Development rheological and anti-corrosion property of epoxy polymer and its composite

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ABSTRACT

Epoxy polymer, namely, decaglycidyl pentamethylene dianiline of phosphorus (DGPMDAP) was synthesized in three steps. The synthesis of epoxy polymer DGPMDAP was investigated by nuclear magnetic resonance spectroscopy, rheological analysis, scanning electron microscope (SEM), stationary and transient electrochemical methods (PDP and EIS), respectively. The rheological properties of composite (DGPMDAP/MDA/TiO2) without and with different percentages of titanium dioxide (0%, 5%, 10% and 15%) increase with both the increase in frequency and with rate of load of titanium dioxide. Besides, SEM micrographs shows a good dispersion of the titanium dioxide charge in the composite (DGPMDAP/MDA/TiO2) elaborated. The results of PDP show that epoxy polymer DGPMDAP acts as mixed type inhibitor and reaches maximum corrosion inhibition efficiency reaches 92% at 10⁻³ M. Besides, EIS results indicate that DGPMDAP act as good inhibitor for carbon steel in 1 M HCl solution and its efficiency reaches 91 % at 10⁻³ M of DGPMDAP. Furthermore, the adsorption of DGPMDAP on carbon steel surface obeyed Langmuir isotherm.

1. Introduction

Epoxy polymers are very interesting in the industrial field and are presented in an unlimited number of applications ranging from coatings to plastic in aeronautics [1, 2, 3, 4, 5, 6]. In addition, the physical and chemical properties of epoxy polymers and their composites for high potential applications are numerous: hardening, adhesive property, mechanical strength and highest resistance to corrosion [7, 8, 9, 10, 11, 12, 13, 14, 15, 16]. The carbon steel is one of the most widely employed materials for many industrial applications because of its excellent mechanical resistance. Hydrochloric acid is the most used because of its aggressive nature and its low cost. The addition of corrosion inhibitor into the aggressive solution is employed to reduce the corrosive Provoked by the direct contact of acid with the metallic surface [17]. The synthesis, elaboration, formulation and identification of epoxy polymer and its composites are granted and established in the literature [18]. There are many classes of curing agents (aromatic and aliphatic amine, anhydride acid, isocyanate) which convert epoxy polymer into composite materials with a three-dimensional (3D) structure [19, 20]. The presence of aromatic cycles give the epoxy polymer cured resistance, adhesion, thermal stability... [21, 22].

Recently, the research developed in the field of epoxy polymer aims to replace bisphenol A with a precursor without degrading the physical and chemical properties [23, 24]. In this paper, we presented the synthesis of a new epoxy polymer from a reagent containing four mobile hydrogen atoms such as methylene dianiline (MDA) with phosphorus pentachloride (PCl₅) [25].

The rheological properties of the epoxy polymer and its composite are very interesting [26]. Furthermore, the incorporation of the titanium dioxide as a load into the polymeric matrix can alter the intermolecular interaction and improve the rheological properties [27]. The improvement of the rheological properties depends on the nature of the polymer-charge interaction and dispersion quality of charge in the macromolecular matrix [28].

The epoxy polymer was investigated by NMR spectroscopy. Rheological properties of epoxy polymer DGPMDAP and its composite (DGPMDAP/MDA/TiO₂) were evaluated by the HAAK MARS rheometer. In addition, micrographs of prepared composite were determined by...
SEM. The results of PDP and EIS show that the epoxy polymer DGPMDAP is a good inhibitor for carbon steel in 1.0 M HCl solution.

