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Synthesis of New Guanidine Benzothiazole Derivative and Its

Application as Eco-Friendly Corrosion Inhibitor

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Abstract

In this study, new derivative of guanidine benzothiazole, benzo[d]thiazol-2-yl-carbamidioyl-N,N-dimethylformimidamide(GBC), was synthesized under eco-friendly procedural conditions. This derivative showed an efficient activity as a corrosion inhibitor for carbon steel in acidic medium. The structure was confirmed using FTIR, 1HNMR and 13C NMR analysis techniques. The corrosion inhibition behavior of the synthesized compound was investigated using electrochemical technique such as potentiodynamic polarization(PDP). Surface morphological features were studied using scanning electron microscope (SEM) coupled with (EDX). DFT theory was studied to compute theoretical parameters that related to the electronic properties of GBC and shows a good correlation with experimental data. The inhibition efficiency (IE %) increases with increasing the concentration of the prepared compound (GBC) from 125 to 500 as follow 78% to 83%.

Keywords: Guanidine benzothiazole (GBC); potentiodynamic polarization; scanning electron microscope; Quantum Chemical Chemistry

1. Introduction

Organic compounds are important precursors in many industrial applications that benefit life as a whole. The majority of products, such as drugs, cleaning products, detergents, disinfectants, agrochemicals, food chemicals, food additives, and a wide range of materials (fibers, plastics, elastomers, and so on), are all products of various organic synthesis processes. Organic compounds, which contain heteroatom and p-electrons as active sites, could also be used as corrosion inhibitors [1-4]. The active hetero atoms in the heterocyclic compounds can be adsorbed on the metal or alloy to form a film that protect the surface against corrosion [5]. Corrosion is one of the major problems in petroleum refineries and in petrochemical plants [6]. Several industries use acidic solution as cleaning agents for pickling operations and activating or simulating oil wells, which has a significant impact on steel pipeline corrosion [7, 8]. Heterocyclic compounds bearing hetero-atoms with high electron density such as sulphur, nitrogen or oxygen are well known as corrosion inhibitors and provide superior inhibition [9-12]. Benzothaizole derivatives, in general, have demonstrated superior biological activities [13-15], and have recently been extensively studied as corrosion inhibitors for steel with high efficiency [16-19]. Because of its structure as a planar-fused heterocyclic molecule, it can form strong bonds with metal ions via donor atoms such as N and S to form metal complexes. The benzothiazole molecule has three surface-adsorption-friendly anchoring sites: the nitrogen atom with its lone pair of electrons, the sulphur atom, and the aromatic rings [20]. One of benzothiazole derivatives which showed high efficiency as corrosion inhibitors is 2-

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hydrazinobenzothiazole (HBT) [21]. The inhibitor efficiency of (HBT) has been increased with increasing temperature, concentration and immersion time.

Quantum chemical chemistry has been considered a critical approach that combines reactivity with the chemical structure. Hence, it can provide a good prediction for the inhibition efficiency configuration at the density functional theory level has been used to undertake theoretical calculations [22].

The goal of this research is to synthesize and develop a cheap, environmentally friendly, and efficient corrosion inhibitor of new derivatives of guanidine benzothiazole, benzo [d] thiazol-2-yl-carbamidioyl-N, N-dimethyl formimidamide (GBC) with a substituted guanidine moiety that is structurally similar to (HBT). The work will extend to investigate its inhibitory effects on carbon steel by Potentiodynamic Polarization technique. Surface morphology was studied by SEM and EDX analysis.

2. Experimental
2.1. Synthesis and preparation of Novel inhibitor (GBC)

Melting points were measured on a SMP3 melting point apparatus using open capillary tubes and are uncorrected. IR spectra were recorded on FTIR plus 460 or pye unicam SP-1000 spectrophotometer using KBr pellets. ^{1}H NMR(400 MHz) and ^{13}C NMR (100 MHz) were done in NMR unit at Faculty of Pharmacy, Beni-Swef University, and spectra were recorded on a Bruker advance (III) model Ultra Shield NMR spectrometer in DMSO-d6: as a solvent using tetra methyl silane (TMS), as an internal standard and chemical shifts are reported as δppm units. Progress of the reactions was monitored by TLC using aluminium sheet coated with silica gel MERCK 60F 254 and was visualized by UV lamp. The reagents and solvents were purchased in commercially available grade purity. Guanidine benzothiazole (3) was prepared previously reported [23].

