

**CHAPTER (2)**

**LITERATURE REVIEW**

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### **2. LITERATURE REVIEW**

Nanoparticles have finding applications in a variety of situations or some of these applications are in well established technologies where the role of nanoparticles is to improve efficiency/effectiveness. More exciting applications of nanoparticles are however in those technologies which could have not been even conceived without nanoparticles. One therefore expects demand for nanoparticles to increase rapidly. A variety of techniques have been developed to synthesize nanoparticles using bottom to top approach. Some of these are in quite widespread use but none of them is understood quantitatively a prerequisite for their large scale synthesis. [Perala, 2006]

Singh *et al.*, (2009) has reported the reduction of silver and gold salts by methanolic solution of sodium borohydride in tetrazolium based ionic liquid as a solvent at 30 °C leads to pure phase of silver and gold nanoparticles. Silver and gold nanoparticles so-prepared were well characterized by powder X-ray diffraction measurements (XRD), transmission electron microscopy (TEM) and Quasi-elastic light scattering (QELS. XRD) analysis revealed all relevant Bragg's reflection for crystal structure of silver and gold metal. XRD spectra also revealed no oxidation of silver nanoparticles to silver oxide. TEM showed nearly uniform distribution of the particles in methanol and it was confirmed by QELS. Silver and gold nanoparticles in ionic liquid can be easily synthesized and are quite stable too.

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Liu *et al.*, (2007) made a new method to prepare silver nanoparticles, which based on aqueous-gaseous phase reaction of silver nitrate solution and ammonia gas. The proposed method is effective, rapid, and convenient. Transmission electron microscope (TEM) and X-ray diffraction (XRD) were used to characterize the structure of the particles, respectively. It was found that the average diameter of the particles was about 10 nm and the shape was spherical. And the strong fluorescence signal of the silver nanoparticles solution can be quenched after the adding of the calf thymus DNA (*ct*-DNA) solution. The quenched fluorescence intensity was linear with the concentration of *ct*-DNA in the range of 0.5 to  $5.0 \times 10^4$  ng mL<sup>-1</sup> with a detection limit of 0.3 ng mL<sup>-1</sup>.

Torreggiani *et al* (2009) are concerned with the surface enhanced Raman scattering (SERS) activity of silver colloids obtained by a radiolytic method. Ag nanoparticles were successfully prepared by  $\gamma$ -radiolysis of Ag<sup>+</sup> aqueous solution containing *t*-BuOH or *i*-PrOH at room temperature without the addition of aggregating or stabilizing substances. The metal colloids were characterised by UV/vis spectroscopy and scanning electron microscopy. Many experimental conditions were tested (i.e. Ag<sup>+</sup> concentration, dose and  $\cdot$ OH scavenger alcohol) in order to obtain the best controlled size of nanoparticles as well as the high stability of colloidal silver with time. The use of relatively low irradiation doses and Ag<sup>+</sup> concentrations allowed obtaining very stable suspensions of Ag nanoparticles without adding any colloid stabilizer, a source of further spurious bands in the Raman spectra. The suitability of the  $\gamma$ -irradiated colloids in SERS spectroscopy was tested by using thiram, a known fungicide. Micro-SERS and SERS spectra of good

quality was achieved at very low concentration of adsorbate, without the overlapping of impurities normally present in conventional citrate colloidal suspensions of Ag nanoparticles.

**Yoksana and Chirachanchai (2009)** have studied the fabrication of silver nanoparticles in chitosan–aqueous acetic acid solution using  $\gamma$ -ray irradiation under simple conditions, i.e., air atmosphere. The formation of silver nanoparticles is confirmed by the specific surface Plasmon resonance (SPR) band and electron micrograph. The effects of  $\gamma$ -ray dose (2.5–25.0 kGy), chitosan concentration (0.1 and 0.5 %, w/v) and silver nitrate content (0.02–0.10 m mole) on the particle size and particle number are also investigated. In addition, the antimicrobial activity of the silver nanoparticles is studied.