2. Material and methods

2.1. Synthesis of epoxy polymer DGPMDAP

The decafunctional epoxy polymer, namely, decaglycidyl of pentamethylene diaminine of phosphorus (DGPMDAP) was synthesized in three steps:

In the first step, we mixed 24.10⁻³ mol of methylene dianiline with 4.8.10⁻³ mol of phosphorus pentachloride in the presence of methanol as solvent with magnetic stirring for 48 h at 100 °C (Scheme 1). During the second step, we condensed 0.048 mol of epichlorohydrin to pentamino pentamethylene diaminine of phosphorus with magnetic stirring at 70 °C for 4 h (Scheme 2). In the third step, we added 0.036 mol of the triethylamine base with magnetic stirring for 3 h at 40 °C (Scheme 2). The methanol and the triethylamine were removed by using the rotary evaporator. All the used products chemicals were purchased from Sigma Aldrich Chemical Co (world headquarters).

2.2. Hardening and formulation of epoxy polymer DGPMDAP

The decafunctional epoxy polymer DGPMDAP is crosslinked with MDA in stoichiometric quantities [26]. Furthermore, epoxy equivalent weight (EEW), amine hydrogen equivalent weight (AHEW) and amount of the desired load were determined by using Eqs. (1), (2), and (3), respectively. In addition, the ratio by weight of the crosslinking relative to the decafunctional matrix was evaluated per 100 parts per hundred of epoxy resin (PHR) (Eq. 4).

\[
\text{EEW} = \frac{M_w(\text{DGPMDAP})}{f}
\]  

(1)

dianiline bears two amine functions, of which the four hydrogens can be substituted while the formation of the three-dimensional network mainly involves condensation reactions between the oxirane rings of the polymers and the amine functions of the hardener [29]. The protocol consists of preheating the approximately stoichiometric amounts of the polymer and the hardener. Furthermore, DGPMDAP and MDA are raised in oven at 70 °C and 120 °C, respectively. Then, MDA is condensed with decafunctional matrix DGPMDAP to give a single liquid aspect. In addition, the prepared samples were sealed in molds in the desired geometric form for 24 h at 70 °C [5]. Besides, we performed the same protocol above in the curing reaction of 1 g of DGPMDAP with 0.31 g of MDA and titanium dioxide (TiO₂) at various percentages (0%, 5%, 10% and 15%) as a filler. Finally, the final material obtained is hard, infusible and insoluble [26] (Fig. 1).
Scheme 3. Epoxy polymer DGPMMDAP crosslinked by MDA.

Fig. 1. Preparation of material composite (DGPMMDAP/MDA/TiO₂).
AHEW = \frac{M_w(MDA)}{f} \quad (2)

y = \frac{x}{\text{resin} + \text{MDA} + x} \quad (3)

PHR = \frac{\text{AHEW}}{\text{EEW}} \times 100 \quad (4)

Where f, f', x and y denote the number of the functionality of DGPMDAP, the number of the mobile hydrogens, the amount of the DGPMDAP and the amount of the titanium dioxide, respectively.

2.3. Rheological properties

Rheological properties of decafunctional epoxy polymer DGPMDAP and its composite (DGPMDAP/MDA/TiO_2) were evaluated according to HAAK MARS rheometer.

2.4. Stationary and transient electrochemical methods

Stationary and transient electrochemical methods were realized by means of an assembly of the electrochemical cell with three electrodes such as carbon steel (work electrode), counter electrode (platinum electrode) and reference electrode (saturated calomel), respectively. The contact surface of carbon steel with corrosive solution is 1cm^2. PDP study was evaluated according to potentiostat/galvanostat SP-200 BioLogic Science Instruments. Carbon steel is immersed in the corrosive solution for 30 min with scanning speed of 0.5. Then, inhibition efficiency is realized according to Eq. (5). Moreover, EIS study was employed by same apparatus with a signal amplitude (10 mV). Besides, the frequency domain used varies from 100 kHz to 10 mHz. Then, inhibition efficiency is calculated according to Eq. (6).

\text{IE\%} = \left( \frac{i_{corr}}{i_{corr}^0} \right) \times 100 \quad (5)

\text{IE\%} = \left( \frac{R_{ct}}{R_{ct}^0} \right) \times 100 \quad (6)

With \text{icorr} and \text{icorr}^0; \text{Rct} and \text{Rct}^0 denote the corrosion current densities and the charge transfer resistances without and in the presence of various concentrations of DGPMDA, respectively.

