhibitor

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An anti-corrosion inhibitor is one of the most useful methods to prevent metal corrosion toward different media. In comparison with small molecular inhibitors, a polymeric inhibitor can integrate more adsorption groups and generate a synergetic effect, which has been widely used in industry and become a hot topic in academic research.

Keywords: metal corrosion ; anti-corrosion inhibitor ; polymeric inhibitor

1. Phosphorus-Containing Synthetic Polymeric Inhibitor

Phosphorus can coordinate with metal surfaces more strongly than oxygen, which has been introduced to polymeric inhibitor as additional adsorption site in the form of both coating and solution. Generally, there are two kinds of strategies to prepare phosphorus-containing synthetic polymeric inhibitor: (i) post-modification of natural polymer ^[1]; and (ii) direct polymerization of phosphorus-containing monomer ^[2].

Grafting phosphorus-containing groups to natural polymers not only can introduce additional adsorption sites, but also improve their water solubility. David et al. functionalized chitosan with phosphonic acids (P-1 in **Figure 1**) via the Kabachnik-Fields reaction and subsequent hydrolysis ^[1]. The introduction of phosphonic acid groups greatly decreased the dynamic viscosity of chitosan. Moreover, compared with the native chitosan, P-1 showed improved adsorption on the carbon steel surface and better corrosion inhibition property. Meanwhile, such material can also be used in metallic aluminum corrosion protection. David et al. ^[1] fabricated inhibitive coating by the layer-by-layer (L-b-L) technique from native chitosan or synthesized phosphorylated chitosan (P-1 in **Figure 1**) combined with alginate functionalized chitosan. It was found that the coating can create a physical barrier that acts mainly by reducing the active surface area and has the effect of blocking the penetration of the aggressive species into the metal substrate. According to the result of electrochemical impedance spectroscopy (EIS) in 0.1 M Na₂SO₄ solution, the as prepared coating showed improved corrosion resistance for aluminum alloy 3003.



Figure 1. Chemical structures of typical phosphorus-containing polymeric inhibitors (P1–P8) and monomers (M1–M3).

Phosphonic acids can also be introduced to chitin. As demonstrated by Hebalkar and coworkers, they synthesized phosphorylated chitin (P-2 in **Figure 1**) to solve the water solubility of native chitin and improve their coordination ability with metal surface ^[3]. According to the gravimetric analysis in NaCl solution, only 200 ppm P-2 can protect copper very well with maximum inhibition efficiency as high as 92%.

By using simple phosphorylation, phosphonic acids can be introduced to ethyl cellulose. Ben Youcef et al. ^[4] reported the method of microcapsulation to encapsulate almond oil as inhibitor by using phosphorylated ethyl cellulose (P-3 in **Figure 1**). It was found that the P-O⁻ functionalized groups strongly interacted with the metal ions in the metal substrate, and therefore, generated synergetic anti-corrosion effect for almond oil.

Comparing with the post-functionalization of natural polymer, the direct polymerization of phosphorus-containing monomer provides accurate structure control and rational design of polymeric inhibitors ^[5]. The most commonly used phosphoruscontaining methacrylate-base monomers are shown in **Figure 1** (M-1 and M-2). For example, Keil et al. ^[6] reported UVcured polyester acrylate coatings on zinc and iron as anti-corrosion surface. Moreover, Pebere et al. ^[7] extended the study on the UV polymerization of M-1 and M-2 to prepare effective corrosion protection. It was found that coatings containing phosphonic acid methacrylate (M-2) showed better anti-corrosive performance than those containing methacrylate phosphonic dimethyl ester (M-1), owing to the improved adsorption of phosphonic acid to metal surface than corresponding ester. Similarly, Ilia et al. ^[8] copolymerized vinylphosphonic acid (VPA) and dimethyl vinylphosphonate (DMVP) to prepare PVPA-*co*-PDMVP copolymer P-5 (**Figure 1**) via free radical photopolymerization. Interestingly, they found that the presence of phosphonate groups from DMVP in copolymers was beneficial and a molar ratio VPA:DMVP 4:1 and 3:1 enhanced the anticorrosion for iron surface in comparison with homopolymer of vinylphosphonic acid (P-4). In other words, the coordination between P-OH and metal surface competes with the formation rate of uniform protective layer. While copolymers with VPA:DMVP 4:1 may show higher diffusion coefficient, and therefore, faster formation of protective film than other polymers.