**Sileikaite *et-al.*, (2006)** have produced silver colloid by chemical reduction of silver salt (silver nitrate  $\text{AgNO}_3$ ) solution. As a reducer sodium citrate was used. UV-VIS spectrometry indicated formation of nanoparticles. The surface plasmon resonance peak in absorption spectra of silver colloidal solution showed an absorption maximum at 450 nm. Comparison of theoretical (Mie light scattering theory) and experimental results showed that diameter of silver nanoparticles in colloidal solution is about 100 nm. Silver nanoparticles were deposited on two substrates: silica ( $\text{SiO}_2$ ) and polyethylene terephthalate (PET) modified with various polymers (polycarbonate (PC), polymethyl methacrylate (PMMA), polystyrene (PS)). The colloidal silver was incorporated by dip-coating to the polymer-substrate structures. X-ray fluorescence spectroscopy (XRFS) and atomic force microscopy (AFM) results indicate that produced structures include silver nanoparticles. It was found that during

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deposition, silver nanoparticles forms aggregates on the surface. The size of aggregates varied from 240 nm to 400 nm.

**Kim (2007)** has prepared Nanoparticles of silver by reduction of silver ions in the presence of polymer. In particular, silver nanoparticles were prepared by reduction of silver nitrate in ethanol in the presence of chemical reduction and poly [*N*-vinylpyrrolidone (PVP)] as a protective agent. The UV-visible spectrum of the material shows a strong plasmon resonance band centered at 402 nm. The band position depends on PVP: silver nitrate weight ratios of silver colloids were influenced by the protective agent was absorbed to the particle surface. The rate constant remarkably depends in a first order on PVP: AgNO<sub>3</sub> weight ratios and reaction temperature.

**Azzam et al (2009)** have studied the self-assembling of some synthesized thiol surfactants namely (mercaptopropane-, mercaptohexane, mercaptooctane, and mercaptodecane sodium sulfonate) on the fabricated gold nanoparticles. The self-assembling of these surfactants on gold nanoparticles characterized using different techniques such as FTIR spectroscopy, UV spectroscopy, and transmission electron microscopy (TEM). Spectroscopic evidence suggests that the synthesized thiol surfactants have been attached to the gold nanoparticles. The effect of self assembling of these surfactants on the size of the gold nanoparticles was studied using TEM images. The growth of the gold nanoparticles was investigated with respect to the increase of alkyl chain in the synthesized thiol surfactants. The results show that the stabilization of gold nanoparticles was affected by the increase in alkyl chain length of these surfactants. The effect of gold nanoparticles on the interfacial

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tension and the emulsion stability of these surfactants with crude oil were studied.

**Zhou et al., (2007)** have synthesized three new aliphatic thiol surfactants by reacting alkyl bromide with hexamethyldisilathiane under a mild condition. This approach provides an easy access for the direct synthesis of various different length thiol surfactants which play a crucial role in tuning the properties of gold nanoparticles. Gold nanoparticles encapsulated with one of our synthetic thiols were prepared and well characterized by <sup>1</sup>HNMR, UV-vis, FT-IR, and TEM. The hybrid nanoparticles are very stable in both organic solvents and the solid state.

**Azzam et al (2008)** have investigated the self-assembling behaviour of the synthesized dodecylcysteine hydrochloride surfactant comparing with that of cysteine compound on the prepared gold nanoparticles using transmission electron microscope (TEM) measurements. The effect of self-assembling of this surfactant on the size of gold nanoparticles was studied using TEM images.

**Zhang et-al., (2003)** reported A novel method for the assembly of metal nanoparticles, in which polymer microspheres have been employed as both stabilizers and transport vehicles. We prepared sulfonated polystyrene microspheres and then introduced silver ions into the microspheres by ion exchange. Dispersal of the polymer microspheres in DMF led to the silver ions in them being reduced by the solvent; the resulting Ag nanoparticles were stabilized by the polymer network. In aqueous solution, the polymer microspheres are negatively charged and,

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by using layer-by-layer method, the prepared Ag nanoparticles can be self-assembled into films using the microspheres as transport vehicles.

