



Linear polyesters as effective corrosion inhibitors for steel rebars in chloride induced alkaline medium – An electrochemical approach



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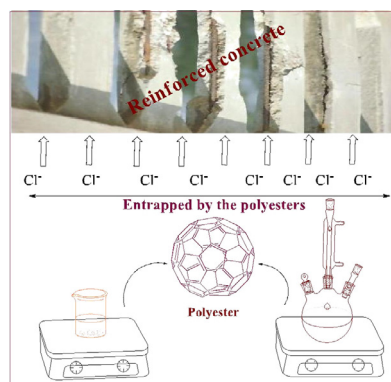
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HIGHLIGHTS

- Effect of polymers at minimum concentrations were evaluated.
- Regression co-efficient close to unity intended best fit with Langmuir adsorption.
- Cathodic inhibition was projected from polarisation technique.
- Increase in R_{ct} values increased inhibition efficiency.
- Proposed mechanism favoured higher inhibition for aromatic than aliphatic moieties.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 15 July 2017

Received in revised form 10 December 2017

Accepted 14 January 2018

Keywords:

Polyesters
Langmuir adsorption
Simulated concrete pore
Cathodic inhibition
Barrier

ABSTRACT

As a motto of framing the strategies to minimise the deterioration in steel reinforced concrete structures as well as the fact that the work carried out with polymers are rare and scanty, polyesters namely Poly (Glycerol azealate) (PGAZ) and 4-(1-(4-methoxy phenyl) cyclo hexyl)phenyl 9-oxodecanoate (MPOD) were synthesised and evaluated for rebar corrosion in simulated concrete pore solution (SCP) for the first time at selected concentrations of 10, 100, 1000 ppm levels with an aid of electrochemical impedance and potentiodynamic polarisation techniques. Nyquist plots favoured increased R_{ct} values suggesting the formation of barrier. Tafel plots represented cathodic inhibition. The experimental datas were best fitted with Langmuir adsorption isotherm facilitating monolayer adsorption. Both the methods employed were in good agreement with each other favouring maximum inhibition efficiency of 71.81% for MPOD and minimum of 57.64% for PGAZ primarily due the difference in aromatic sites as revealed in mechanism.

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1. Introduction

The deterioration of reinforced concrete structures has been internationally recognised holding huge expenditure to overcome. In general, protection of steel bars are carried out in alkaline incubator combined with coatings or inhibitors to minimize its degradation when exposed to corrosion inducing factors like elevated