In addition to introducing additional adsorption sites to polymeric inhibitors, low surface energy monomers have also been used in the design of efficient inhibitor. Moratti et al. ^[9] prepared block copolymer P-6 (**Figure 1**) via free radical polymerization of heptafluorodecyl methacrylate and (dimethoxyphosphoryl) methyl methacrylate. The copolymers were then immobilized as a monolayer film to the surface of 316L stainless steel by treatment of dilute solutions in trifluoroacetic acid for 30 min followed by rinsing. Owing to the presence of fluorinated block and the presence of adsorption site from the phosphorus block, the resulting polymeric inhibitor exhibited excellent anti-corrosion property and long-term stability.

Champagne et al. ^[2] reported the nitroxide-mediated polymerization (NMP) to prepared polystyrene-*b*poly(dimethyl(methacryloyloxy)methyl phosphonic acid) (P-7 in **Figure 1**) and its graft onto polysaccharide chitosan (P-8 in **Figure 1**). The controlled radical polymerization feature of NMP allowed rational design of the repeat unit and the topology of the resulting polymer. It can be anticipated that the resulting grafted polymers are potential candidate for excellent inhibitor due to the coexistence of chitosan and phosphorus-containing polymers (**Table 1**).

In addition to the radical-based polymerization, sol-gel method has also been developed for the preparation of phosphorus-containing polymeric inhibitors. Mandler et al. ^[10] incorporated phenylphosphonic acid (PPA) to a sol-gel film to enhance the corrosion protection of metallic aluminum; however, it was found that such method resulted in aggregation and phase separation. To overcome such problem, Choudhury et al. ^[11] proposed the design of networked methacrylate-hybrid by copolymerizing 2-(methacryloyloxy)ethyl phosphate (M-3 in **Figure 1**), containing a polymerizable methacrylate group and functional phosphate group with 3-[(methacryloyloxy)propyl] trimethoxysilane ^[12]. It was found that the proposed network not only enhanced the binding of the coating to the metal substrate via the acid-base interaction of the P-O- group of the phosphate with the Mn+ of the metal substrate, but also resulted excellent anti-corrosion property.

Table 1. Inhibition property of typical phosphorus-containing synthetic polymeric inhibitor.

Inhibitor	Metal	Corrosion Medium	Test Method	Highest IE (%)	Reference
P-1	aluminum alloy 3003	pH = 5 acetic acid	EIS	I	[1]
P-2	copper	200 × 10 ⁻³ g/L NaCl	weight loss, EIS	92	[<u>3]</u>
P-3	mild steel	3.5% NaCl	salt spray test	I	[4]
M-1/M-2	low-carbon steel	0.1 M NaCl	EIS	85	[6]

Inhibitor	Metal	Corrosion Medium	Test Method	Highest IE (%)	Reference
M-3	mild steel	3.5% NaCl	PDP, EIS		[<u>11</u>]
P-4/P-5	iron coin	3% NaCl	PDP, EIS	83.5	[8]
P-6	316L stainless steel	trifluoroacetic acid	long term stability tests	I	<u>[9]</u>

2. Sulfur-Containing Synthetic Polymeric Inhibitor

As a classic coordination atom, sulfur can form a stable complex with different metal ions. Generally, there are two kinds of sulfur-containing polymeric inhibitors: (i) polythiophene and (ii) polysulfone.

