Asian Journal of Chemistry; Vol. 37, No. 10 (2025), 2599-2604
ASIAN JOURNAL OF CHEMISTRY
https://doi.org/10.14233/ajchem.2025.34540

# Manganese(III) Complexes of Porphyrin-based Ligand: Synthesis, DFT Study and Catalytic Application

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*Received*: 26 July 2025 *Accepted*: 25 September 2025 *Published online*: 30 September 2025 AJC-22145

Four new Mn(III) porphyrin complexes with 1,2-diaminoethane were synthesized, with the general formula [$Mn^{III}$(TEHPP)X(dae)], where TEHPP is 5,10,15,20-tetra(3-ethoxy-4-hydroxyphenyl)porphine, X represents $Cl^-$, $Br^-$, $NCS^-$ or $N_3^-$ and 'dae' is 1,2-diaminoethane. These complexes were characterized with ultraviolet-visible (UV-visible) spectroscopy, Fourier transform-infrared (FT-IR), elemental analyses, magnetic susceptibility measurements and conductivity measurements. The synthesized novel Mn(III) porphyrins exhibit catalytic activity in the oxidation of aromatic alcohols to aldehydes. The catalytic performance of these complexes was evaluated for the oxidation of benzyl alcohol derivative to corresponding aldehyde at room temperature (30 ºC) using oxidants such as $NaIO_4$, $H_2O_2$ and NaOCl. Density functional theory (DFT) calculations were also conducted to optimize the complex structures and HOMO–LUMO energies were determined using NBO analysis at the same theoretical level.

**Keywords: Mn(III) porphyrins, 5,10,15,20-Tetra(3-ethoxy-4-hydroxyphenyl)porphine, 1,2-Diaminoethane, Catalytic activity.**

## INTRODUCTION

Metalloporphyrins are broadly found in nature and found to be engaged in different biochemical reactions therefore they are named as pigments of life [1]. Metalloporphyrins are very efficient functional systems since they show catalytic reactions and various transformation chemical reactions [2-7]. This nature is due to the fast electron dynamics and effective energy transfer taking place between the transition M (metal) and its coordinated-nitrogen ligands [2]. The M-to-L or L-to-M charge transfer is done by the mixing of M-L orbitals [2].

Evolution of manganese reliant oxygen in green plant photosynthesis [8,9] oxidation catalysis [9,10] and oxidative DNA cleavage [11-14] involve the significant role of manganese porphyrins. The outer ligands can be designed in such a way to improve the electronic structure as required without interfering with the chemical composition in the porphyrin central part (M centre and coordinated-N ligands) [15].

Few manganese porphyrins previously synthesized in our lab have demonstrated efficient catalytic activity for alcohol oxidation under mild conditions and short reaction times [16]. Based on this, our research group has extensively reported the diverse catalytic capabilities of Mn(III) porphyrins across various reactions [17-20]. In view of their broad potential, we now report the synthesis, characterization and catalytic application of novel Mn(III) porphyrin complexes in the oxidation of benzyl alcohol derivatives.

## EXPERIMENTAL

4-Methoxybenzyl alcohol and 1,2-diaminoethane were procured from Chemical Centre, Mumbai, while ethyl vanillin (3-ethoxy-4-hydroxybenzaldehyde), hydrogen peroxide and sodium periodate were obtained from S.D. Fine-Chem Ltd., India; sodium hypochlorite was sourced from Merck, India. All chemicals used were of analytical reagent (A.R.) grade and were purified and dried as required, following standard procedures [21-23]. Pyrrole from Merck, Germany and acetylacetone from S.D. Fine-Chem Ltd., India were freshly distilled

before use. Distilled water was used throughout the experimental procedures to ensure accuracy and purity of results.

**Physical measurements:** FT-IR spectral study was done with Shimadzu IR Spirit FT-IR-8000 spectrophotometer using KBr pellets (4000-400 $cm^{-1}$). Electronic spectra were obtained on spectrophotometer UV-1900 series from 200-800 nm using chloroform as solvent. Elemental analyses were conducted using a Euro Vector E 3000 Elemental Analyzer, with data obtained from SAIF, CDRI, Lucknow, India, while magnetic susceptibility measurements at room temperature were performed using a Vibrating Sample Magnetometer. Electrical conductivity data were recorded in our laboratory at room temperature using a TOSHCON Auto Ranging Conductivity Meter TCM 15+, with $10^{-3}$ M solutions prepared in ethanol.

