Subject Area : Synthetic Organic Chemistry

MECHANISTIC INSIGHTS INTO THE CORROSION INHIBITION PROPERTIES OF NOVEL ORGANIC COATINGS: SYNTHESIS AND CHARACTERIZATION

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ARTICLE INFO	ABSTRACT
	Polyaniline (PANI) was synthesised by unique oxidation polymerisation chemical process of aniline in laboratory conditions. This polymer blends with resin, colorants and other expedients obtained paint by mixing a solvent-based dispersion. Similarly, different concentration paint is prepared. The main idea was to investigate the characteristics of organic coatings containing polyaniline and polyaniline in blend with some other anti-corrosive ingredients. The adhesion, anti-corrosion and other properties of the coatings having polyaniline and selected chemically active colorants were studied as well as the combination of polyaniline with inorganic materials. The study of the total anticorrosion effectiveness of coatings with the various combinations of pigment volume concentration (PVC) that achieve high anticorrosion effectiveness.
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INTRODUCTION

"Corrosion, an insidious and pervasive phenomenon, represents a significant threat to the longevity and functionality of materials across a vast spectrum of applications. From the structural integrity of bridges and pipelines to the performance of microelectronic devices, the detrimental effects of corrosion are felt globally, incurring substantial economic costs and posing considerable safety risks. At its core, corrosion is the deterioration of a material, typically a metal, due to its reaction with its environment. This degradation is a complex interplay of physical, chemical, and increasingly recognized, biological processes. Understanding the fundamental mechanisms that drive corrosion, therefore, is paramount to developing effective prevention and mitigation strategies. This research paper will delve into the diverse causes of corrosion, examining the underlying electrochemical reactions, the influence of environmental factors, and the burgeoning role of microorganisms in accelerating material degradation.

"Within the realm of protective coatings, conducting polymers, particularly polyaniline (PANI), have garnered considerable attention due to their unique electrochemical properties, environmental stability, and relatively low cost. The relentless march of scientific progress has, for the past few decades, been profoundly influenced by the burgeoning field of polymeric and hybrid materials. [1-6] These materials, characterized by their uniquely tunable chemical and physical properties, have ignited a wave of research interest across diverse scientific disciplines. This fascination stems not only from their inherent flexibility in design and synthesis but also from their increasingly vital roles in a vast spectrum of applications. From revolutionizing photonics and electronics to enabling advanced sensors, conductive materials, and breakthroughs in biotechnology and medicine, polymeric and hybrid materials are at the forefront of innovation. [7-15] This research paper will delve into the specific properties that make these materials so compelling and explore their impactful contributions to addressing critical challenges in pollution control and other key areas, ultimately highlighting their transformative potential for the future.
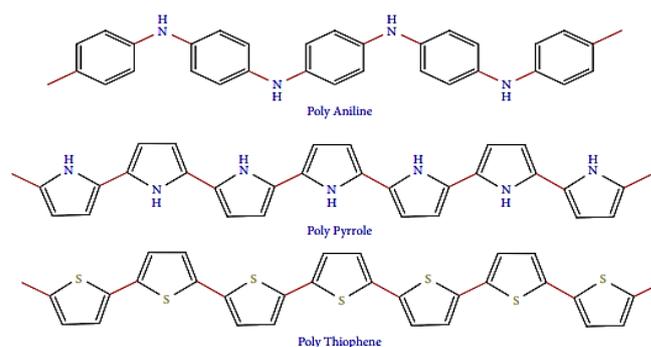

Fig.1.

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"The pursuit of advanced materials with tailored functionalities has driven significant research into conductive polymers. Among these, polyaniline (PANI) (Fig. 1) and its derivatives, alongside polypyrrole and polythiophene(Fig. 1), have emerged as frontrunners, garnering considerable attention in recent years [16-18]. This widespread adoption stems from a potent combination of desirable characteristics: robust electrical properties, commendable environmental stability, and cost-effectiveness, making them attractive alternatives in various applications. Polyaniline, historically known as "black aniline," exhibits a particularly fascinating characteristic: its existence in multiple forms dictated by its oxidation state. This versatility, coupled with its inherent simplicity [19-20] and notable stability, has cemented PANI's position as a prominent research subject. This paper delves into the structural characteristics of PANI, exploring its formation through 1,4-coupling of aniline monomers and examining how FTIR can be effectively utilized to characterize its diverse oxidation states. Understanding these fundamental aspects of PANI is crucial for optimizing its performance and expanding its potential across diverse technological fields.
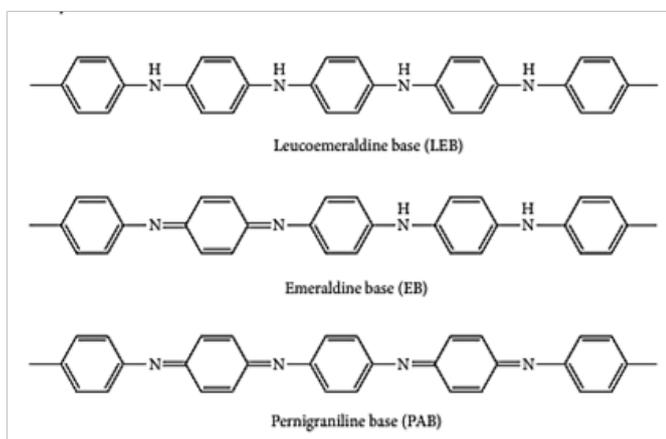
(Fig. 2) Structure of Polyaniline in changeable oxidation states: Leuco-emeraldineBase (LEB), EmeraldineBase (EB), and Pernigraniline Base (PGB)