Normally, intrinsically conducting polymers or conjugated polymers, such as polythiophene, polypyrrole, and polyaniline. have been widely used as protective coating for corrosion protection of steel [13]. Polythiophene was first reported as corrosion protection in 1989 [14]. The facile electropolymerization of thiophene and its derivatives allows the preparation of homogeneous polymer films on the surface of different metals with good electrical properties and chemical stability [15]. Gonzalez-Rodriguez and coworkers ^[16] compared the anti-corrosion property of poly(3-octyl thiophene) (P3OT) and poly(3-hexylthiophene) (P3HT) (P-9 in Figure 2). The plate they used was a commercially available 1018 carbon steel sheets, and copper wires were welded to the plate, which was used as a reaction platform for electrode deposition. The polymer solution was then deposited on the electrode, evaporated solvent, dried, and annealed to afford the P3OT/P3HT coating. Both polymer films were found to be effective in protecting the substrate from corrosion by decreasing the critical current necessary to passive the substrate, increasing the pitting potential, and broadening the passive interval, and P3HT was found to be more effective due to a much lower number of defects than P3OT films. Interestingly, P3HT gave better protection than P3OT, because of the lower defects in the film of P3HT than that for the P3OT films. Thiophene can also be copolymerized with other monomers to improve the anti-corrosion property. For example, Branzoi et al. [17] investigated the anti-corrosive properties of poly(N-methylpyrrole-Tween20/3-methylthiophene) coatings on carbon steel type OLC 45 in 0.5 M H₂SO₄ medium. The surfactant Tween 20 was a dopant used in the electropolymerization process, which could improve the anti-corrosive properties by hindering the corrosive sulfate ion penetration. The corrosion rate of PNMPY-TW20/P3MT-coated OLC 45 has been indicated to be ~10 times reduced in comparison with uncoated OL 45, and the corrosion protection efficiency of the coating is above 90%. More importantly, the anti-corrosion property of such coating can be tuned by the condition of electropolymerization, such as electrodeposition current and time, highlighting the potential application of such technique.



Figure 2. Chemical structures of typical sulfur-containing polymeric inhibitors.

Furthermore, the anti-corrosion performance of polythiophene can be improved by blending with other polymers ^[19]. Meanwhile, blending can also improve the processability and mechanical strength of the material and reduce the cost of expensive conductive polymers ^[19]. For example, Nicho et al. ^[20] blended P3OT with polystyrene (PS) and deposited it onto stainless steel sheets using the drop-casting technique, where a solution of the blend is added dropwise to the steel sheet, the solvent is evaporated, then dried and annealed. Subsequently, the room temperature corrosion behavior of the prepared P3OT/PS coated 304 stainless steel was studied under 0.5 M NaCl. According to their study on the temperature effect, it was found that high temperature (e.g., 100 °C) can increase the adhesion degree between coating and substrate, making the coating less porous and defective to give a denser surface, therefore giving it better inhibition performance. Furthermore, they systematically investigated the anti-corrosion property of P3HT, P3HT/PS and P3HT/PMMA (polymethyl methacrylate) blends coatings on A36 steel corrosion protection in 0.5 M H₂SO₄ solution. It was found that blends of P3HT with PMMA and PS improved the protection of steel in comparison with native P3HT. P3HT/PMMA blend gave the best protraction to the steel. To enhance the interface interaction between different polymers in the blend, Huang et al. ^[21] reported the preparation of P3HT/poly(styrene-*co*-hydroxystyrene) blend (P-10 in **Figure 2**) via the intermolecular hydrogen bonding not only improved the miscibility between

two polymers, but also enhanced the adhesion force between iron and coating layer. Compared with native P3HT, the inhibition performance of the blend improved and the decreased upon thermal treatment.