The synthesis was carried out at room temperature, where a mixture of guanidine benzothiazole(3) (0.02mole) and N,N-dimethylformamidedimethylacetale (DMF-DMA) (0.06 mol) was stirred for 1 hour. The precipitate was collected by filtration and allowed to dry at room temperature. A hot solution of petroleum ether and ethyl acetate 90:10 ml was used for further purification.

2.2. Electrochemical measurements

In this study Low carbon steel grade 1018 (UNS G10180) with the following composition; (wt %): C (0.15-0.20), Mn (0.60-0.90), S (0.050), P (0.040) and the remainder is iron has been used. All specimens were mechanically polished with a sequence of emery papers of various grades (150, 320 and 600) and thoroughly washed with distilled water and acetone.

A typical electrochemical cell with three electrode Pyrex glass cell at room temperature using Autolab Potentiostat/ Galvanostat PGSTAT 302N connected to a computer. A reference electrode (silver/silver chloride, E0=0.203 V; vs. SHE), the counter electrode used was made up of a platinum foil and a piece of carbon steel , 1cm2 area exposed as working electrode in 1 M HCL. Before each experiment the open circuit potential (OCP) was applied. The potentiodynamic polarization curves were recorded to study the effect of new derivatives of guanidine benzothiazole, benzo [d] thiazol-2-yl-carbamidioyl-N, N-dimethyl formimidamide at room temperature, all polarization measurements were performed in the potential of -1000 to -200 mv, with scan rates of 1 mVs-1 for different GBC concentrations. Evaluation of both current density and corrosion potential was performed from the intersection of the linear anodic and cathodic branches of the Tafel plots. The inhibition efficiency has been calculated using Tafel extrapolation method.

2.3 Theoretical calculation

Quantum chemical calculations for the optimized geometry compound were carried out using Density Functional Theory (DFT). Dipole moment (D) and different quantum descriptors were calculated [24, 2]. Such as the energy gap ΔEg (EL − Eh), Mulliken population of the charge, Ionization potential (I=-Eh), electron affinity (A= -EL), Absolute electron affinity (the half of summing of ionization potential and electron affinity), the global hardness (η= have the difference between the ionization potential and electron affinity.

2.4 Surface morphology

The surface morphology of the carbon steel specimens in1M HCl without and with inhibitors 500 ppm GBC are performed on scanning electron microscope (JSM-6510) equipped with an energy dispersive X-ray spectrometer (Quantax75).

.3. Results and Discussion
3.1. Synthesis and characterization

Guanidine benzothiazole (3) was used as a starting compound to synthesize benzothiazole

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incorporating a substituted guanidine moiety (4). The reaction of o-aminothiophenol(1) with 1-cyanoguanidine (2) in acidic medium under reflux for 2 hours yielded guanidine benzothiazole (3) in good yield. The latter was subjected to a one-hour reaction with DMF-DMA at room temperature, resulting compound (4) as shown in Scheme 1. And IR, ^{1}H NMR, and ^{13}C NMR confirmed the structure of the compound (GBC).
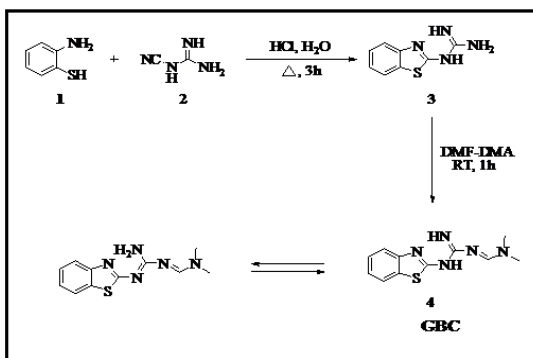
Scheme1. Chemical synthesis of guanidine benzothiazole, benzo [d]thiazol-2-yl carbamidioyl-N, N dimethylformimidamide