Djoković *et-al.*, (2009) investigated Adsorption of sulfide ions onto a surface of starch capped silver nanoparticles upon addition of thioacetamide. UV-vis absorption spectroscopy revealed that the adsorption of the sulfide ion on the surface of the silver nanoparticles induced damping as well as blue-shift of the silver surface plasmon resonance band. Further increase in thioacetamide concentration led to shift of the resonance band toward higher wavelengths indicating the formation of the continuous Ag S layer on the silver surface. Thus fabricated nanoparticles were investigated using electron microscopy techniques (TEM, HRTEM, and HAADF-STEM) and X-ray photoelectron spectroscopy (XPS), which confirmed their core-shell structure.

Sarkar et al (2009) have reported Solution-phase synthesis of nanometer- sized silver particles by sodiumborohydride reduction of AgNO<sub>3</sub>. Two isomericdicarboxylates (sodium maleate and sodium fumarate) have been used as stabilizing agents the dicarboxylate stabilized silver nanoparticles are characterized by UV–vis spectroscopy, high resolution transmission electron microscopy (HR-TEM) and fourier transform infrared (FTIR) spectroscopy. A qualitative comparison is made between the UV–vis spectral characteristic of the SPR band and the simulated curve obtained from modified Mie’s theory Silver nanoparticles prepared using these two isomeric dicarboxylates show intense fluorescence in the visible region. DFT with hybrid functional (B3LYP) based frequency (IR) calculation of both the dicarboxylates and

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the respective nanoparticles are in good agreement with the experimental IR frequenc On the basis of this calculation amodel has been proposed for the stabilization of silver nanoparticles by these two isomeric dicarboxylate anions

**de Paz Banez *et-al.* (2000)** had synthesized A series of new cationic polymeric surfactants, comprising a short hydrophobic n-alkyl ( $C_{12}$  or  $C_{16}$ ) end-group and a long hydrophilic sequence of 17–68 2-(dimethylamino)ethyl methacrylate (DMA) units, by oxyanion-initiated polymerization. A previously titrated solution of  $DMSO \cdot K^+$  was used for quantitative formation of the potassium alcoholate initiators from the corresponding  $C_{12}$  or  $C_{16}$  alcohols. Optimum polymerization conditions were found to be at or above room temperature, using THF as solvent. Under these conditions the polymers had relatively narrow polydispersities ( $M_w: M_n \approx 1:25$ ) and both GPC and NMR spectroscopy showed no residual monomer. NMR were studied of the surfactants in  $CDCl_3$  also confirmed that good control over the degree of polymerization was achieved, indicating very high initiator efficiency. Cloud points of aqueous solutions of selected polymeric surfactants were determined by turbidimetry and decreased with increasing DMA chain length, as expected.

**Sayyah *et al* (2006)** have synthesized new aniline derivative surfactants where Meta-hydroxyaniline was reacted with decyl bromide, dodecyl bromide, and cetyl bromide to produce new aniline derivative surfactants. The monomers (0.4 mol=L) were polymerized in aqueous solution of hydrochloric acid (1.0 mol=L) using sodium dichromate (0.1 mol=L) as oxidant at room temperature. The crystallinity of the prepared

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polymer samples was investigated by X-ray diffraction and electron microscopic analysis. The thermal behavior of these polymers was studied by thermogravimetric analysis (TGA), also the electrical properties of the obtained polymer samples were measured and discussed. The a.c.-electrical conductivity was found to be increased by the increase of number of carbon atoms in the substituted alkyl group in the following order (cetyl > dodecyl > decyl).

**Wang et al., (2009)** have synthesized Polyaniline (PANI) “sunflowers” made of arrays of oriented nanorods by chemical oxidative polymerization of aniline in the presence of cetyltrimethylammonium bromide (CTAB) and suitable concentration of  $\text{HNO}_3$  at about  $0^\circ\text{C}$  (ice bath). The reaction conditions, such as the concentration of reagents and reaction temperature were systematically investigated and controlled on the preparation of PANI “sunflowers”. The results also suggest that  $\text{HNO}_3$  probably plays a key role in forming PANI “sunflowers”. A possible forming mechanism of the PANI nanostructures is offered.

