Microwave Assisted Synthesis of Benzotriazole Derivatives for Anti-Corrosive Study on Mild Steel in Acidic Medium

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Abstract

Four novel benzotriazole derivatives 1-(Chloromethyl)-1H-Benzotriazole; 1, N-(benzo[e][1, 2, 4]triazin-4(3-H)-yl)methylbenzenamine; 2, 1-((1-H-benzo[d][1,2,3]triazol-1-yl)methyl) hydrazine; 3 and 1-((1-H-benzo[d][1,2,3]triazol-1-yl)methyl) phenyl hydrazine; 4 were synthesized through microwave irradiation (MWI) method. These derivatives were characterized and tested for anticorrosive action on iron coupons in 0.5M acidic medium at different concentration. The anti-corrosive study was done using weight loss method and corrosion rate (CR) analysis. All the synthesized derivatives gave remarkable corrosion protection effect. Major outcome of the study shows corrosion inhibitor efficiency of derivatives follows order 3, 4, 2 and 1 at 100mg/L, 3, 2, 4 and 1 at 200mg/L, 3, 4, 2 and 1 at 300mg/L. Compound 1-((1-H-benzo[d][1,2,3]triazol-1-yl)methyl)hydrazine; 3 emerges as leader in all concentration in this study while 1-(Chloromethyl)-1H-Benzotriazole; 1, shows moderate impact. This study imparts an understanding for the new class of anticorrosive benzotriazole derivatives which can be of industrial use.

Keywords: benzatriazole, corrosion inhibitor, microwave, corrosion rate, mild steel, weight loss, anticorrosion.

One report released by FHWA, USA, 2002 reveals that the cost to the economy in USA due to corrosion was estimated USD276 billion which was equivalent to approximately 3.1% of the U.S. Gross Domestic Product (GDP). Similar studies in other countries have shown a similar percentage of GDP [1]. Another report published by NACE international, USA, 2016 reveals the estimated cost to the world economy due to corrosion was 3.4% of the GDP [2], which is summarized in table-1.
Table 1: Global cost of corrosion by region (Billion USD 2013)

| Economic region         | Total GDP USD billion | COC % GDP |
|-------------------------|-----------------------|-----------|
| United states           | 16,720                | 2.7%      |
| India                   | 1,670                 | 4.2%      |
| European Region         | 18,331                | 3.8%      |
| Arab world              | 2,789                 | 5.0%      |
| China                   | 9,330                 | 4.2%      |
| Russia                  | 2,113                 | 4.0%      |
| Japan                   | 5,002                 | 1.0%      |
| Four Asian Tigers plus  | 2,302                 | 2.5%      |
| Rest of the World       | 16,057                | 3.4%      |
| **Global**              | **74,314**            | **3.4%**  |

These data elaborates that corrosion is a global phenomenon, and has the potential to give impact to the global economy. Polymers, metals, ceramics all can undergo corrosion. There are several types of corrosion e.g. galvanic corrosion, microbial corrosion, general corrosion, pitting corrosion etc.

This work focuses on corrosion inhibition activity of metals, as mostly metals are used everywhere. Metals, when they are kept under an open environment where they are not stable they undergo corrosion. Sculptures made up of bronze generally corrode and its appearance and property of being persistent reduces [3-5]. Corrosion can be prevented by using like modification of environment, plating and coating [6-9]. The materials used for protection must be removed easily and their colour should not change [10]. There are various types of corrosion inhibitors: Inorganic and Organic inhibitors. Chromates, silicates, nitrites, phosphates, arsenates and carbonates are few examples. Organic inhibitors are compounds of sulphur like thiourea, thioester etc., heterocyclic compounds of nitrogen and hydrazine [11].

Thin and persistent films are made by organic inhibitors which help in decreasing corrosion rate as they lead to slow down the reactions taking place i.e. cathodic and anodic reactions. There are so many organic compounds that have polar groups including nitrogen and sulphur. They have been seen showing the anti-corrosive behaviour against number of metals [12-15].