Figure1 shows the FTIR spectrum of the compound GBC, Strong absorption band at 3251 cm[-1] which related to NH group, the band at 3073 cm[-1] is due to stretching vibration of C-H (Aromatic), and the stretching vibration at 2752 cm[-1] is related to C-H (Aliphatic), the stretching vibration at 1694 cm-1 is related to C=N group and stretching vibration at 1610 cm[-1] is related to C=C group.
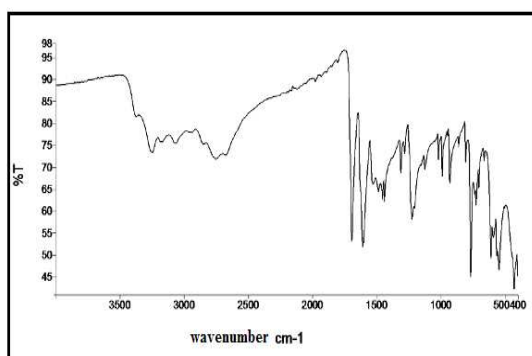

Figure1. FTIR Spectrum of the prepared GBC

Figure2 shows ^{1}H-NMR spectrum of the compound GBC, Six protons from two methyl groups were represented as a singlet at 3.19 ppm., two multiplet peaks ranging from 7.10 to 7.29 ppm and two doublets at 7.56 and 7.73 ppm in the spectrum confirmed the presence of the benzothiazole ring. Furthermore, the presence of the proton of the CH=N group was confirmed by a singlet signal at 8.74 ppm
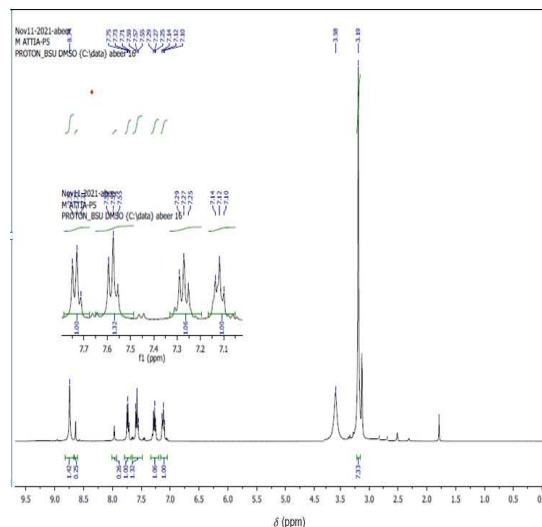

Figure2. HNMR Spectrum of the prepared GBC

Figure 3 shows ^{13}C NMR spectrum of the compound GBC, the two carbons of two methyl groups were represented by a single peak at 35.1 ppm.
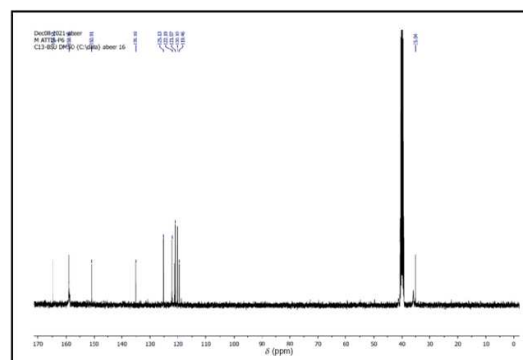

Figure3. ^{13}C NMR Spectrum of the prepared GBC

3.2. Potentiodynamic polarization measurements (PDP)

The results of PDP curves are shown in Figure 4. Electrochemical corrosion parameters obtained from polarization measurements for carbon steel with and without different concentration of GBC in 1.0 M HCl at 25o C were quoted in Table 1. The electrochemical parameters which have been recorded through

polarization measurements (Icorr, Ecorr, βa, βc, and Corr. rate).Where Icorr is the corrosion current density, Ecorr is the corrosion potential, βa, βc are anodic and cathodic Tafel slopes respectively according to Stern-Geary relation [25]:

$$I_{corr} = \frac{B}{Rp} \quad , \quad B = \frac{\beta a * \beta c}{2.303\,(\beta a + \beta c)} \quad (1)$$

Where Icorr (A.Cm-2) and B (V). βa and βc are the anodic and cathodic Tafel slopes.

$$I_{corr} = \frac{1}{Rp} \times \frac{\beta a * \beta c}{2.303\,(\beta a + \beta c)} \quad (2)$$

The corrosion rate can then be calculated using Faraday equation [26]:

$$CR = 3.27 * 10^{-3} I_{corr} Ew/d \quad (3)$$

Where Ew is the equivalent weight of the metal and d its density.