**Xia and Wang, (2001)** had prepared Polyaniline (PANI) nanoparticles through ultrasonic assisted inverse micro emulsion polymerization method. Polymerization of aniline was confined to a nanoreactor named ‘water pool’ surrounded by surfactant molecules in the apolar continuous phase. The size of the PANI nanoparticles decreases with the decrease of the  $w$  value. The spherical nanoparticles (10–50 nm) can further form the uniform sub micrometer aggregates with a size of  $200 \sim 400$  nm induced by ethanol, and the size of the aggregate decreases with the decrease of the  $w$  value. The morphology of aggregates as well as aggregation behavior of PANI nanoparticles were characterized

by TEM. The polymerization rate, UV–vis absorption spectra, FTIR spectra, XRD, as well as the conductivity were examined at different [water] / [surfactant] molar ratio, i.e.  $w$  value. Ultrasound enhances the polymerization rate of aniline that is usually very slow under conventional stirring in inverse micro emulsion and contributes to produce spherical nanoparticles. Also, ultrasound irradiation promotes the diffusion of HCl molecules and improves the degree of doping. Polymerization of aniline occurred in the confined nanoreactor in micro emulsion and strengthens the hydrogen-bonding of amine and imine of PANI molecular chains, which improves the degree of crystallinity. The conductivity of obtained PANI is in the magnitude of  $10^{-1} \text{Scm}^{-1}$ , and is changed with  $w$  value.

**Yan and Toshima., (1995)** had performed Polymerization of aniline and its derivatives by using the cerium (IV) sulfate as an oxidant instead of conventional ammonium peroxodisulfate. Reaction conditions were optimized with emphasis on conductivity and yield. Reaction of 3,5-dimethylaniline and 2,3-dimethylaniline by using cerium(IV) sulfate gave the polymeric products, while no polymeric products were obtained by using the ammonium peroxodisulfate as an oxidant. The polymers were characteristic of Emeraldine structure on the basis of IR spectra. The partially belt-like morphology of the polyaniline, when prepared in an acidic medium, was observed by scanning electron microscope.

**Bouazza et al., (2009)** have carried out the chemical deposition of silver particles in polyaniline (PANI) powder via the reduction of  $\text{Ag}^+$  ions by PANI in various concentrations of  $\text{AgNO}_3$  aqueous solutions. It is found that the rate of Ag (I) reduction and the size of the metal particles

incorporated were strongly dependent on the reaction medium, the PANI redox form and the stirring method used. Homogeneous distribution of silver nanoparticles into PANI matrix was obtained at low Ag (I) concentration, PANI in emeraldine base form and short reaction time under ultrasonic stirring. The presence of silver particles dispersed into porous polyaniline structures was confirmed using X-ray diffraction, scanning electron microscopy and cavity microelectrode (CME) technique in acidic aqueous electrolyte. The electrochemical study of Ag-PANI composite by CME showed that the redox system of silver depends on the size and the distribution of metal particles incorporated into PANI.

Vimala et-al, (2009) have reported the preparation of semi interpenetrating hydrogel networks (SIHNs) based on cross-linked poly (acrylamide) prepared through an optimized rapid redox-solution polymerization with N,N0-methylenebisacrylamide (MBA) in presence of three different carbohydrate polymers, namely gum acacia (GA), carboxymethylcellulose (CMC) and starch (SR). Highly stable and uniformly distributed silver nanoparticles have been obtained with hydrogel networks as nanoreactors via in situ reduction of silver nitrate ( $\text{AgNO}_3$ ) using sodium borohydride ( $\text{NaBH}_4$ ) as reducing agent. The formation of silver nanoparticles has been confirmed with ultraviolet visible (UV-vis) spectroscopy, Fourier transform infrared (FT-IR) spectroscopy, X-ray diffraction (XRD) analyses. Thermogravimetric analysis (TGA) provides the amounts of silver nanoparticles exist in the hydrogel networks. Transmission electron microscopy (TEM) results demonstrate that acacia employed hydrogels have regulated the silver nanoparticles size to 2–5 nm where as CMC and starch composed hydrogel networks result in a heterogeneous size from 2 to 20 nm. The

preliminary antibacterial activity performed to these hydrogel–silver nanocomposites.