3. Results and discussion

3.1. Nuclear magnetic resonance

Figs. 2 and 3 show the \textsuperscript{1}H NMR and \textsuperscript{13}C NMR spectra of the decafunctional epoxy polymer DGPMDAP. The alphabetical character s, d, t, q, and m denote singlet, doublet, triplet, quadruplet, and multiplet. The assignment of various chemical displacements of decafunctional epoxy polymer DGPMDAP is as follows.

\textsuperscript{1}H RMN (ppm): 1.2 (solvent); 2.5 (d, 20H, CH\textsubscript{2} of oxirane); 3.05 (m, 10H, CH of oxirane); 3.65 (d, 20H, CH\textsubscript{2} bond to oxirane); 3.8 (s, 20H, CH\textsubscript{2} bond to benzene); 4 (s, 5H, H bond to nitrogen); 6.8–7.3 (d, 40H, aromatic hydrogen).

\textsuperscript{13}C RMN (ppm): 41 (s, 5C, CH\textsubscript{2} between two benzenes); 45.8 (s, 10C, CH\textsubscript{2} of oxirane); 46.8 (s, 10C, CH of oxirane); 70 (s, 10C, CH\textsubscript{2} bond of oxirane); 112–114 (s, aromatic carbon in the ortho position of the amine II and III); 129–130 (s, aromatic carbon in the ortho position of the methylene group); 146 (s, aromatic carbon bond to amine III).
3.2. Storage modulus (\( G' \)) and loss modulus (\( G'' \))

3.2.1. \( G' \) and \( G'' \) according to temperature

Storage modulus (\( G' \)) and loss modulus (\( G'' \)) of decafunctional epoxy polymer DGPMDAP according to temperature is presented in Fig. 4. Moreover, these rheological behaviors increase with increasing of the temperature to a glass transition temperature (\( T_g \)) [30, 31]. Then, from this \( T_g \) the \( G' \) and \( G'' \) decrease. Furthermore, at temperature below the \( T_g \), the response of decafunctional epoxy polymer DGPMDAP is of the gel type. However, at temperature above the \( T_g \) the response of macromolecular matrix DGPMDAP is of the liquid type [32]. Additionally, the \( T_g \) of storage modulus and loss modulus are the same (127 °C).

3.2.2. \( G' \) and \( G'' \) as function of frequency

Figs. 5 and 6 present \( G' \) and \( G'' \) of (DGPMDAP/MDA/TiO\(_2\)) prepared composite as function of frequency at various percentage of titanium dioxide (0%, 5%, 10% and 15%) as a load, respectively [33, 34]. Furthermore, \( G' \) and \( G'' \) increase with both the increase in frequencies.
and with the rate of titanium dioxide as filler integrated in (DGPMDAP/MDA/TiO₂) composite [35]. This could explain that the charge of zinc oxide added to the various composites prepared is well formulated. Moreover, at lower frequency, the molecular relaxation process is sufficiently long, which makes the storage modulus and loss modulus measurement more sensitive. Besides, increased storage modulus and loss modulus for different composites is a common phenomenon for epoxy prepolymer crosslinked and reinforced, which can be explained by the interaction between the DGPMDAP and TiO₂ hindering the movement of the DGPMDAP macromolecular chains [36, 37, 38].

Fig. 7 shows the log(G') according to log(G''0) for (DGPMDAP/MDA/TiO₂) composite at different formulations. The Cole-Cole graph can be

Fig. 6. G' as function of frequency at different composite (DGPMDAP/MDA/TiO₂).

Fig. 7. Cole-Cole chart for composite (DGPMDAP/MDA/TiO₂) at different formulation.

