CHAPTER-VI

CONCLUSION

The inhibition of corrosion on mild steel in 1 N sulphuric acid by substituted piperidin-4-one with semicarbazone and thiosemicarbazone have been investigated by various monitoring techniques such as weight loss, potentiodynamic polarization, Quantum Chemical Parameters Calculations and electrochemical impedance spectroscopic methods. The structure of the compounds studied by FT-IR and NMR spectroscopy. From these studies various conclusions were drawn out which are as present as below.

The inhibiting efficiency and corrosion rates with varying concentration (0.2, 0.4, 0.6 and 1mM) of inhibitors on mild steel in 1 N H₂SO₄ have been determined by weight loss technique. It has been found that the efficiency increases while the corrosion rate decreases with increase in concentration of inhibitors. Also very high inhibition efficiencies were found in the compounds even at very low concentration of inhibitors, suggesting that they act as efficient inhibitors for the corrosion of mild steel.

Effect of temperature on inhibition has also been studied at temperatures 303, 313, 323 and 333 K and it has been found that inhibition efficiency increases with respect to increase in temperature. The large negative values of free energy of adsorption (-ΔG_ads) 33-41 KJ/mole indicate the spontaneity of adsorption and support chemisorptions mechanism.

Potentiodynamic polarization studies were for the corrosion of mild steel in 1N H₂SO₄ solution with and without inhibitors. Various parameters such as corrosion potential (E_corr) corrosion current (I_corr) and Tafel constants (b_a and b_c) were obtained from this study. The effect of concentration of inhibitors on these parameters has been studied. The decrease in I_corr values with the increase in concentration of inhibitors suggesting their inhibiting ability. The tafel constants b_a and b_c were not much affected by the change in concentration and
the $E_{\text{corr}}$ values were shifted to less negative side. All these observations acts as a mixed type inhibitors with more cathodic behavior.

The corrosion behavior of inhibitors has been studied by electrochemical impedance spectroscopy method. Parameters like charge transfer resistance ($R_t$) and double layer capacitance ($C_{dl}$) were obtained. The impedance diagrams were found to be almost semicircular. The value of charge transfer resistance indicates the electron transfer across the interface. The results of the effect of concentration of inhibitors on the $R_t$ and $C_{dl}$ support the adsorption mechanism.

Thus, all the three methods give similar results. The surface coverage ($\theta$) for various inhibitor concentrations (C) has been calculated. The experimental data were found to obey Langmuir adsorption isotherm.

Even though these compounds contain ring nitrogen the main inhibition is due to >N-NH-CO-NH₂ and >N-NH-CS-NH₂.

The order of inhibition efficiency of (01SC), (02SC), (03SC), (01TS), (02TS) and (03TS) in the 1 N Sulphuric acid environment was found to be (01TS) > (02TS) > (03TS) > (01SC) > (02SC) > (03SC).

Variously substituted piperidin-4-ones with thiosemicarbazone have more inhibited character than variously substituted piperidin-4-ones with semicarbazone because more electronegativity factor and greater polarizability of the sulfur atom than oxygen atom.

Standards of the synergistic parameters ($S_I$ and $S_0$) show that the corrosion inhibition developed by the substituted 2,6-diphenyl piperidi-4-ones with semicarbazone and thiosemicarbazone with halide ions and metal ions fusion is synergistic in nature. The influence of I⁻, Br⁻ and Cl⁻ anions raise the inhibition efficiency of the substituted 2, 6-diphenyl piperidin-4-ones with semicarbazone and thiosemicarbazone due to synergistic effect. The synergistic effect of halide ions is formed in the following order: KI>KBr>KCl. The high synergistic effect of iodide ion over the others is
attributed to its large ionic radius, high hydrophobicity and low electronegativity. The synergistic effect metal ions is more with Zn$^{2+}$ ions than Cd$^{2+}$ ions.

Antibacterial activity of (01SC), (02SC), (03SC), (01TS), (02TS) and (03TS) was determined using the disc diffusion method. The zone of inhibition was observed in bacteria (*Pseudomonas aeruginosa, Salmonella sp. Klebsiella pneumonia, Staphylococcus aureus*) at different concentration. The zone of inhibition efficiency rises with increase the concentration of the compounds. The bacteria *Staphylococcus aureus* exhibits the highest zone of inhibition efficiency with the compounds (02SC), (01TS) and (02TS) (16mm) in concentration 100 µg/ml. In all the above organism *Klebsiella pneumonia* has the highest zone of inhibition efficiency rises in the compounds (02TS), (03SC) and (02TS).

The FT-IR, $^{13}$C NMR and $^1$H NMR studies of variously substituted piperidin-4-ones with semicarbazones and thiosemicarbazones were analyzed to conclude the structure of the compounds.

The molecular structure and vibrational spectra of inhibitors have been calculated with the help of density fundamental theory (DFT) using B3LYP/6-31G basis sets. The solid phase FT-IR and FT-Raman spectra of inhibitors recorded in the range 4000-400 cm$^{-1}$ and 4000-100 cm$^{-1}$ respectively. Computed and experimental parameters, the vibrational frequencies of the inhibitors have been compared. The difference between the observed and scaled wave number values of the fundamentals are very small.

With the help of density functional theory calculations, the most optimized geometrical structures were predicted. Quantum chemical calculations help us to identify the structural and symmetry properties of the chosen compounds. The value of $E_{\text{HOMO}}$ of alkyl substituted piperidin-4-ones with semicarbazone and thiosemicarbazone is almost the same. The results
good agreement with calculated values. The calculated HOMO-LUMO energies also show that charge transfer occurs within the molecule. The energy gap decreases, the reactivity of the molecule increases, leading to increase in the percentage of inhibition efficiency molecule. The high value of the dipole moment, probably increases the adsorption between chemical compounds and metal surface.
Scope for future work

These inhibitors may be tested as a corrosion inhibitor for metals and alloys other than mild steel in acids and neutral environments and also for mild steel in acids apart from \( \text{H}_2\text{SO}_4 \) in neutral environments.