Both leuco-emeraldine and pernigraniline forms of polyaniline are not stable and they only exist under specific provisions (Fig. 2).
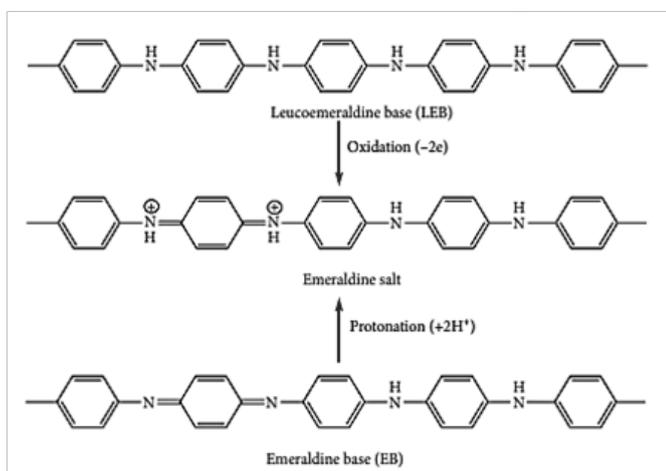

Fig.3.

Polyaniline, a conductive polymer belonging to the family of intrinsically conducting polymers (ICPs), has garnered sig-

nificant attention across diverse scientific and technological fields due to its ease of synthesis, environmental steadiness, tunable electric characteristics, and comparatively lower rate. This versatility has led to its implementation in a wide array of applications, comprising solar cells [21], lithium batteries [22], supercapacitors [23], fuel cells [24], flexible electrodes [25], corrosion-resistant coatings [26], water pollutant removal [27], screen printing [28], and sensors [29].Early advancements in PANI research focused on enhancing its processability and conductivity. Notably, Yue and Epstein et al. [30] pioneered the synthesis of protonic acid self-doped PANI, achieving a conductivity of approximately 0.1 S/cm. Subsequent investigations, such as the work by Iuliana Dumitrescu et al. [31], explored the use of surfactants like dodecylbenzene sulphonic acid (DBSA) and polymers like polyvinylpyrrolidone (PVP) to upgrade the water solubility of PANI. Further contributing to this area, Chen et al. [32] described the synthesis of water soluble self-doped PANI. Beyond conductivity and solubility, significant research has been dedicated to exploring PANI's protective capabilities, particularly in corrosion resistance.

D. E. Tallman et al. [33] investigated the electrochemical and chemical oxidation preparation of polyaniline using K2Cr2O7 as an oxidising agent and H2SO4 as a doping agent. Wessling et al. [34] have studied the overall efficiency of polyaniline primer coatings on steel by salt spray test, electrochemical impedance spectroscopy and scanning Kelvin Probe in 3.0 % sodium chloride. The coating system with polyaniline primer has been found to be very effective corrosion resistant. Further demonstrating its promise in this area, Mengoli et al. [35] described that polyaniline carrying paints show high good corrosion-resistant coatings.

Acrylic resins stand as a cornerstone in the paint industry, valued for their versatility and performance characteristics. These commercially significant polymers are synthesized via polymerisation of acrylic acid and methacrylic acid, or more commonly, the ester derivatives of acid. As a binder material, acrylic resins are integral to a diverse array of paints and coatings that underpin critical sectors such as automotive, appliance, and coil manufacturing (D.I. Lee et al. [36]). The widespread adoption of acrylic coatings stems from their exceptional resistance to hydrolysis during prolonged outer exposure, offering robust protection against weathering. Further enhancing their appeal are properties such as superior block impeding, stiffness, gloss holding, and strong alkali and oxidation protection (R. SedaTigli et al. [37]). Significant research efforts continue to explore innovative production methods, with emulsion polymerization proving to be a particularly effective technique for synthesizing both acrylic homopolymers and copolymers. Furthermore, the development of water soluble acrylic emulsions, characterized by high solid content, low viscosity, exceptional stability, and desirable film-forming capabilities (Wen Tao Huang et al. [38]), underscores the ongoing pursuit of environmentally friendly and high-performance water-based paint solutions. This research paper will delve further into the synthesis, characterization, and application of synthetic polyaniline for anticorrosion.