In addition to polythiophene-based inhibitor, polysulfone represents another kind of sulfur-containing polymeric inhibitor. For example, the non-toxic amino acid methionine was used as a sulfur-containing corrosion inhibitor for mild steel because of the coexistence of N, O, and S atoms in one molecule. Moreover, the corrosion inhibition performance of methionine, methionine sulfoxide, and methionine sulfone in HCI for mild steel has been studied previously and moderate inhibition efficiency has been achieved. To enhance the adsorption of inhibitor and metal substrate, polysulfone may be potential strategy [22]. Ali and coworker carried out systematical study on the anti-corrosion performance of series of polysulfones. Butler's cyclopolymerization of diallyl ammonium salts and their copolymerization with SO₂ was used to synthesize a series of polysulfones with residues of essential amino acid methionine (P-11 and P-12 in Figure 2) [23]. Especially, in the copolymer P-14 (Figure 2), half of the sulfide was oxidized to corresponding sulfone, which greatly improved the water solubility. More importantly, the copolymer P-14 demonstrated superior inhibition of mild steel in 1 M HCl at 60 °C with inhibition efficiency of 99% at concentration of 25 ppm, while corresponding monomer can only achieve inhibition efficiency of 31% at the same concentration, highlighting the contribution of polymer configuration. In another study, they treated polymer P-11 with H_2O_2 to generate corresponding sulfone P-12 and sulfoxide P-13 ^[24]. All of these polymers can achieve inhibition efficiency (IE) up to 87%, even at a very low concentration of 6 ppm in 1 M HCl. It revealed that the sulfoxide (S=O) base sequence was more effective in mitigating mild steel corrosion in comparison with sulfide (S) and sulfone (O=S=O). The copolymerization methodology also allowed the introduction of multiple adsorption groups to one inhibitor. For example, they [24] also synthesized a new tripolymer P-15 (Figure 2) consisting of carboxylate, sulfonate, and phosphonate using the Butler cyclopoymerization technique and copolymerization with sulfur dioxide. They evaluated the performance of P-15 as a corrosion inhibitor for St37 carbon steel. It was found that the as prepared inhibitor demonstrated protection efficiency of 79.5% and 61.1% in HCl and H₂SO₄ media at a concentration of 1000 mg/L, respectively. Interestingly, it was found that the addition of KI can greatly enhance the performance to give IE as high as 93.5%, which may due to the synergistic effect of the cooperative co-adsorption of I⁻ on the metal surface. They also synthesized series of poly(bis-zwitterion) (P-16, P-17, and P-18 in Figure 2) with chelating motifs of [NH⁺(CH₂)₂NH⁺ (CH₂CO₂⁻)₂] via the same strategy. These polymers were found to be very good inhibitors of mild steel corrosion in 1 M HCI. Similarly, the addition of KI (400 ppm) can generate synergistic effect to achieve 98% inhibition of mild steel corrosion for a duration 24 h at 60 °C [25].

Compared with polysulfone, polythioether can bind with metal surface more strongly due to the S-Fe bond. In order to improve the anti-corrosion property of native polythioether, the group designed a series of cobaltocenium-containing polythioether type metallo-polyelectrolytes (P-19, P-20, and P-21 in **Figure 2**). The synthesized diolefin monomers were first sulfonated to introduce sulfonate groups, followed by photo-induced thiol-ene polymerization to synthesize the polymer, then azidation and copper-catalyzed post-click modification to graft the cobalt dichloride groups to the polysulfide backbone to obtain the target polymer (The synthesis process is shown in **Figure 3**). It can be found that there are multiple interactions between these polymers and metal surface, such as coordination between S and metal, triazole and metal, electrostatic interactions, and the potential ion- π interaction. According to the weight loss experiments and electrochemical study, all these polymers were found to be effective inhibitors, which can achieve inhibitive efficiency as high as 95% at concentration as low as 10 mg/L. Moreover, the study also revealed the structure–property relationship for the design of new polymeric inhibitor, highlighting the important role of flexible linkage between the polymer main-chain and the charged group and the number of charged groups [26].



In addition to the above sulfur-containing polymeric inhibitors (**Table 2**), sulfur has also been introduced to resin coating. For example, Mohammad El-Sawy and coworkers compared the inhibition performance of modified urea (P-22) with thiourea formaldehyde (P-23) resins for steel surfaces. As expected, owing to the presence of sulfur atoms in thiourea resin, P-23 demonstrated the best protection performance and adhesion ^[18].

Inhibitor	Metal	Corrosion Medium	Test Method	Highest IE (%)	Reference
P-9	1018 carbon steel	0.5 M H ₂ SO ₄	EIS	I	[16]
P-10	iron	3.5% NaCl	EIS, PDP	96	[21]
P-11/P-14	mild steel	1 M HCI	EIS	99	[23]
P-12/P-13	mild steel	1 M HCI	Weight loss	P-12: 94 P-13: 87	[24]
P-15	St37 carbon steel	15% HCI/15% H ₂ SO ₄	EIS, PDP, linear polarization resistance, electrochemical frequency modulation	79.5/61.1	[19]
P-16/P- 17/P-18	mild steel	1 M HCI	Weight loss	P-16: 92.3 P-18: 95.7	[25]
P-19/P- 20/P-21	mild steel	5% HCI	weight loss, EIS, PDP	95	[26]
P-22/P-23	cold-rolled mild steel	3.5% NaCl	weight loss,	I	[18]

 Table 2. Inhibition property of typical sulfur-containing synthetic polymeric inhibitor.