Fig. 8. Morphology of different composite (DGPMDAP/MDA/TiO₂) at various percentages (0, 5, 10 and 15%) of TiO₂.
realized to analyze the rheological behaviors of the epoxy polymer [39]. It shows a linear relationship between $G_0$ and $G_00$ for homogeneous composites polymer. However, composite heterogeneous present the deviation occur from the line and the graph will be circle. The slope is less than 2 with a good correlation ($R^2 = 0.98, 1$ and $0.98$) for the different composites. This indicated the homogeneity of the composites (DGPMDAP/MDA/TiO$_2$) prepared. Furthermore, the composite (DGPMDAP/MDA/TiO$_2$) with 10% of the titanium dioxide was more homogeneous than that of (5% TiO$_2$ and 15% TiO$_2$). In addition, the composite containing 15% of titanium dioxide had a viscous behavior. However, the composite containing 5% TiO$_2$ was more elastic [28].

### 3.3. Morphology of different composite (DGPMDAP/MDA/TiO$_2$)

The SEM micrographs of (DGPMDAP/MDA/TiO$_2$) different composites prepared without and with different percentages (0 %, 5 %, 10 % and 15 %) of TiO$_2$ as load were determined by using a scanning electron microscope (Fig. 8). Furthermore, the micrographs of (DGPMDAP/MDA/TiO$_2$) different materials composites formulated at different percentages shows the good dispersion of titanium dioxide filler incorporated composite, so the quantity of the formed composite material within the charge of TiO$_2$ is highly formulated and the composite material chains movement is decreased [40]. This behavior indicates that homogeneous TiO$_2$ dispersion in the composite is solely realized at a percentage less than 15%. Some agglomerate was observable, especially at the higher particles content of TiO$_2$ (15%) [41].
3.4. PDP study

PDP curves for carbon steel in corrosive solution (1 M HCl) without and with various concentrations of DGPMMDAP at 298 K are presented in Fig. 9. From Fig. 9, the careful observation reflects that of polarization curves without and in the presence of various concentrations are similar which indicate that epoxy polymer inhibit metallic corrosion by blocking the active sites present over the metallic surface without changing the mechanism of corrosive dissolution [42]. Obviously, this observation indicates that epoxy polymer is acted as mixed inhibitor type. Then, electrochemical parameters are grouped in Table 1. From Table 1, it is clear that \( i_{\text{corr}} \) values decreased sharply when the epoxy polymer DGPMMDAP was added to the corrosive environment [43, 44]. Correspondingly, the inhibitory efficiency increases with increasing of DGPMMDAP concentration and reaches a maximum value of 92% at \( 10^{-3} \text{M} \). The highest inhibitory efficiency of epoxy polymer DGPMMDAP could be explicated due to higher molecular volume and the presence several heteroatoms that could be adsorb to metal surface. Moreover, the investigated epoxy polymer revealed relatively very good corrosion inhibitory efficiency as compared to most of the previously traditional corrosion inhibitors at lower concentration [45, 46].

3.5. EIS study

The EIS curves and Bode plots for metallic substrate in 1.0 M HCl without and with various concentrations of epoxy polymer DGPMMDAP at 298 K are shown in Figs. 10 and 11. The electrochemical parameters values such as resistance of solution (\( R_s \)), charge transfer resistance (\( R_{ct} \)) and double layer capability (\( C_{dl} \)) of epoxy polymer are grouped in Table 2. The results from Fig. 10 present alone capacitive loops in the Nyquist diagrams, which can be explicated to a single charge transfer resistance. From Figs. 10 and 11, careful examination of Nyquist and Bode diagrams reveals that the capacitive loop diameter increases with increasing of DGPMMDAP concentration [47, 48, 49]. Besides, this indicates that epoxy polymer DGPMMDAP was adsorbed on the metallic surface and the resistance for the corrosive dissolution of carbon steel in 1.0 M HCl solution is increasing. Furthermore, the results in Table 2 show that the double layer capability (\( C_{dl} \)) values decrease, however the charge transfer resistance values increase with the increase of DGPMMDAP concentration [50, 51, 52]. Then, inhibitory efficiency increases with the increase of DGPMMDAP and reaches 91% for \( 10^{-3} \text{M} \) of DGPMMDAP [53, 54, 55]. Moreover, the electrochemical equivalent circuit used to model experimental data is shown in Fig. 12. \( R_s, R_{ct} \) and CPE denote resistance of solution, charge transfer resistance and constant phase element.

