APPLICATION OF LINEAR POLYESTERS AS CORROSION MITIGATORS FOR MILD STEEL, REBAR, TITANIUM ALLOY AND AS ELECTROLYTE IN ENERGY STORAGE DEVICES

Thesis submitted to the Bharathiar University in partial fulfillment of the requirements for the award of the degree of

DOCTOR OF PHILOSOPHY

IN

CHEMISTRY

By

Mrs. C. NUSRATH UNNISA, M.Sc., M.Phil.,

a doctoral fellow

Under the guidance of

Dr. (Mrs.) SUBRAMANIAN CHITRA, M.Sc., M.Phil., Ph.D.,

Associate Professor and Head

DEPARTMENT OF CHEMISTRY

PSGR KRISHNAMMAL COLLEGE FOR WOMEN

College of Excellence 2018 - nirf-16th Rank

(An Autonomous Institution-Affiliated to Bharathiar University)

(Reaccredited with 'A' Grade by NAAC)

An ISO 9001: 2015 Certified Institution

Coimbatore – 641 004

AUGUST - 2018

CHAPTER - X

SUMMARY AND CONCLUSIONS

The research work documented in this thesis broadly consists of two parts, ie., part A and part B.

Part-A of this thesis reveals the brief introduction about the importance, causes, factors that are responsible for corrosion followed by the classification and monitoring methods in chapter I.

A series of linear aliphatic and aromatic polyesters were synthesised and structurally confirmed by FT-IR and NMR spectral analysis. TGA and DSC analysis was made to reveal its thermal response as reported in chapter II.

The synthesised aliphatic and aromatic polyesters were evaluated for their anti-corrosive potential towards mild steel in 0.5 M H_2SO_4 medium and presented in chapter III and chapter IV. Both the series resulted similarly with increased inhibition efficiency on increasing the concentration in the range of 10, 50, 00, 500, 1000 ppm. Obtained data for all the polymers were best fitted with Langmuir adsorption isotherm. Influence of temperature in the range of 303 K -333 K resulted with decrease in inhibition efficiency favouring physisorption mechanism. Thermodynamic and activation parameters like E_a, ΔG^o_{ads}, ΔH^o_{ads} and ΔS^o_{ads} were calculated. Selected concentrations of the inhibitors were involved in electrochemical studies where AC impedance measurements were associated with increased R_{ct} and potentiodynamic polarisation techniques were associated with decreased I_{corr} values suggesting mixed type of inhibition. Techniques adopted stair cased the inhibition efficiency of aliphatic polyesters in the order of PGSE > PGAZ > PGSU > PGP > PGA > PGG > PGS > PGM with a maximum inhibition efficiency of 77.63 % for PGSE at 1000 ppm due to +I effect and its moderate efficiency was due to the absence of aromatic sites and heteroatoms like N, P and S. Similarly the cardo polyesters employed can be arranged in the order of MPOU > MPOD > MPON > MPOO > MPOHP > MPOHX > MPOP > MPOB with a maximum inhibition efficiency of 92.98 % for MPOU which was due to the aromatic pi-electron density along with +I effect of methylene moieties. To confirm the adsorption of aliphatic and aromatic polyesters on the metal specimens, surface morphological studies were carried out using FT-IR, XRD, SEM, EDS, AFM and XPS techniques. Morphological studies was also performed with a suitable mechanistic information.

Chapter V was targeted towards enhancing the inhibition efficiency of linear polyesters by dispersing the nano fillers like SnO, ZnO and CuO metal oxides with the polymeric back bone using ethanolic extract of *Persia Americana* (avocado) seeds along with their respective salts. The dispersion of nano fillers was confirmed from FT-IR, XRD, SEM-EDS and TEM analysis. Pronounced inhibition efficiency upto 97 % was noticed compared with parent polyesters (PGM, PGS, PGG). Smaller size of nano fillers would have provided larger surface area in which CuO predominates than ZnO and SnO nano dispersion.

Chapter VI has been designed as a comparative approach, where selected inhibitors were tested for its inhibition efficiency for mild steel corrosion in 1 M HCl, 0.5 M H_2SO_4 and 0.3 M H_3PO_4, out of which 1 M HCl favoured better inhibition than rest of the acids resulting in the order 1 M HCl > 0.5 M H_2SO_4 > 0.3 M H_3PO_4.

Study of selected inhibitors PGAZ, PGSE, MPOB and MPOU in corrosion inhibition of rebar in simulated concrete pore solution has been portrayed in chapter VII. Electrochemical investigations carried out in chloride induced medium resulted in the order MPOU > MPOD > PGSE > PGAZ. The surface covered by the inhibitors was predicted using SEM analysis.

The degradation behaviour of Ti-6Al-4V alloy in simulated body fluid medium has been discussed in chapter VIII. Electrochemical impedance and potentiodynamic polarisation studies carried out in the absence and presence of selected inhibitors (PGSE and MPOU) showed increased resistance and decreased I_{corr} value. The surface coverage by the added additives was confirmed by the SEM-EDS analysis.

Chapter IX shown in part B of this thesis, reported the film forming capability of poly (glycerol suberate) polyester with poly vinyl alcohol. Addition of various concentrations of the dopant increased the conductivity. FT-IR, XRD, DSC and SEM-EDS analysis were used to characterise the prepared electrolytes. Free standing films obtained at an optimum concentration of 1g PVA: 0.75g PGS: 0.6g NH_4SCN resulted with highest conductivity of 3.01 x 10^{-4} Scm^{-1}. Under external load, a drop in OCV was noticed from 1.51 V to 1.46 V which was stable up to 70 hours.

Summary and conclusions of all the chapters are presented in chapter – X followed by scope for future work in chapter – XI.

CHAPTER - XI

SCOPE FOR FUTURE WORK

The following ideas can be implemented for future work,

- Synthesised polyesters can be tested for the inhibiton of various metals like copper, aluminium, brass and pipe line steel.
- Besides immersion tests, coating of inhibitors on the metal specimens can be adopted for better adhesion.
- Inhibition efficiency can be studied under different electrolytic medium.
- Microencapsulation of polymers can be studied.
- Entrapping different nano-metal oxides from natural extracts and blending with polyesters can be done.
- Polymer electrolyte film can be used in lithium based batteries and can be extended to fuel cells.
- Anticorrosive potential of the synthesised inhibitors evaluated under lab scale can be extended towards metal industries.