A well-known compound which is an organic inhibitor for copper and its alloys is 1, 2, 3-benzotriazole (BTA) and its derivatives [16-18]. It is an anticorrosive agent used in airports. Benzotriazole leads to formation of a film less than 50 Armstrong. Many chemists have studied the...
interaction of benzotriazole with bronze\textsuperscript{[19-22]} and mild steel\textsuperscript{[23]}. BTA forms a complex with Cu (I) and Cu (II) which forms a protective thin and persistent film\textsuperscript{[24-25]}. The rate of growth and the density of film depend on concentration of benzotriazole and how much time BTA immerse on the surface of metal. It binds to metals and forms the strong bonds through the triazole ring. So benzotriazole is an organic inhibitor used to reduce pitting of the surfaces, surface strains and tarnished surfaces formed by corrosion\textsuperscript{[26-27]}. Few other benzotriazole derivatives as surveyed in literature for potent corrosion inhibitors are illustrated in Table 2.

### TABLE 2: ANTICORROSIVE ACTIVITY FOR BENZOTRIAZOLE DERIVATIVES

| Benzotriazole Derivative | IUPAC Name | Anticorrosive against metal | Reference |
|--------------------------|------------|----------------------------|-----------|
| (Complex of Benzotriazole) | Copper | [28-29] |
| Benzotriazole | Mild steel | [30] |
| 5-Methyl-1H-Benzotriazole | Mild steel | [30] |
| 5,6-Dimethyl-1H-BenzotriazoleMonohydrate | Mild steel | [30] |
| 1-Methanesulfonyl-1H-Benzotriazole | Mild steel | [30] |
1-(α-Chloroacetyl)-1H-Benzotriazole

Mild steel [30]

2-H-benzotriazole with a diethylene glycol

Copper [31]

5-dodecyl-1,2,3-benzotriazole

Bronze [32]

[5-(1-undecyl)dodecyl]-1,2,3-benzotriazole

Bronze [32]

5-hexyl-1,2,3-benzotriazole

Bronze [32]

5-Chlorobenzotriazole

Copper (HCl) [33]

5-Methylbenzotriazole

Copper (HCl) [33]

5-n-butyl benzotriazole

Copper (HCl) [33]
In the present work, four novel derivatives of benzotriazole had been synthesized, characterized and studied for anticorrosive activity against iron coupons in acidic medium.

**MATERIALS AND METHODS:**

a. The synthesis of the compounds was carried out using RAGA’s microwave systems microwave oven of 700W. The purity of the compounds was checked using TLC plates (glass) coated with silica gel. The developed chromatographic plates were visualized under iodine chamber. IR spectra were recorded
using KBr on Shimadzu FTIR model 8400 spectrophotometer. 1H NMR spectra were recorded through Bruker Advance II 400 NMR spectrometer.

b. **Weight loss method for determination of corrosion rate (CR)** [39]

The corrosion rate was determined by weight loss analysis method. The weight loss is converted to corrosion rate using the formula given below-

\[
\text{Corrosion Rate (CR)} = \frac{\text{Weight loss (g) } \times \text{K}}{\text{Alloy Density (g/cm}^3\text{) } \times \text{Exposed Area (A) } \times \text{Exposure Time (hr)}}
\]

Where K is constant; 8.76 x 10^4, metal density is 7.85g/cm^3

**EXPERIMENTAL WORK:**

The synthesis of compounds 1-4 was carried out as per scheme 1.

**Scheme I**

**Synthesis of 1-(Chloromethyl)-1H-Benzotriazole; 1**

0.048mole of benzotriazole was mixed with 30 ml of DMF and 0.048 mole of K_2CO_3 was mixed well with 30 ml of DCM. Both the mixtures were added to RBF and were treated under microwave irradiation for 3 min. at 300 watt. Product thus obtained was added to 75 ml ice cold
water followed by filtration and recrystallization by hot water. TLC was checked using hexane: ethyl acetate (7:3). **FT-IR (KBr\textit{\upsilon}\text{max cm}^{-1})**: 3002 (Ar C-H) str, 1558 and 1610 (C=C Arenes) str, 1264 (C-N Aromatic amine) str, 850 (C-Cl halo compound) str. **NMR (400 MHz, DMSO, $\delta$/ ppm)**: 8.1 (m, 1H, Ar), 7.9 (m, 1H, Ar), 7.6 (d, 1H, Ar), 7.8 (s, 2H, CH$_2$).

