

SUMMARY AND CONCLUSION

The use of environment friendly compounds for the corrosion inhibition studies has gained much attention in the recent years because of the environmental concerns. In this aspect, a versatile and non- toxic polymer Chitosan was modified into its Schiff base derivatives for the evaluation of Corrosion resistance of mild steel in 1M HCl. The synthesized eco-friendly Chitosan Schiff bases were also investigated for a preliminary study on scale removal of CaCO_3 in synthetic water samples. The outcomes of the study can be briefed as follows:

- The yield of synthesized Chitosan Schiff bases was about 60-65% since the degree of deacetylation is 76.99% by which substitution of aldehydes in the Chitosan matrix may not be possible at 100%.
- The elemental and spectral studies (UV and FTIR) confirmed the incorporation of aldehyde groups in the Chitosan backbone. The increase in C/N ratio from elemental analysis and the presence of imine vibrations in the FTIR spectra of Chitosan Schiff bases clearly evident the formation of Schiff base in the polymer matrix.
- The thermal stability of the Chitosan Schiff bases were found to be lesser than the native Chitosan which may be due to the anchoring of aldehyde groups into the Chitosan matrix.
- The weight loss technique revealed that all the studied Schiff base polymers exhibited 90% IE for the concentration of 1500 ppm at 12 hours of immersion. Schiff base polymers showed a consistent inhibition performance up to 333 K with a slight decline at 343 K.
- The performance of the Schiff bases was analyzed by estimating Fe^{2+} ions in the solution left after weight loss experiments using AAS studies. The results are in agreement with weight loss results. The UV analysis of the above solution proved the formation of Fe-complexes in the solution.
- The parallel nature of the Tafel curves obtained in the potentiodynamic polarization method suggested the activation controlled mechanism of inhibition. The reduction in

cathodic, anodic current densities and corresponding current density (i_{corr}) and also negligible E_{corr} values collectively suggested Chitosan Schiff bases as mixed type inhibitors.

- From the results of impedance measurements, a single semicircle representation of the Nyquist plot corresponding to one time constant in the Bode plot implies that the metal dissolution process is controlled by a single transfer process. The diameter of the semicircle in the Nyquist plot and the magnitude of the Bode modulus increased with increasing concentration of the inhibitors that suggested the formation of a protective layer on the metal surface. The impedance $|Z|$ as well as the phase shift (θ) of mild steel increased with increase in concentration because of the adsorption of inhibitor molecules on the active sites of the metal surface.
- Basic information on the interaction of the inhibitor molecules and the metal surface was provided by adsorption isotherms. The best fit was obtained with Temkin isotherm with R^2 close to unity for all the investigated inhibitors. The larger K_{ads} values suggested the greater inhibition efficiency and more stable protective layer with better inhibition performance. The ΔG_{ads} values are negative and greater than 40 kJ mol^{-1} , ΔH_{ads} values are positive and greater than 100 kJ mol^{-1} indicated the chemical mode of adsorption of the inhibitors. Similarly, the values of ΔS_{ads} confirmed the orderly arrangement of inhibitor molecules.
- The adsorption of Chitosan Schiff bases on the mild steel surface was explained by cyclic voltammetric studies. The absence of redox peaks and also the existence of strong droop in presence of inhibitors suggested the protective layer formation. The reduction in ΔQ (charge accumulation) with immersion time confirmed the effective adsorption of Schiff base polymers on the metal surface.
- The formation of protective layer on the metal surface was confirmed using SEM, EDX, AFM, contact angle measurements, FTIR and XPS techniques. SEM technique revealed the extensive coverage of polymeric clusters on the metal surface. The atomic percentage of elements observed from the EDAX spectrum indicated the presence of nitrogen and sulphur atoms that confirmed the formation of protective layer on metal surface. AFM studies revealed a homogeneous film formation by the inhibitors on the metal surface. The film formation was further confirmed by the increase in hydrophobicity (increased contact angle) of the metal surface exposed to

inhibitor solution. The main confirmation of the Fe-inhibitor bonding was confirmed by FTIR and XPS analysis. Both the technique revealed the presence of Fe-N bonding.

- Quantum chemical calculations viz., semi-empirical and DFT methods were employed to identify the active adsorption sites responsible for the inhibition. Both the studies revealed that imine group and hydroxyl group on the Schiff base polymers as electron donating sites with high E_{HOMO} . Moreover, the presence of Mulliken charges on the hetero atoms of the inhibitors also suggested the coordinating sites responsible for the inhibition.
- Based on the experimental and theoretical results, the plausible mechanism for the inhibition action of Chitosan Schiff bases was proposed with some supporting evidences.
- The Chitosan Schiff bases are found to be capable of inhibiting CaCO_3 scale formation in synthetic water samples. 400 ppm concentration of inhibitors with 6 h of bath time and at 65 °C of bath temperature effectively inhibited the scale formation. The changes in the crystal morphology were studied using SEM analysis and the thermal stability of the scale crystals were analyzed using TGA analysis.