The Icorr values are used to calculate the inhibition efficiency (IE %) using the following Equation:

$$IE\% = [I - I_{corr}/I] * 100 \quad (4)$$

Also the notable decrease in the corrosion potential (Ecorr) while increasing the concentration of the prepared compound (GBC) is attributed to formation of a compact film on the surface of the working electrode which caused by the adsorption of the prepared compound (GBC) on the active sites of the carbon steel which in turn retards the reduction of H+ ion evolution [27]. Also the anodic dissolution of the metal decreases due to the adsorbed molecules on the anodic active sites of the carbon steel which is also indicates that the prepared compound (GBC) is a good corrosion inhibitor.

Both cathodic and anodic branches of the polarization curve are sifted towards more negative values and to lower current densities with the increase in the   Both cathodic and anodic branches of the polarization curve are sifted towards concentration of the prepared compound (GBC) as compared with the blank which attributed to the formation of more stable and passive film on the surface of the sample.
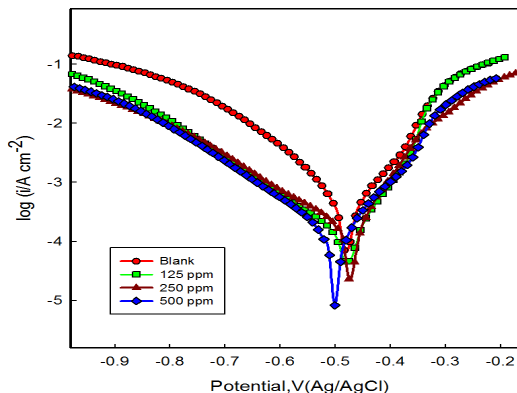

Figure4. Potentiodynamic Polarization curves of on carbon steel in 1M HCl and different concentrations of (GBC) at room temperature

Table 1
Electrochemical corrosion parameters obtained from polarization measurements for carbon steel with and without different concentration of GBC in 1.0M HCl at 25ºC

Sample	Conc. ppm	βa mVdec	βc (mV/dec)	Ecorr mV	icorr uAcm⁻²	CR Mmy⁻¹	Pol. res. Ω	IE %
Blank	0	122.4	129.2	-479	466.1	5.42	58.58	---
GBC	125	157.3	70.1	-471	79.7	1.17	209	78
	250	55.1	99.6	-468	88.2	1.03	174.4	81
	500	97.8	85.9	-503	76.2	0.93	249.0	83

Also Corrosion rate of carbon steel immersed in 1M HCl decreased in presence new prepared inhibitor (GBC) as its concentrations increased. These behavoir matched with increased efficiency % that increased with increasing concentrations of inhibitor till 500ppm. As shown in Figure 5.
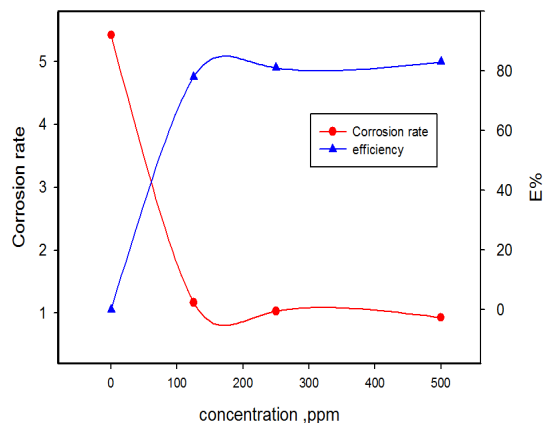

Figure 5. Variation of corrosion rate and inhibition efficiency with various concentrations of inhibitor (GBC)

3.3. SEM and EDX Analysis

To establish the inhibition effect of the investigated inhibitor, scanning electron microscope was taken. The SEM micrographs of the carbon steel after immersion in1M HCl with and without 500 ppm of GBC were recorded at the same magnifications for comparison.