**Synthesis of N-(benzo[e][1, 2, 4]triazin-4(3-H)-ylmethylbenzenamine; 2**

0.006 mole of compd. 1 was mixed with 10 ml DMF followed by addition of 0.018 mole of p-aminotoluidine and 0.006 mole of K$_2$CO$_3$ in the RBF. The complete mixture was treated under microwave irradiation for 4 minutes; 240 watt. To the reaction mixture 10% HCl was added to remove excess of p-toluidine. The desired product was extracted by chloroform (3×10) ml and the product was recrystallized with hot water. At last TLC was checked using hexane: ethyl acetate as solvent system in the ratio (7:3). **FT-IR (KBr\textit{\upsilon}\text{max cm}^{-1})**: 752.26 (C-H Ar-rings) bending, 1161.9 (C=C Arenes) str, 1274.03 (C-N Ar amine) str, 669.32 (N-H) wagging, 2956.01 (Ar C-H) str. **NMR (400 MHz, DMSO, $\delta$/ ppm)**: 7.6-8.0 (m, 8H, Ar), 7.45 (m, 2H, CH$_2$), 8.10 (m, 1H, NH), 2.5 (s, 3H, CH$_3$).

**Synthesis of 1-((1-H-benzo[d][1, 2, 3]triazol-1-yl)methylhydrazine; 3**

0.006 mole of 1-(Chloromethyl)-1H-Benzotriazole was mixed with 10 ml of DMF in RBF to which 0.006 mole of hydrazine hydrate was added. In the mixture 0.018 mole of hydrazine and 0.006 mole of K$_2$CO$_3$ were added. The entire mixture in RBF was treated under microwave irradiation for 4 minutes; 240 watt. To this reaction mixture, 10% HCl was added to remove excess of hydrazine. The desired product was extracted by chloroform (3×10) ml. At last TLC was checked using DCM as solvent system. **FT-IR (KBr\textit{\upsilon}\text{max cm}^{-1})**: 752.26 (C-H Ar-rings) bending, 1558.54 (C=C Arenes) str, 1262.45 (C-N Ar amine) str, 619.26 (N-H) wagging, 2955.04 (Ar C-H) str. **NMR (400 MHz, DMSO, $\delta$/ ppm)**: 7.65-7.95 (m, 4H, Ar), 7.45 (m, 2H, CH$_2$), 8.10 (m, 1H, NH), 3.7 (s, 2H, NH$_2$).

**Synthesis of 1-((1-H-benzo[d][1, 2, 3]triazol-1-yl)methylphenyl hydrazine; 4**

0.006 mole of 1-(Chloromethyl)-1H-Benzotriazole was mixed with 0.006 mole of phenyl hydrazine and 8 ml of ethanol. This mixture was added to RBF and was treated under microwave irradiation for 3 minutes; 210 watt. It was poured into 4 ml concentrated H$_2$SO$_4$. The mixture was stirred for 20 minutes with heating at 40-50°C and then was cooled. After that it was poured into 20 ml ice cold water, filtered and recrystallized with methanol. At last TLC was checked using hexane: ethyl acetate as solvent system in the ratio (7:3). **FT-IR (KBr\textit{\upsilon}\text{max cm}^{-1})**: 3002 (Ar C-H) str, 1558 and 1610 (C=C Arenes) str, 1264 (C-N Aromatic amine) str, 850 (C-Cl halo compound) str.
cm$^{-1}$): 752.26(C-H Ar-rings) bending, 1161.19 and 1558.54(C=C Arenes) str, 1262.45(C-N Ar
amine) str, 667.36(N-H) wagging. **NMR (400 MHz, DMSO, δ/ ppm):** 7.8-8.0 (m, 9H, aromatic), 7.4 (m, 2H, CH$_2$), 8.1 (m, 1H, NH), 3.6 (s, 1H, NHar).

The yield analysis of the synthesized compounds is given in Table 3.