CORROSION PROTECTION OF POLYANILINE
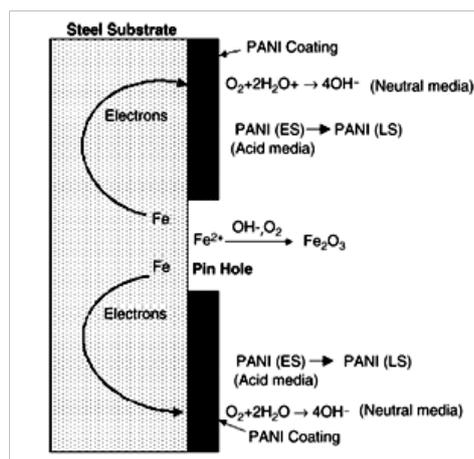
(Fig. 4) Graphic representation showing non-reactiveness of iron using polyaniline containing paint on steel

The procedure of making steel non-reactive polyaniline containing coating is graphically shown in above figure 4. Because of conducting character of the coating, the redox reaction occurs on the coating, while the ferrous ions oxidised to iron oxides by exposure of iron surface at pin hole areas and inside the film in neutral condition. However, in acidic condition the non-reactiveness of pin holes occur by the cathodic reaction of PANI (ES) → PANI (LS). Because of the change of LS in acidic medium, the coating is transferred from conducting to non-conducting state, which is indicated by the raised capacitive action of coating with intentness of time [39]. The ultimate goal is to provide a comprehensive overview of the present condition of polyaniline-based anticorrosive coatings. And also, to highlight the key areas for future research and development. This research paper will delve into the synthesis, characterization, and performance of PANI-based anticorrosive coatings.

WATER SOLVABLE RESIN

Synthetic water soluble resins are a class of polymers characterized by their ability to dissolve, disperse, or swell in water. This interaction with water results in significant modifications to the physical properties of aqueous systems. Specifically, these resins can induce gelation, increase viscosity (thickening), or facilitate emulsification and stabilization of mixtures. Their polymeric structure typically features repeating units or blocks, with hydrophilic groups strategically incorporated. These water-loving groups, crucial for their solubility, can be either substituents along the polymer chain or integrated directly into the polymer backbone. Furthermore, the nature of these hydrophilic groups can vary, existing as non-ionic, anionic, cationic, or even amphoteric moieties, allowing for a wide range of applications and functionalities.

Water soluble resins are indispensable materials employed across a spectrum of industries due to their unique properties and ability to dissolve in water. From enhancing food products and delivering pharmaceuticals to improving the performance of paints, paper, and construction materials, their applications are remarkably diverse. They play crucial roles in adhesives, coatings, water treatment processes, and beyond. Generally, water-soluble resins are broadly classified into two primary categories: synthetic resins, which are manufactured through chemical processes, and natural resins, derived from plant or animal sources. This research paper will explore the characteristics, applications, and distinctions between these two fundamental categories of water-soluble resins, highlighting their significance in various sectors. [40].

EXPERIMENTAL WORK

Synthesis of Water Soluble Polyaniline Pigment

There have been numbers of articles for the oxidative polymerization technique of PANI preparation. Here the chemical method shown has a higher performance in comparison to yield.

9.3 ml aniline was added into 100 ml volume of distilled water, kept under mechanical stirrer for 10 minutes. 20.3 ml Dodecyl benzene sulphonic acid (DBSA) then added to the above suspension and the mixture was stirred at room temperature vigorously until a homogeneous solution was obtained. 22.8 g ammonium persulfate (APS) was dissolved in 300 ml of purified water; this solution was added drop wise to the aniline DBSA solution. The reaction mixture was kept under mechanical stirring for 3 hours at 0 to 5°C temperature. The initially milky homogeneous solution turns to dark green. Finally, a dark green coloured polyaniline was obtained.

The synthesized polyaniline was specified by FTIR and UV-VIS spectroscopy, X-Ray Diffraction, SEM & EDX. The conductivity of the polymer was obtained using a hand held conductometer.

Synthesis of Water Soluble Acrylic Resin

The emulsion polymerization process was performed in a three necked round bottom flask (RBF) placed in water bath under mechanical stirring with thermometer for temperature control. 45 ml Methyl methacrylate and 45 ml ethyl acrylate were taken into 1:1 ratio was added into RBF 1 ml acrylic acid added into RBF. 1.25 g Sodium Lauryl Sulphate was taken into 20 ml distilled water in terms of total emulsifier in the recipe and monomers (MMA, EA &AA) were added into the RBF. 0.25 g potassium persulphate was dissolved into minimum quantity of distilled water, & remaining amount of distilled water was added into RBF. The RBF was purged with bubbling nitrogen for 10 min. The polymerization was carried out for about 3 h at a temperature of 70 to 80 ∘C. It was characterized by FT-IR spectroscopy, Gel Permeation Chromatography, Refractometer, % of Volume Solids.