The surface morphology of the uninhibited carbon steel is shown in Figure 6A. Avery rough metal surface with cracks and pits covered with thick porous oxide layer due to severe corrosion. It can be concluded that carbon steel surface was greatly damaged in absence of GBC. The morphology of the inhibited carbon steel is shown in Figure 6B. In the presence of 500 ppm GBC, the surface is somewhat smoother than in its absence what points to the inhibiting effect. The rough surface was reduced with protected dense layer of metal's surface indicating that the GBC can be regarded as a good inhibitor. This conclusion is consistent with the results obtained by the electrochemical experiments.

From Figure it can be concluded that the carbon steel surface morphology is remarkably improved in presence of GBC. This finding further suggests the formation of protective film of GBC on carbon steel surface.
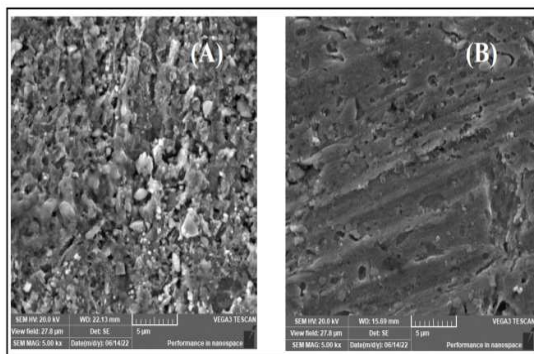

Figure 6. SEM images of the carbon steel surface after exposed to (A) 1 M HCl solution in (B) in the presence of 500 ppm GBC

The EDX spectra of carbon steel was recorded in 1M HCl in absence and presence of 500 ppm GBC is shown in Fig 7A represent the EDX spectra on uninhibited carbon steel and Figure7B show the EDX spectra of inhibited steel shows peaks for oxygen, nitrogen and sulphur, which suggest that the GBC form a protective film on carbon steel surface [28].
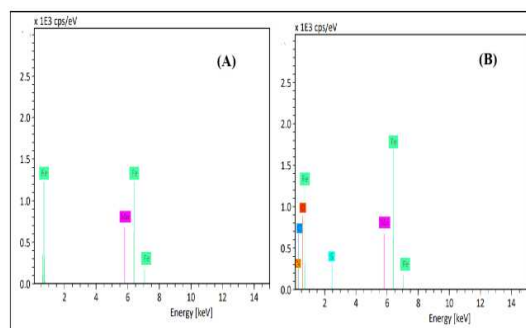

Figure 7. EDX spectra of carbon steel (A) in 1MHCl, (B) in presence 500 ppm GBC

3.4. Quantum chemical calculation

The optimized geometry configuration of GBC was obtained with a minimum total energy of 13.63eV Figure 8 (A and B) shows the electron charge density surface of the studied molecules, the blue color to identify the most negative potential, and the electron-rich region. In contrast, the red color determines the e's poorest areas. The electron density is localized mainly in the vicinity of nitrogen atoms and benzene rings. The distribution of electrons around the investigated facilitates both the donation and acceptance of the electron to and from the metal surface, and this is also confirmed by the Mulliken population of the charge as Table 2.
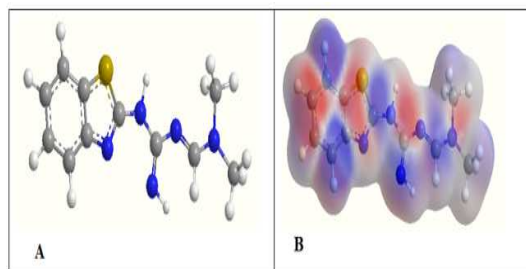

Figure 8. (A) The optimized structure and (B) Electrostatic potential surface of GBC

According to quantum explanation, the energy of the highest occupied molecular orbital (Eh) is frequently linked to a molecule's capacity to donate electrons. The greater Eh value, the ease offered of electrons to the unoccupied d orbital of the metal surface, and the better the inhibition efficiency of the inhibitors. Similarly, the lower the energy value of the lowest unoccupied molecular orbital (EL), the simpler it is for electrons to accept from the metallic surface.