**TABLE 3: YIELD ANALYSIS OF SYNTHESIZED COMPOUNDS**

| Comd. | Molecular formula | Molecular Weight (g/mol) | Reaction Time; watt of MWI | Recrystallization Solvent | Rf | Yield (%) | Melting point |
|-------|-------------------|---------------------------|----------------------------|----------------------------|----|------------|---------------|
| 1     | C$_7$H$_6$ClN$_3$  | 167                       | 3 minutes; 300 watt         | Water                      | 0.51 | 84         | 134°C         |
| 2     | C$_{14}$H$_{14}$N$_4$ | 246                      | 4 minutes; 240 watt         | Chloroform                 | 0.65 | 71         | 129°C         |
| 3     | C$_7$H$_9$N$_5$    | 163                       | 4 minutes; 240 watt         | Chloroform                 | 0.60 | 93         | 154°C         |
| 4     | C$_{12}$H$_{11}$N$_5$ | 225                      | 3 minutes; 210 watt         | Methanol                   | 0.57 | 88         | 141°C         |

**ANTICORROSIVE STUDY:**

The iron coupons (5x 2x 0.2 cm) were washed with distilled water and cleaned with acetone. Coupons were weighed and processed in acidic medium (0.5M H$_2$SO$_4$) with four synthesized derivatives of benzotriazole at conc. 100mg/L, 200mg/L and 300mg/L for 24 hours. One control was set which was made to run without adding compound (only 0.5M H$_2$SO$_4$ with iron coupon of weight 8.35g). The process followed is illustrated as given below (Fig. 1).

**Fig. 1: 24 hours analysis of weight loss with derivatives of benzotriazole**
The iron coupons were analysed for pre and post corrosion treatment i.e. before and after putting them in acidic medium in each case. The status of iron coupons is displayed below (Fig.2).

**Fig. 2: Iron coupon after analysing for 24 hours in control and a benzotriazole derivative**

The corrosion study was carried out with a set of two samples (A and B) for control and derivatives and the average of the data collected is taken. The weight loss was analysed in each case and result is given in Table 4.

**TABLE 4: AVERAGE WEIGHT LOSS OF IRON STRIP IN 0.5 H₂SO₄ SOLUTION**

| Compd. | Control (0mg/L) | 100mg/L | 200mg/L | 300mg/L |
|--------|----------------|---------|---------|---------|
| 1      | 3.3g           | 0.99g   | 0.83g   | 0.53g   |
| 2      | 3.3g           | 0.75g   | 0.49g   | 0.36g   |
| 3      | 3.3g           | 0.30g   | 0.22g   | 0.10g   |
| 4      | 3.3g           | 0.65g   | 0.51g   | 0.34g   |

**Determination of Corrosion rate (CR):**

The corrosion rate for synthesized compounds 1, 2, 3 and 4 were determined by weight loss analysis method. The weighed iron coupons under consideration was introduced into the process, and later removed after 24 hours. The strip was then cleaned for all corrosion products and is reweighed. The calculated corrosion rate for synthesized compounds is given in Table 5.
TABLE 5: CORROSION RATE OF SYNTHESIZED COMPOUNDS IN 0.5 M ACIDIC SOLUTION

| Compound | Inhibitor Concentration (mg/L) | Weight before corrosion (g) (avg.) | Weight after corrosion (g) (avg.) | Weight Loss (g) (avg.) | Corrosion rate (CR) (mm/y) |
|----------|-------------------------------|-----------------------------------|---------------------------------|------------------------|---------------------------|
| Control  | 0                             | 8.35                              | 5.05                            | 3.3                    | 67.28                     |
| 1        | 100                           | 8.19                              | 7.20                            | 0.99                   | 20.18                     |
| 1        | 200                           | 8.23                              | 7.40                            | 0.83                   | 16.92                     |
| 1        | 300                           | 8.52                              | 7.99                            | 0.53                   | 10.80                     |
| 2        | 100                           | 8.31                              | 7.56                            | 0.75                   | 15.29                     |
| 2        | 200                           | 8.35                              | 7.86                            | 0.49                   | 9.99                      |
| 2        | 300                           | 8.43                              | 8.07                            | 0.36                   | 7.34                      |
| 3        | 100                           | 8.08                              | 7.78                            | 0.30                   | 6.11                      |
| 3        | 200                           | 8.46                              | 8.24                            | 0.22                   | 4.48                      |
| 3        | 300                           | 8.33                              | 8.23                            | 0.10                   | 2.03                      |
| 4        | 100                           | 8.58                              | 7.93                            | 0.65                   | 13.25                     |
| 4        | 200                           | 8.16                              | 7.65                            | 0.51                   | 10.39                     |
| 4        | 300                           | 8.28                              | 7.94                            | 0.34                   | 6.93                      |