**Filippo et al., (2009)** have introduced a colorimetric hydrogen peroxide sensor based on localized surface plasmon resonance of poly(vinylalcohol) capped silver nanoparticles. The silver nanoparticles are directly synthesized in the PVA matrix by a one-step method based on the reduction of the inorganic precursor  $\text{AgNO}_3$  through thermal treatment in aqueous medium. No other reagent is used. These nanoparticles are characterized using UV–vis spectroscopy, transmission electron microscopy and X-ray diffraction. Then they are used for the preparation, characterization and calibration of a highly sensitive, cost-effective and renewable localized surface plasmon resonance-based hydrogen peroxide sensor. The silver nanoparticles have the catalytic ability for the decomposition of hydrogen peroxide; then the decomposition of hydrogen peroxide induces the degradation of silver nanoparticles. Hence, a remarkable change in the localized surface plasmon resonance absorbance strength could be observed. As a result, the yellow colour of the silver nanoparticle polymer solution was gradually changed to transparent colour. Furthermore, when this transparent solution was subjected to thermal treatment, it became again yellow and the UV–vis spectroscopy confirmed that nanoparticles were again formed, suggesting the renewability of this sensor. The determination of reactive oxygen species such as hydrogen peroxide has possibilities for applying to medical and environmental applications.

**Purcar et al., (2009)** have prepared hybrid films containing different loadings of silver nanoparticles (0.5, 1 and 2.5 wt. %) by using a

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sol-gel method. Three types of materials were prepared in order to compare their properties: (AgNPs) on hybrid film containing methyltrimethoxysilane (Ag-MeTMS), a similar hybrid material containing both MeTMS and 3-mercaptopropyltrimethoxysilane (MPTMS) as modifier (Ag-MeTMS/MPTMS) and finally a Ag-MeTMS film to which starch was added as modifier/reducing agent (Ag-MeTMS/starch). Characterized materials by TEM showed that, in general, Ag materials had a bimodal particle size distribution, with sizes of 7–8, 12–16 nm as well as some bigger agglomerates (on some of them) of 24–25 nm present at increasing silver loadings. The catalytic properties of the supported AgNP were investigated in the oxidation of styrene. Ag-MeTMS/MPTMS materials were comparatively more active and selective than Ag-MeTMS or Ag-MeTMS/starch, regardless of the Ag content in the materials, providing high conversions and selectivities to styrene oxide.

**Zubillaga et al (2009)** have synthesized anodic alumina films containing polyaniline and either  $\text{TiO}_2$  or  $\text{ZrO}_2$  nanoparticles on an AA2024T3 Aluminum alloy by a single step anodizing procedure in an oxalic acid electrolyte. The morphology and composition of the films were examined by SEM, TEM, GDOES and XPS. The resultant coatings, of thickness about 2.2  $\mu\text{m}$ , displayed a nanoparticle -rich layer in the near surface-regions, of thickness in the range 100–250 nm. Potentiodynamic polarization behavior revealed that the polyaniline and  $\text{TiO}_2$ -containing films on the AA2024T3 Aluminum alloy show a passive current density two orders of magnitude lower than for films with  $\text{ZrO}_2$  nanoparticles and films without nanoparticles. The coatings with  $\text{TiO}_2$  nanoparticles, but without polyaniline, showed intermediate behavior, with a passive current density one order of magnitude lower than the coatings with polyaniline

and TiO<sub>2</sub> nanoparticles. The improved barrier protection offered by the TiO<sub>2</sub> nanoparticle containing coatings is attributed to the presence of the nanoparticle-rich layer formed on the outer part of the coating that blocks access to the pores of the anodic alumina film.