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Table 2.
Mullikan population charge of GBC

Atom	Charge	Atom	Charge	Atom	Charge	Atom	Charge	Atom	Charge
C(1)	-0.066	N(7)	-0.557	N(13)	-0.859	H(19)	0.020	H(25)	0.036
C(2)	0.101	S(8)	0.702	C(14)	0.177	H(20)	0.012	H(26)	0.038
C(3)	-0.159	C(9)	0.193	N(15)	0.414	H(21)	0.013	H(27)	0.038
C(4)	-0.141	N(10)	0.283	C(16)	-0.057	H(22)	0.074	H(28)	0.036
C(5)	-0.131	C(11)	0.360	C(17)	-0.050	H(23)	0.116	H(29)	0.037
C(6)	-0.142	N(12)	-0.530	H(18)	0.019	H(24)	-0.019	H(30)	0.036

In addition, a negative value of Eh and the lower value of EL for the investigated inhibitors indicate significant donation and acceptance ability of the electrons to and from the metal surface, respectively. Also, the enhancement of the inhibition efficiency by the formation of a coordinated covalent bond that formed between GBC and the metal surface was confirmed by a lower value of energy gap (ΔEg = 8.23eV), Ionization potential (I= 7.31), and hardness (η = -7.379eV) [29-31], as shown in Figure 9
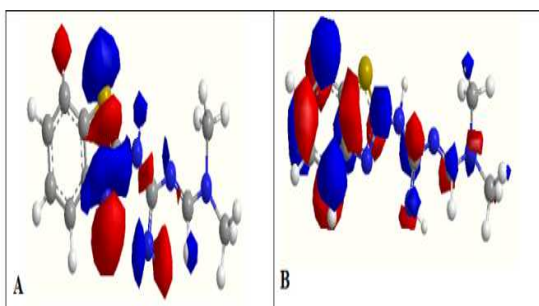

Figure 9. HOMO and LUMO representation of GBC

Another important parameter that related to the electronic distribution in the molecule is dipole moment, lower dipole moment value (D= -

6.978Debye) indicate that GBC favors to accumulate on the metal surface results in higher protection efficiency [31].

3.5. Mechanism of corrosion inhibition

The nitrogen and sulpher containing groups as well as the double bonds in the prepared compound (GBC) were the basic factors led to decreased corrosion rate where the adsorption of the prepared compound on the surface of the carbon steel decrease its dissolution; this adsorption can be explained as follows: in the acidic/ corrosive medium Fe ions dissolves from the surface of the carbon steel leaving negative charges on surface which then interacts with NH+ groups of the inhibitor (GBC), the positive charge on nitrogen of the inhibitor could interact with metal surface. The -electrons of the double bonds in heterocyclic structure of the inhibitor could also be adsorbed on the vacant d-orbitals of the carbon steel surface through donor-acceptor interactions. Also two week electron donating groups were indicated in HNMR spectra at δ3.19 (s, 6H, 2CH3), which have the ability to be adsorbed on the surface of the metal [32].

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Based on last proposed adsorption mechanism represented as Figure 10, the increased concentration of the inhibitor resulted in formation of a compact film on the surface of the metal which in turn resulted in decreased corrosion rate and increased efficiency
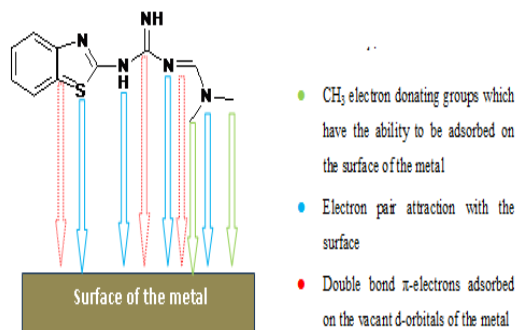

Figure 10. Proposed mechanism of adsorption of (GBC) on the surface of the metal

4. Conclusions

. The prepared compound (GBC) is a good corrosion inhibitor based on the following results:

1-   The inhibition efficiency (IE %) increases with increasing the concentration of the prepared compound (GBC).

2-   The potentiodynamic polarization results showed a decrease in the rate of anodic reaction (metal dissolution) over cathodic reaction (hydrogen evolution reaction).

3-   The potentiodynamic polarization indicated a decrease in the corrosion potential (Ecorr) of the working electrode with increasing the concentration of the prepared compound (GBC). The result can be attributed to formation of compact film layer of the prepared compound on the surface of the metal.

4-   The results of SEM showed a compact protective film on the surface of the metal.

5. Funding

6. Declarations

Conflict of interest No conflict exist and the authors declare that they have no conflict of interest.

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