RESULT AND DISCUSSION:

For compound 1-(Chloromethyl)-1H-Benzotriazole, weight loss as compared to control (3.3g) was less and was getting reduced to 0.99g, 0.83g and 0.53g for concentration 100mg/L, 200mg/L, 300mg/L in 0.5M acidic solution respectively. Similarly, CR was getting reduced to 20.18, 16.92, 10.80mm/h, respectively. The graph is shown as given below (fig. 3). Similarly for compound N-(benzo[e][1, 2, 4] triazin-4(3-H)-ylmethylbenzenamine, weight loss as compared to control (0.33mg) was less and was getting reduced to 0.75mg, 0.49mg and 0.36mg for concentration 100mg/L, 200mg/L, 300mg/L in 0.5M acidic solution respectively. Similarly, CR was getting reduced to 15.29, 9.99 and 7.34mm/h, respectively. The graph is shown as below (Fig. 4).
For compound 1-((1-H-benzo[d][1,2,3]triazol-1-yl)methyl)hydrazine, weight loss as compared to control was less and was getting reduced to 0.30mg, 0.22mg and 0.10mg for concentration 100mg/L, 200mg/L, 300mg/L in 0.5M acidic solution respectively. Similarly, CR was getting reduced to 6.11, 4.48, 2.03mm/h, respectively. The graph is shown in Fig. 5. For compound 1-((1-H-benzo[d][1,2,3]triazol-1-yl)methyl)phenyl hydrazine, weight loss as compared to control was less and was getting reduced to 0.65mg, 0.51mg and 0.34mg for concentration 100mg/L, 200mg/L, 300mg/L in 0.5M acidic solution respectively. Similarly, CR was getting reduced to 13.25, 10.39, 6.93mm/h, respectively. The graph is shown in Fig. 6.
Comparative study of CR for synthesized compounds at three different concentrations: The comparative analysis of the synthesized compounds with respect to 100mg/L, 200mg/L and 300mg/L is given in Fig. 7.

![Bar chart showing comparative analysis of CR for compounds 1, 2, 3, and 4 at 100mg/L, 200mg/L, and 300mg/L concentrations.]

**Fig. 7: Comparative analysis of CR for compd. 1, 2, 3 and 4.**

7. CONCLUSION:

All the synthesized benzotriazole derivatives are found effective corrosion inhibitors. Compounds 1-(Chloromethyl)-1H-Benzotriazole; 1, N-(benzo[e][1, 2, 4] triazin-4(3-H)-yl)methylbenzenamine; 2, 1-((1-H-benzo[d][1,2,3]triazol-1-yl)methyl)hydrazine; 3 and 1-((1-H-benzo[d][1,2,3]triazol-1-yl)methyl)phenyl hydrazine; 4 demonstrated remarkable anticorrosive activity at 300mg/L concentration and least anticorrosive activity at 100mg/L concentration in 0.5 M H₂SO₄ solution.

At 100mg/L, the effectiveness of corrosion inhabitancy of synthesized compounds follow the order 3, 4, 2 and 1.

At 200mg/L, the effectiveness of corrosion inhabitancy of synthesized compounds follow the order 3, 2, 4 and 1.

At 300mg/L, the effectiveness of corrosion inhabitancy of synthesized compounds follow the order 3, 4, 2 and 1.

Compound 1-((1-H-benzo[d][1,2,3]triazol-1-yl)methyl)hydrazine; 3 emerges as a leader in this study which shows potent anticorrosive activity against mild steel at all concentration in 0.5 M H₂SO₄ solution. Similar observation is obtained for compound 1-(Chloromethyl)-1H-Benzotriazole; 1, which shows moderate anticorrosive activity against mild steel at all concentration in 0.5 M H₂SO₄ solution.
CONFLICT OF INTEREST
The authors declare that they have no conflict of interest.

ACKNOWLEDGEMENT
The authors are very thankful to Dr. Ramesh Thakur, HOS, Lovely Professional University, Punjab for providing the research facilities.

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