Migahed *et al.*, (2009) synthesized the dodecyl cysteine hydrochloride surfactant. The surface properties of this surfactant were studied using surface tension technique. The nanostructure of this surfactant with the prepared gold nanoparticles was investigated using TEM technique. The synthesized surfactant and its nanostructure with the prepared gold nanoparticles were examined as non-toxic corrosion inhibitors for carbon steel in 2M HCl solution using potentiodynamic polarization and electrochemical impedance spectroscopy techniques. The results show that the percentage inhibition efficiency ( $\eta$  %) for each inhibitor increases with increasing concentration until critical micelle concentration (CMC) is reached. The maximum inhibition efficiency approached 76.6 % in the presence of 175 ppm of dodecyl cysteine and 90.8 % in the presence of the same concentration of dodecyl cysteine hydrochloride self-assembled on gold nanoparticles. Polarization data indicate that the selected additives act as mixed type inhibitors. The slopes of the cathodic and anodic Tafel lines ( $\beta_c$  and  $\beta_a$ ) are approximately constant and independent of the inhibitor concentration. Analysis of the impedance spectra indicates that the charge transfer process mainly controls the corrosion process of carbon steel in 2 M HCl solution both in the absence and presence of the inhibitors. Adsorption of these inhibitors on carbon steel surface is found to obey the Langmuir adsorption isotherm. From the adsorption isotherms the values of adsorption equilibrium constants ( $K_{ads}$ ) were calculated. The relatively high value of ( $K_{ads}$ ) in case of dodecyl cysteine hydrochloride self-

assembled on gold nanoparticles reveals a strong interaction between the inhibitor molecules and the metal surface.

**Abd El Rehim et.al, (2008)** have reported the results of potentiodynamic polarization and electrochemical impedance spectroscopic (EIS) measurements on the corrosion inhibition of Al (Al-2.5% Cu and Al-7.0% Cu) alloys in 1.0 M H<sub>2</sub>SO<sub>4</sub> solution carried out in different concentrations of linear-sodium dodecyl benzene sulfonate as an anionic surfactant (LAS) and temperature range from 10 to 60 °C. The data revealed that the inhibition efficiency increases with increasing surfactant concentration and time of immersion, and decreases with solution temperature. Energy dispersion X-ray (EDX) observations of the electrode surface confirmed the existence of (LAS) adsorbed film on the electrode surface. The surfactant acted mainly as cathodic inhibitor. Maximum inhibition efficiency of the surfactant is observed at concentration around its critical micelle concentration (CMC). The inhibition occurs through adsorption of the surfactant on the metal surface without modifying the mechanism of the corrosion process, which tested by UV-spectroscopy. The potential of zero charge (PZC) of aluminum and Al-7.0% Cu was studied by ac-impedance, and the mechanism of adsorption is discussed. The adsorption isotherm is described by Temkin adsorption isotherm. Thermodynamic functions for activation and adsorption process were determined

**Rafiquee et al., (2008)** have evaluated the corrosion inhibition characteristics of 2-aminophenyl-5-mercapto-1-oxa-3,4-diazole (AMOD) on mild steel in HCl solution by weight loss studies and potentiodynamic polarization. AMOD is a good corrosion inhibitor in HCl solution and its inhibition efficiency is increased markedly in presence of surfactants

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(SDS, CTAB, TX-100). TX-100 is found to be most effective among the tested surfactants. Weight loss measurements showed that the inhibition efficiency increased with the increasing surfactant concentration and attained a maximum value around  $0.2 \text{ mol dm}^{-3}$ . In presence of surfactant, the adsorption of AMOD on the mild steel surface obeyed Langmuir's adsorption isotherm. The influence of inhibitor concentration, solution temperature, and acid concentration on the corrosion rate of mild steel has also been investigated. The deduced thermodynamic parameters for adsorption reveal a strong interaction between the inhibitor and mild steel surface. The negative values of  $G_{\text{ads}}$  indicate the spontaneous adsorption of inhibitors on the mild steel surface. Potentiodynamic polarization studies show that these surfactants are mixed-type inhibitors.