RESULTS AND DISCUSSIONS

Characterization of Water Soluble Polyaniline UV-Visible Spectroscopy

UV-Vis Spectrum(Fig. 5) of Polyaniline was obtained by using Evolution 600 UV-Vis Spectrophotometer from Thermo Scientific (US). 1 ml Polyaniline was diluted in 5 ml distilled water and the UV-Visible spectra was measured from 200 nm to 1000 nm, which is shown in spectra of polyaniline.
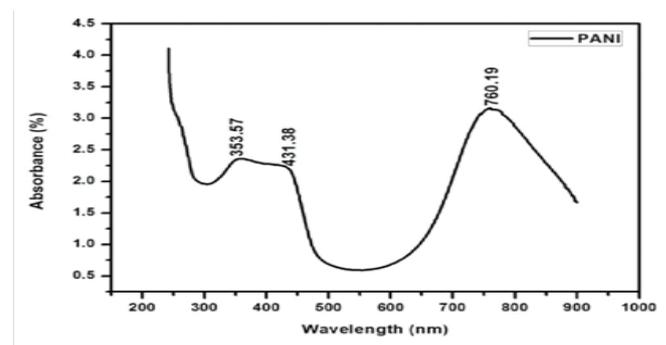

Figure 5 shows the UV-Vis spectra of synthesized water soluble polyaniline

Table 1

Wavelength (nm)	Observation
353.57	π π* transitions of benzoid ring
438.31	n π* the donor-acceptor interaction of quinonoid ring
760.19	shifting electron benzoid to quinoid ring shifting electron benzoid to quinoid ring

From this result shown in **(Table 1)** the synthesized polyaniline is in its emeraldine salt form.

FTIR Spectroscopy

The Fourier Transform Infra-Red (FTIR) the Spectra of

Synthesized water soluble Polyaniline was carried out by FTIR (Bruker) TENSOR27. The FT-IR spectra were recorded preparing with one or two drops of polyaniline in KBr powder. The FTIR spectra was measured from 3500 to 500 cm-1 which is shown in below figure 6.
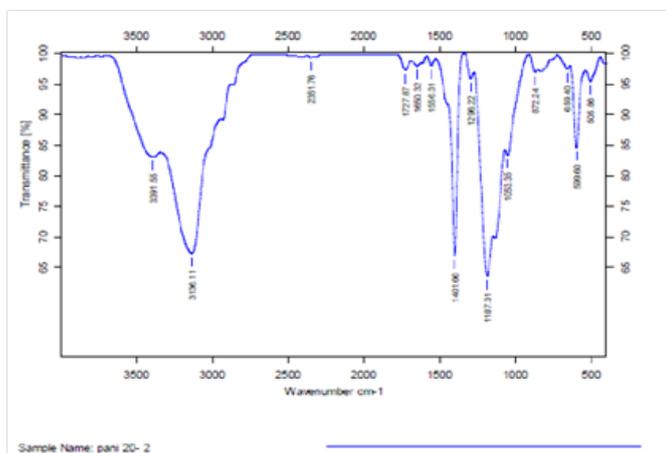

Figure 6 FT-IR Spectra of synthesized Water Soluble Polyaniline

Table 2

Frequency cm-1	Observation
1187	C-H bending
1296	C-N Stretching
1401 -1556	Benzenoid stretching
1650 – 1727	Quinoid stretching

X-Ray Diffraction (XRD)

The XRD Characterization of synthesized water soluble polyaniline was carried out by XRD (Bruker)D8 ADVANCED to identify the resultant phases present in synthesized water soluble polyaniline. polyaniline was coated on glass slide

The X-Ray diffractometer was used with Cu Kα radiation at 40 Kv and 30 mA and scan rate of 1°sec-1 within the range of 2Θ value obtained from between 20° to 80°.the wavelength of radiation used was 1.54060 A°. The peaks of 2Θ value d-spacing values are shown in below figure 7.

The values of 2Θ and d spaces are given below in the table 3. The peaks from 20° to 80° are assign to the momentum transfer and periodicity and perpendicular to the chain direction. Prominent peaks observed at 2θ ≈ 19° and 2θ ≈ 25.36° provide valuable insights into the material's organization. Specifically,

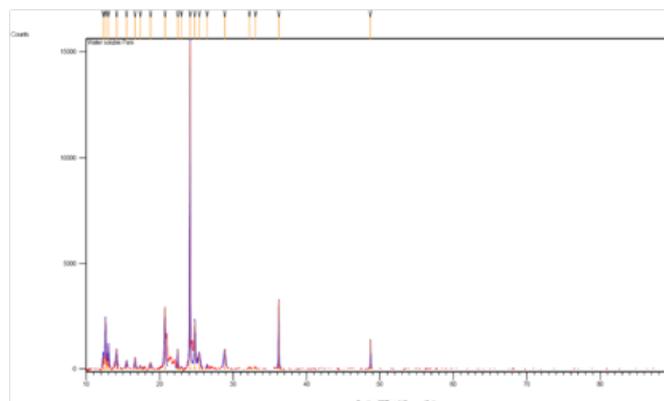the peak around 19° is indicative of van der Waals interactions between aliphatic chains.

Fig. 7 The X-RD pattern of the synthesized polyaniline

The peak at 2θ ≈ 25.36° is characteristic of van der Waals distances between the stacked phenylene rings. Furthermore, the presence of the peak at 19° suggests a well-organized, layered structure within the material. This layered structure implies that stacks of charged aniline backbones are uniformly spaced due to the intercalation or influence of the alkyl tails of DBSA. Most of all the peaks are measured towards lower side value of 2Θ showing more desirable crystalline nature of the synthesised polyamine DBSA polymer.