3. Nitrogen-Containing Synthetic Polymeric Inhibitor

Nitrogen-containing synthetic polymeric inhibitor represents a major class of polymeric inhibitors. Most of the nitrogencontaining polymeric inhibitors belong to polyelectrolyte, such as poly(quaternary ammonium), polyethyleneimine, polyaniline, and so on.

4. Other Type of Polymeric Inhibitors

In addition to the above well-studied systems, other kinds of polymeric inhibitors have also been investigated because modern polymer chemistry allows the introduction of hydrophilic and adsorption groups to the side chains on the basis of the main chain structure and copolymerization with other monomers (**Table 3**).

Polyacrylic acid (PAA) is the most well-known vinyl polymer corrosion inhibitor used in the early study of anti-corrosion. Recently, polyacrylate or acrylamide copolymer corrosion inhibitors have become more popular. Lin et al. ^[27] prepared poly(methyl acrylate)-*co*-poly(acrylic acid imidazoline) (MA-ACI, P-38 in **Figure 4**) from methyl acrylate and acrylic imidazoline with azo diisobutyronitrile as initiator. According to the rotating hanging plate method, P-38 showed inhibition efficiency as high as 82% at a concentration of 0.10 g/L in 1 mol/L H_2SO_4 at 30 °C. Taghi et al. ^[28] prepared neodymium-poly acrylic acid complex (Nd-PAA, P-39 in **Figure 4**) by adding neodymium to PAA and applied them to the corrosion protection of ST-12 type in 0.1 M NaCl. Due to the anionic nature of PAA, they deposited densely and crack-free ultrafine Nd-PAA films on the steel surface.



Figure 4. Chemical structure of the poly methyl acrylate-acrylic acid imidazoline, and cobaltocenium-containing waterborne polymeric inhibitors.

Moreover, chemical modification or grafting of inorganic material by synthetic polymer has also been developed to afford efficient inhibitors. For example, as shown in **Figure 5**, Yu et al. ^[29] modified graphene oxide with polystyrene by in situ microemulsion polymerization. The resulted hybrid demonstrated significant improvement in the corrosion resistance in comparison with native graphene and polystyrene, with the corrosion resistance efficiency increasing from 37.9% to 99.5%.

Inhibitor	Metal	Corrosion Medium	Test Method	Highest IE (%)	Reference
P-38	N80 steel sheet	$1 \text{ M H}_2 \text{SO}_4$	PDP, EIS	90.2	[27]
P-39	ST-12 type steel sheets	0.1 M NaCl	PDP, EIS	I	[28]
P-40/P-41/P-42/P-43	Mild steel	4 M HCI	weight loss, EIS, PDP	98	[<u>30</u>]



Figure 5. Synthesis of organo-functionalized graphene oxide ^[29]. Reproduced with permission from ref. ^[29]. Copyright 2014 Royal Society of Chemistry.

Since water solubility is a crucial problem in the design and application of inhibitors, water soluble polymers, such as waterborne polyurethane, have also been used as inhibitors. The research group developed a series of cobaltocenium-containing polyurethanes (P-40, P-41, P-42, and P-43 in **Figure 4**) via the reaction between hydroxyl-terminated cobaltocenium monomer and different kinds of commercially available diisocyanates (the synthetic process is shown in **Figure 6**). The presence of charged cobaltocenium group and its counterion endow the resulting polyurethane good water

solubility. Owing to the multiple interactions between these waterborne polyurethanes and metal surface and the inter/intramolecular hydrogen bonding between urethane groups, these polymers can strongly adsorb on the metal surface and therefore demonstrated excellent corrosion protection property. According to the weight loss experiment, the inhibition efficiency can achieve as high as 98.0% at a concentration as low as 20 mg/mL toward mild steel in 4 M HCI ^[30].



Figure 6. Synthetic procedure for cobaltocenium-containing polyurethane-type inhibitors: P-40/P-41/P-42/P-43 ^[30].

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