Yeh et.al, (2007) have prepared a series of poly(o-methoxyaniline) (PMA)/ $\text{Na}^+$  montmorillonite (MMT) clay nanocomposite ( $\text{Na}^+$ PCN) materials by in situ emulsion polymerization in the presence of inorganic nanolayers of hydrophilic  $\text{Na}^+$  MMT clay with DBSA and APS as surfactant and initiator, respectively. The as-synthesized  $\text{Na}^+$ PCN materials were characterized by Fourier-transformation infrared (FTIR) spectroscopy, wide-angle powder X-ray diffraction (XRD) and transmission electron microscopy (TEM).  $\text{Na}^+$ PCN materials in the form of coatings with low loading of  $\text{Na}^+$  MMT clay (e.g., 5 wt.%, CLMA5) on cold rolled steel (CRS) were found much superior in corrosion protection over those of neat PMA based on a series of electrochemical measurements of corrosion potential, polarization resistance, corrosion current and impedance spectroscopy in 5 wt.% aqueous NaCl electrolyte.

Chang et al., (2006) had prepared a series of polyaniline (PANI)/Na<sup>+</sup>montmorillonite (MMT) clay and PANI/organo<sup>-</sup>MMT nanocomposite materials by in situ emulsion polymerization in the presence of inorganic nanolayers of hydrophilic Na<sup>+</sup>MMT clay or organophilic organo<sup>-</sup>MMT clay with DBSA and KPS as surfactant and initiator, respectively. The as-synthesized Na<sup>+</sup>PCN and organo<sup>-</sup>PCN materials were characterized and compared by Fourier transformation infrared (FTIR) spectroscopy, wide-angle powder X-ray diffraction (XRD) and transmission electron microscopy (TEM). Na<sup>+</sup>PCN materials in the form of coatings with low loading of Na<sup>+</sup>MMT clay (e.g., 3 wt. %, CLAN3) on cold-rolled steel (CRS) were found much superior in corrosion protection over those of organo<sup>-</sup>PCN materials with same clay loading based on a series of electrochemical measurements of corrosion potential, polarization resistance, corrosion current and impedance spectroscopy in 5 wt. % aqueous NaCl electrolyte.

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***AIM OF THE WORK***

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## Aim of the work

The aim of the present work can be summarized as following:

**1**-Synthesis of some monomeric alkyloxy aromatic amine surfactants and their polymersnamely

- a- 6-(3-amino phenoxy) hexane-1- thiol (C6M)
- b- 8-(3-amino phenoxy) octane-1- thiol (C8M),
- c- 10-(3-amino phenoxy) decane-1-thiol (C10M)
- d- 12-(3-amino phenoxy) dodecane-1- thiol (C12M)
- e- poly 6-(3-amino phenoxy) hexane-1- thiol (C6P)
- f- poly 8-(3-amino phenoxy) octane-1- thiol (C8P)
- g- poly 10-(3-amino phenoxy) decane-1- thiol (C10P)
- h- poly 12-(3-amino phenoxy) dodecane-1- thiol (C12P)

**2**-Structure confirmation of the synthesized surfactants using the following spectroscopic techniques

- a- IR
- b- <sup>1</sup>HNMR
- c- TGA &DSC
- d- XRD

**3**-Evaluation of the surface properties of the synthesized surfactants using surface,interfacial tension and emulsion stability techniques.

**4**-Preparation of colloidalmetallo nanoparticles such as silver nanoparticles

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5- Investigation of the self-assembling of the synthesized surfactants onto the prepared metallo nanoparticles using the following techniques

- a- Ultraviolet absorption spectroscopy (UV)
- b- FTIR
- c- X-ray diffraction (XRD)
- d- Transmission electron microscope (TEM)

6- Investigate the effect of the prepared nanoparticles on the surface activity of the synthesized surfactants using surface, interfacial and emulsion stability techniques.

7- Study of the application of the synthesized surfactants with metallo nanoparticles as corrosion inhibitors of steel using the following techniques:

- a- Weight loss measurements.
  - b- Polarization measurements.
  - c- Energy dispersive analysis of X-rays (EDAX).
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