**Measurement of the peaks obtained by XRD patterns for Polyaniline**

Table 3

| Sr. No.	2Θ (degree)	d (A°)
1	12.29	7.19
2	18	4.73
3	20	4.28
4	25.36	3.5
5	28.82	3.09

Scanning Electron Microscope (SEM)

The SEM characterization of synthesized water soluble polyaniline was carried out by SEM (TESCAN, Model VEGA3). Polyaniline was coated on glass slide and was allowed to dry; this glass slide was sputtered with gold in order to make the sample conductive. Coated glass slides were mounted on an aluminium sample holder using a double side carbon tape to avoid particle charging, then sample loaded into the SEM. Surface morphology of the polyaniline were taken at various magnification. SEM images are shown in below figures 8 to 10.
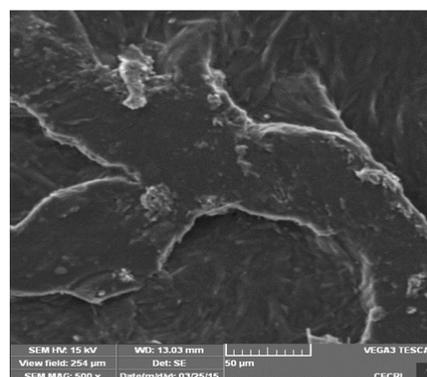
(Fig. 8)The morphology of polyaniline at 500X
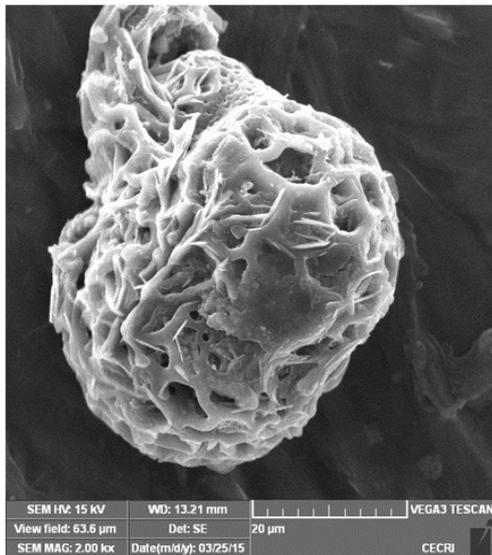
(Fig. 9)The morphology of polyaniline at 2KX
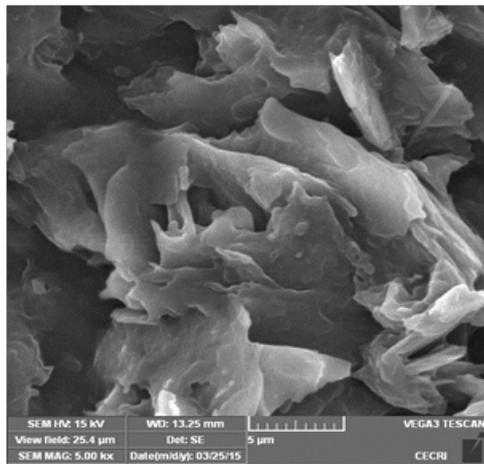
(Fig. 10) The morphology of polyaniline at 5KX

Figures 8 to 10 show the surface morphology of the water soluble polyaniline

From these SEM images it is clear that the synthesized polyaniline exhibits a crystalline morphology. Form third figure (i.e. at 5KX), it can be clearly seen that the polyaniline has lamellar like structure. This crystalline nature of the polyaniline is conformed from the XRD data also.

EDX Analysis.

EDX analysis was also carried out along with SEM analysis. This was used to analyse the % composition of the elements presents in the samples. Figure shows the EDX and table shows the compositional analysis report (Fig.11).
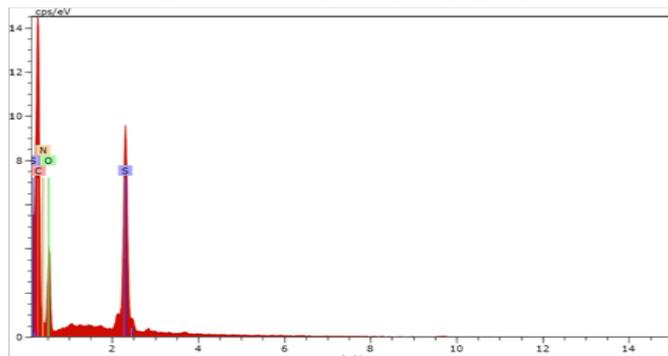

Fig. 11 EDX Analysis of polyaniline
Composition of Elements in Polyaniline
Table-4

Sr. No.	Element Name	Weight % in Polyaniline
1	C	58.59
2	O	21.27
3	N	9.95
4	S	9.65

The elemental analysis was carried out by EDX analysis. Figure-11 gives the EDX spectrum of the polyaniline sample and table-4 give the elemental composition in weight % of the polymer.