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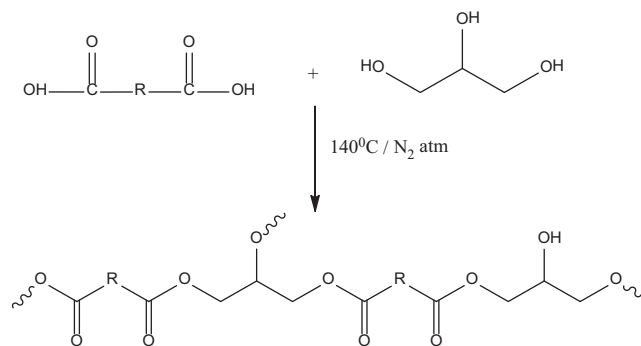
temperature and relative high humidity. In specific, cathode and anode reactions were initiated with pronounced corrosion rate and decreased alkalinity when exposed to carbonation and chloride induced medium [1]. Steel reinforced with concrete has gained vast usage all around the world in designing engineering structures, highways due to its precise strength, durability and bonding ability under sustained conditions. Though the steel reinforcements makes the possibility of governing wide span buildings, the pronounced effect of chloride ions in the atmosphere stands as a primary hindering and challenging factor for civil engineers in limiting the durability of concrete structures [2,3] which was evident from the deterioration effects observed in coastal areas [4]. The catalytic nature of chloride induced corrosion seem to be difficult to terminate but can be minimised. The corrosion problem in reinforced concrete has been extensively investigated by ample researchers and the foremost cost-effective solution of adding inhibitors has been formulated which can be proceeded either preventively or restoratively [5]. Concrete is a form of composite material composed of coarse and fine granular material (aggregate/filler) embedded in hard matrix of material (cement/binder) which fills the space and glues them together. Observation revealed that the cement concrete is a widely used one mainly consisting of tricalcium Silicate (C3S), dicalcium silicate (C2S), tricalcium aluminate (C3A) and clinker phase (C₄AF). On the other hand, pores within the concrete filled with saturated Ca(OH)₂ solution and other alkalines such as NaOH and KOH provided high pH to the concrete environment. Though the steel has a tendency to get corroded, but when reinforced with concrete, physico-chemical protection is favoured by the alkaline environment of the concrete medium thereby generating high pH level of 13, which is a desired level to form a thin passive oxide layer on the metal surface to reduce the metal dissolution [6]. The inability of concrete in forming passive layer arises when the pH value of pore solution goes below 9 resulting in cracking and spalling of concrete structures [7,8]. The main factors inducing reinforced corrosion are carbonation and chloride ingress [9] which mainly depends on nature of electrolyte, oxidising agent and potential difference generated on reinforced bar. Carbonation corrosion is favoured when a chemical reaction gets initiated between carbon dioxide from the atmosphere and the hydration product of cement Ca(OH)₂ in concrete causing a reduction in the alkalinity of concrete thereby neutralising the pH of the pore solution from 14 to 9 [10]. In case of chloride corrosion, tricalcium aluminate (C3A) and calcium aluminoferrite (C₄AF) in the concrete reacts with chloride to form insoluble calcium chloroaluminates and calcium chloroferrites where the chloride is bonded in non-active form [11]. However some active soluble chlorides which remains in equilibrium in the aqueous phase of concrete primarily decides the risk of corrosion depending on its concentration. Though the chloride ion is not consumed in the process, it catalyses the corrosion reaction by depassivating the protective layer Fe₂O₃ thereby initiating quick degradation of properties [10] leading to spalling and cracking finally [12]. The corrosion in reinforced steel is an unavoidable one as it is mainly triggered due to the environmental factors like temperature, humidity, chloride content, pH, interfacial voids as well as potential of steel [13–15] which plays an important role in premature failure of buildings till date. It is noticeable that nitrite based inorganic inhibitors were banned due to its carcinogenic nature as reported by ShaoBo Jiang et al., [16]. Organic inhibitors [17] such as N, N'-Dimethyl aminoethanol, diethanolamine, amines, alkanolamines and carboxylates were studied as inhibitors for rebar corrosion whose synthetic strategy and toxic nature limits its usage. Besides other class of inhibitors, polymers has gained detailed insight due to its multiple adsorption sites, facile synthesis, low cost and capability of adsorbing larger surface [18]. In order to overcome these limitations as well as the fact that limited

research work has been carried out on polymers like poly (vinyl pyrrolidone), poly ethylamine, polyaniline, polyethylene glycol, polysiloxane, poly vinyl alcohol, polyethylene glycol methyl ether in reinforced concrete corrosion, this present work has been undertaken to explore the response of aliphatic and aromatic polyesters in alkaline medium with a detailed discussion of mechanism correlating well with other techniques.

2. Experimental methods

2.1. Synthesis of poly (glycerol azealate) (PGAZ) polyester

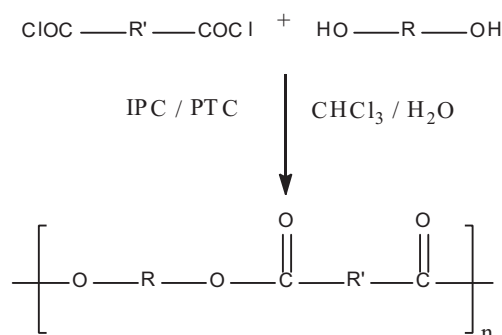
Polymerisation was initiated by charging a two necked round bottomed flask with azelaic acid (0.1 M) and glycerol (0.1 M) in 1:1 ratio and refluxing it for a specified time at 140 °C under the nitrogen atmosphere till the solution becomes homogeneous. It was further equipped with constant mechanical stirring until a viscous product is obtained [19] as illustrated in the scheme.



where R = $-(\text{CH}_2)_7-$

2.2. Synthesis of 4-(1-(4-methoxy phenyl) cyclo hexyl) phenyl 9-oxodecanoate (MPOD) polyesters

Interfacial polycondensation was initiated by introducing 0.025 M sodium hydroxide dissolved in 75 ml of water into a 250 ml beaker accompanied with a mechanical stirrer with a moderate speed. Further the reaction mixture was added with phase transfer catalyst sodium lauryl sulphate (0.25 mg) and diol monomer (0.0125 M) formed by following the procedure reported earlier [20]. Subsequently, diacid chloride (0.0125 M) generated from thionyl chloride and azelaic acid in presence of pyridine was added with a maximum stirring rate. After gentle stirring for few minutes, the polymer formed as illustrated in the scheme was filtered and washed with acetone to remove unreacted monomers. The resulting cardo polyesters were finally dried in vacuum [21]>



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