3.6. Adsorption isotherm

Adsorption isotherm is employed to understand the adsorption mechanism between the atoms of inhibitor epoxy polymer as such phosphorus (P), nitrogen (N) and oxygen (O) and steel atoms at the metal surface [56]. Langmuir adsorption isotherm is calculated according to Eq. (7). Langmuir isotherm has a good straight line between \( C_{\text{int}} \) and \( C_{\text{int}}/\theta \)

![Fig. 13. Langmuir adsorption isotherm of DGPMMDAP by two methods (PDP and EIS) at 298 K.](image)

| Inhibitor | Techniques | \( R^2 \) | \( K_{\text{ads}} \) (M\(^{-1}\).10\(^6\)) | \( \Delta G_{\text{ads}} \) (KJ.mol\(^{-1}\)) |
|-----------|------------|----------|-------------------------------|-----------------|
| DGPMMDAP  | PDP        | 1        | 0.46                          | 42.72           |
| DGPMMDAP  | EIS        | 0.999    | 0.25                          | 40.79           |

![Fig. 14. Image of MEB without (a) and with (b) DGPMMDAP.](image)
with intercept of $K_{ads}$ as presented in Fig. 13. Adsorption behavior of DGPMDAP on carbon steel surface obeyed with Langmuir adsorption isotherm. From the $K_{ads}$ value, the $\Delta G_{ads}$ is determined according to Eq. (8) [57]. The highest values of $K_{ads}$ and the lowest values of $\Delta G_{ads}$ (Table 3) indicate that epoxy polymer DGPMDAP exhibits a strong interaction and a strong adsorption on the carbon steel surface (chemisorption). In addition, the linear correlation coefficient $R^2$ is close to 1, which result that the adsorption on the metal surface obeys the Langmuir adsorption isotherm [58].

$$C_{inh} = \frac{1}{K_{ads}} + C_{ads}$$

(7)

$$K_{ads} = \frac{1}{55.5} \exp \left( -\frac{\Delta G_{ads}}{RT} \right)$$

(8)

With $C_{ads}$, θ, $K_{ads}$, R and T denote concentration of DGPMDA, surface coverage degree, adsorption coefficient, constant of perfect gases and the temperature.

3.7. SEM analysis

Fig. 14 represent the analysis SEM with and without the inhibitor in acidic solution after 24 h immersion. From these images, in absence of inhibitor the surface was deeply attacked by the corrosive solution with a high degree of pitting and pits caused by solution corrosive that indicate, its protected effect of the molecules inhibitor on the surface of electrode [59, 60, 61].

4. Conclusion

Structure of epoxy polymer DGPMDAP was investigated by using NMR spectroscopy. The results obtained from the storage modulus and loss modulus concerning the epoxy polymer DGPMDAP and its composite (DGPMDAP/MDA/TiO$_2$) formulated at different percentage of titanium dioxide load are very interesting. Besides, the micrographs of various (DGPMDAP/MDA/TiO$_2$) composites elaborated show the very good distribution of the load. The results of PDP were consistent with results derived from EIS studies. Moreover, the adsorption of epoxy polymer DGPMDAP on metal surface obeyed Langmuir adsorption isotherm. Besides, EIS revealed the adsorption of epoxy polymer are confirmed with increase in $R_C$ and decrease in $C_{ads}$ values.

Declarations

Author contribution statement

Rachid Hissou: Performed the experiments; Analyzed and interpreted the data, Wrote the paper.

Ahmed Elharfi: Conceived and designed the experiments.

Mohammed Assouag, Mehdi El Bouchti, Mohamed Berradi: Contributed reagents, materials, analysis tools or data.

Omar Dagdag: Analyzed and interpreted the data.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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