Conductivity

Conductivity of Polyaniline was found out by hand held Conductometer. Polyaniline was diluted in distilled water at different concentration and measured conductivity of polyaniline from the value of conductivity resistance value was derived. Different concentration of polyaniline conductance is given in table-5.

Conductivity of Polyaniline (Table 5)

Concentration of solution	Conductance (mS)	Resistance(Ω)
0.5%	0.41	2.43
1%	0.45	2.22
2.5%	0.86	1.16

Table 5 shows the conductivity and resistance of the water soluble polyaniline samples at different dilutions. From the data obtained it can be observed that as the concentration rise the conductivity also rises, indicating that the synthesized polyaniline is of highly conductive.

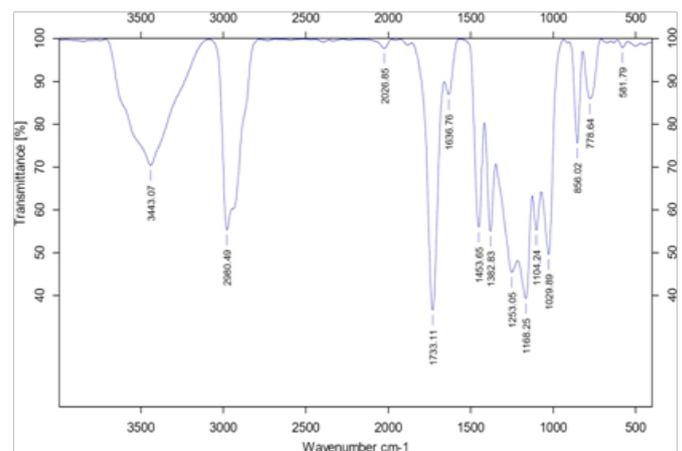**Characterization of Water Solvable Acrylic Resin FTIR Spectroscopy**

Fig. 12 FTIR Spectra Of water solvable Acrylic Resin
Table: FT-IR Spectral data of Water Soluble Acrylic Resin

Sr. No.	Frequency cm-1	Observation
1	2980-2026	Methyl & Methylene
2	1733	Carbonyl
3	1453	C-H stretching

4	1168.25	C-O-C stretching

The FTIR spectrum Figure-12 of the synthesized water solvable acrylic resin and the corresponding table gives the finger print regions of the acrylic polymer.

Determination of Molecular Weight

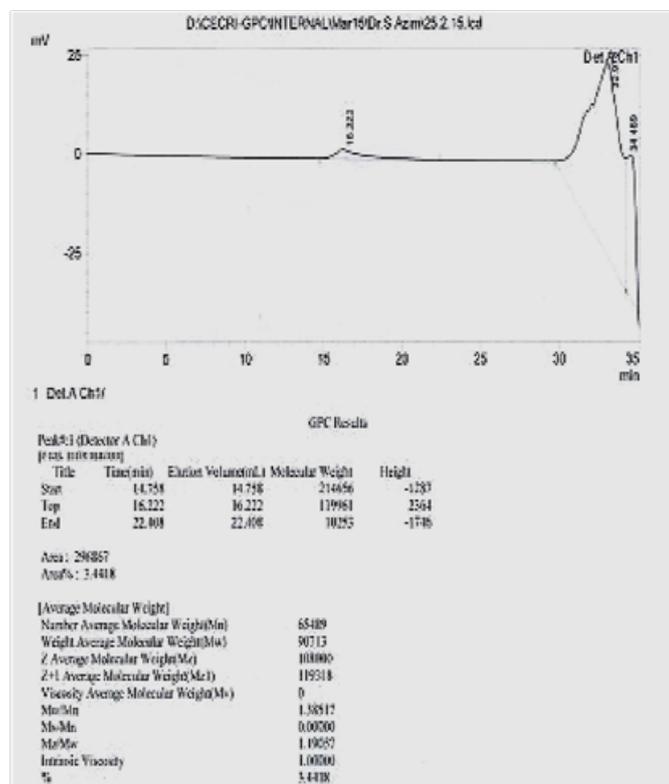The GPC result of water solvable acrylic resin

Fig. 13 GPC report of Neutralized water soluble Acrylic Resin

Form this Fig.-13, the number average molecular weight of the synthesized resin is 65489 the ratio of Mw/Mn is 1.38 indicating that the synthesized polymer has very good polydispersity. Form the monomers molecular weight the number of repeat units present in this polymer was calculates and was found to be 250.

Paint Formulation

Manufacturing of paints depend upon proper composition of paint to meet the specific requirements. These requirements may be; i) Hiding power, ii) Colour fastness, iii) Whether resistance, IV) Consistency. These requirements are met by proper choice of pigments, vehicle and extenders by the paint formulation. Most important concept of paint formulation is; Pigment Volume Concentration (PVC) revealed as percentage of the total volume of non-flammable composition of the paint as non-volatile volume in paint is the summation of pigment and non-volatile vehicle, thereof, Pigment Volume Concentration can be express mathematically as follow,

PVC = Volume of Pigment present in paint/Total Volume of non-volatile constitution of paint

Paint Formulation Methodology

Calculate volume solid & pigment volume concentration.

Weighed pigments were premixed in a mortar. The required amounts of resin were added slowly to the mixture of pre-

mixed pigments and were grinded with pestle with the addition of Solvent (distilled Water) for 30 min. The degree of dispersion of the paint was found out with Hegman Gauge by placing one drop of liquid paint and drawing the film with the help of blade provided with the Hegman Gauge. Table shows the dispersions obtained for each formulated paint.

The methodology centers around a multi-step process. First, polyaniline, in either its reduced (leuco-emeraldine) or oxidized (emeraldine) form, is meticulously combined with a resin, various performance-enhancing additives, and a carefully selected blend of solvents. Crucially, the mixture undergoes a high-energy milling process to ensure homogeneity and optimal particle dispersion. Furthermore, the primer coat formulation can be augmented with an excipient to modify its physical properties and cost-effectiveness. This PANI-based primer is then designed to be overcoated with a topcoat. This topcoat, formulated using resins, color pigments, additives, and solvent mixtures, serves a dual purpose: providing protection for the underlying PANI primer and further enhancing the overall anticorrosion performance of the coating system. This research investigates the potential of this multi-layered approach to deliver superior and long-lasting corrosion protection.

Table-6: Degree of Dispersion

Degree of Dispersion of Paint	
Polyaniline-Primer	5
Top Coat	5
Finish Coat	6

Table-7: Paint formulation details of total paint system

Polyaniline-primer	Top coat	Finish coat
VS: 50%	VS: 50%	VS: 30%
PVC: 20% Pigment	PVC: 25%	PVC: 15% Pigment
Fe2O3: 25%	Pigment	TiO2: 10%
Polyaniline: 5 %	TiO2: 10%	Cr2O3: 20%
Talc: 25 %	MIO: 8%	Silica: 30%
Silica: 25%	Talc: 8%	Talc: 30%
Mica :20 %	Silica: 12%	Aluminium stearate: 10%
	Bentonite: 24%	
	Aluminium stearate: 23%	

higher the refractive index of the pigment, the higher the paint opacity it delivers to a coating. The pigments taken here possess generally refractive index values are greater than 2.0 compare to others.

Paint application

The prepared paints were applied on wire brushed mild steel panels using conventional brushes. First a primer was put on mild steel plate. Then it is let to dry completely at room temperature. Over this primer top coat was applied and allowed to dry thoroughly at room temperature. Over this two-paint coated mild steel panel finish coat was applied and allowed to dry thoroughly. Dried coated mild steel panels were used for evaluation after 7 days of full cure.

EVALUATION OF COATING

1. Physical test method for paint Dry Film Thickness ASTM 1210

Dry film thickness test of painted mild steel was measured by magnetic Gauge [41].

Adhesion Test ASTM D4541

Adhesion test for the dry paint film was measured as per ASTM 4521.The adhesive strength of the coating was found to 1Mpa.

This due to application of paint over wire brushed surface [42].

Coating Performance Evaluation Salt Spray Test

The visual observations made periodically are given in table and figures shows the painted steel samples after salt spray exposure.

Pani primer- System A

Pani primer +Top coat- System B

Pani primer +Top coat +Finish coat- System C

Table-8: Salt Spray Test

Table-8: Salt Spray Test

Sr. No.	System	DFT µm	No. of hours passed
1.	A	22±3	48
2.	B	100±2	144
3.	C	145±2	192

Before Exposure　　　　　　After Exposure

Fig. 14 SYSTEM-A after 48 hours of exposure

Before Exposure　　　　　　After Exposure

Fig.15 SYSTEM-B after 144 hours of exposure

Before Exposure　　　　　　After Exposure

Fig. 16 SYSTEM-C after 192 hours of exposure

From the above data shown in table-8, it can be inferred that the total paint scheme has withstood only up to 192 hours (Fig.16) of continuous salt spray exposure. This is due to the water-based polymer system. Salt spray test was done by ascot s12ot salt spray chamber.

Electrochemical Impedance Spectroscopy

From the Bode plots (Fig. 17) obtained by the EIS studies used to calculate the dry paint film resistance and the double layer capacitance of the total coating system. Figure shows a typical Bode plot of the complete system in 3% sodium chloride solution.
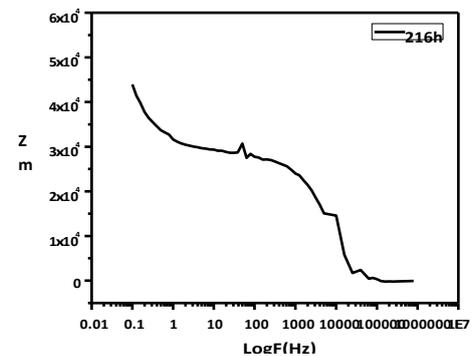

Fig. 17 Bode plot of the complete system in 3% sodium chloride

Next figure 18 shows the change in paint film resistance with respect to time.
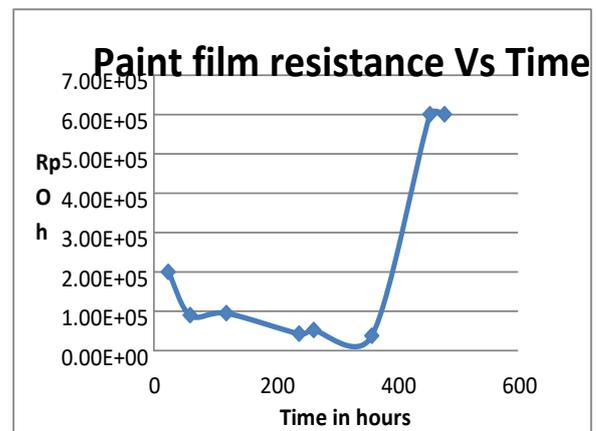

Fig. 18 Paint film resistances Vs Time

From the above plot (Fig. 18), it can be concluded that the initial paint film resistance is $2x10^5$ ohms and decreases up to 300 hours. After 300hours the paint film resistance increases to $6x10^5$ ohms and remains the same for the remaining period of exposure.

565

This increase in paint film resistance is due to passivating ability of the polyaniline-DBSA polymer present in the primer. Polyaniline due to its redox property it passivates the steel substrate and protects the steel substrate from undergoing under film corrosion.

Next figure-19 shows the change of the double layer capacitance with respect to time.
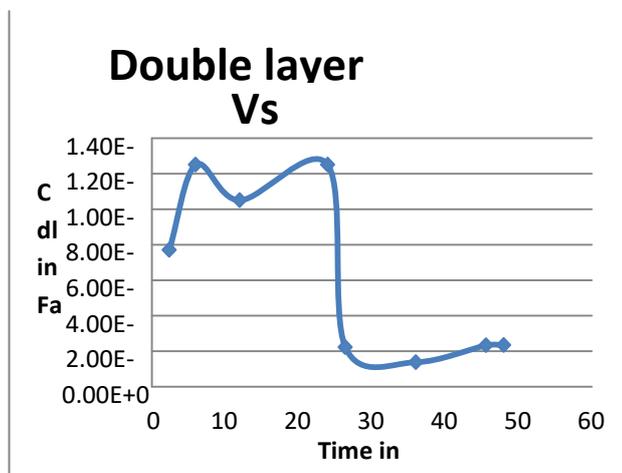

Fig. 19 Paint film double layer capacitance Vs Time

The above figure-19 shows the change in the paint film double layer capacitance with respect to time. The double layer capacitance is a measure of water up take by the polymer present in the paint film. The double layer capacitance increases from 0.77×10^5 Farads 1.89×10^5 Farads up to around 300 hours after which the value decreases to 2.22×10^6 Farads and remains in the same order till the entire exposure duration. This trend of the double layer capacitance also supports that the polyaniline present in the primer passivates the steel substrate and prevents under film corrosion.

Open Circuit Potential Measurements

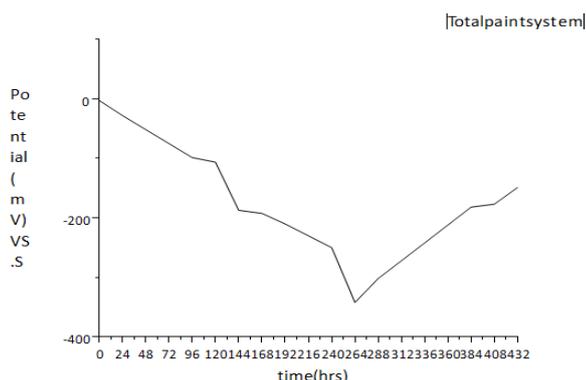The change in OCP was plotted with respect to time and it is shown in figure-20.

Fig. 20 Open circuit potential Vs Time plot

From the above graph (Fig.-20), it can be observed that the Open circuit potential of the total paint structure shifts from -2.5 mV to -342 mV up to 265 hours after that the potential shifts to more positive direction which also indicates the passivation of steel substrate by the polyaniline present in the primer.

Form both electrochemical studies it is proved that the primer coating containing PANI-DBSA prevents under film corrosion by passivating the steel substrate due to its redox property.

CONCLUSION

Successfully synthesized a highly processablepolyanilineDBSA polymer.UV-Vis, FT-IR XRD, SEM and EDX results conforms the presence of conducting polyaniline. The conductivity of the polyaniline was found to be 0.86mS indicating that a highly conducting polyaniline with good solubility in water is synthesized.The synthesized acrylic resin is affirmed by FTIR. The molecular weight of the water soluble resin was found by GPC and it is 65480 with good poly-dispersity. Primer paint was formulated with the synthesized polyaniline as one of the pigments.Top coat and finish coat also were formulated. The performance of these coatings was evaluated for its corrosion resistance properties by conventional method such as salt spray as per ASTM B117 and by electrochemical methods-Electrochemical impedance spectroscopy (EIS) and open circuit potential (OCP) measurements. From the OCP & EIS studies it was confirmed that the polyaniline present in the primer passivates and protects the steel substrate from under film corrosion.

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