**Synthesis of ligand:** 5,10,15,20-Tetra(3-ethoxy-4-hydroxyphenyl)porphyrine ligand ($H_2$TEHPP) was synthesized by following a modified Adler's method [24]. Freshly distilled pyrrole (0.08 mol, 5.6 mL) and 3-ethoxy-4-hydroxybenzaldehyde (0.08 mol, 13.28 g) were added to boiling propionic acid (300 mL) and refluxed for 4 h. After cooling to room temperature, the tarry insoluble product was filtered, washed thoroughly with methanol until the filtrate was colourless and the resulting purple, shiny crystals were recrystallized from a 1:1 mixture of dichloromethane and chloroform.

**Synthesis of [$Mn^{III}$(TEHPP)X]:** For the synthesis of complexes of Mn(III) porphyrin, [$Mn^{III}(acac)_2X$] was initially synthesized, where 'acac' represents acetylacetonate and X shows $Cl^-$, $Br^-$, $NCS^-$ or $N_3^-$, that have role as intermediates in these syntheses. These bis(acetylacetonato)Mn(III) complexes readily coordinate with porphyrins to form Mn(III) porphyrin complexes [25-27]. In brief, [$Mn^{III}(acac)_2X$] and [$H_2$TEHPP] were taken in equimolar ratio (0.003 mol) in a round bottom flask of 500 mL capacity containing glacial acetic acid (105 mL) and acetic anhydride (45 mL). Complete the reaction mixture was refluxed for 4 h. After the solvent evaporation on a steam bath, dark green solid complexes were obtained and subsequently recrystallized using a 1:1 mixture of chloroform and methanol. The resulting complexes were crystalline and non-hygroscopic in nature (**Scheme-I**).

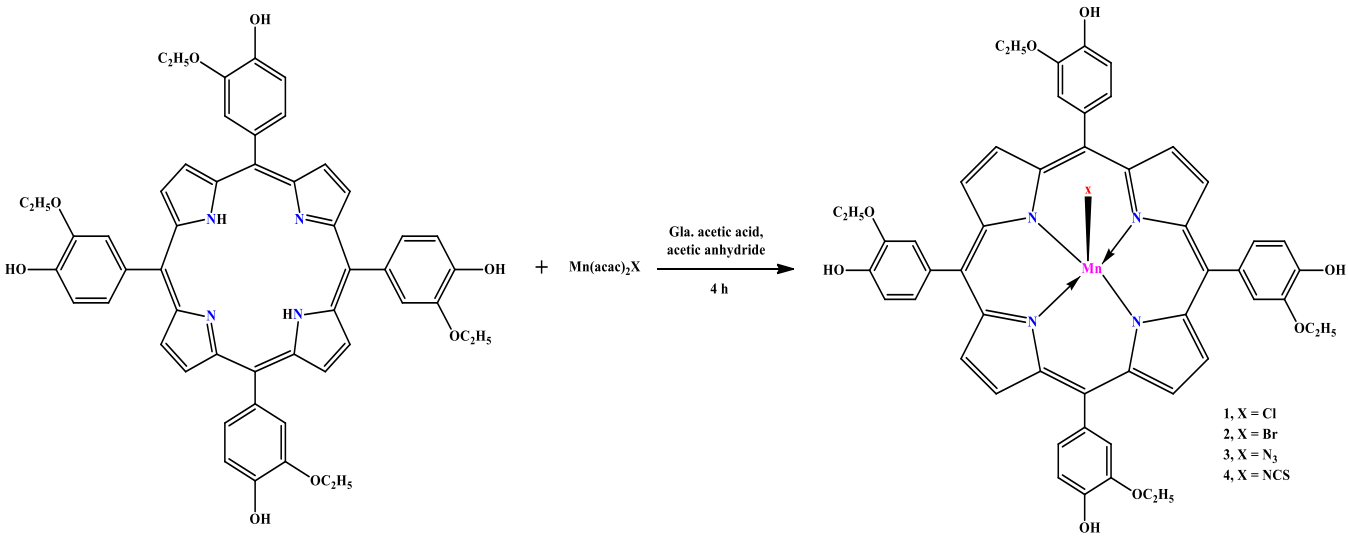
**Synthesis of Mn(III) porphyrin complexes with 1,2-diaminoethane:** A solution of [$Mn^{III}$(TEHPP)X] (0.001 mol) in 4 mL ethanol was treated with 1,2-diaminoethane (0.03 mol), stirred for 20 min at room temperature and then refluxed for 26–30 h. The reaction mixture was cooled to room temperature for 1 h and dried under vacuum over $P_2O_5$, yielding black coloured complexes in all cases (**Scheme-II**). Due to their hygroscopic nature, the [$Mn^{III}$(TEHPP)X(dae)] complexes were stored in a desiccator.

**Catalytic activity:** The oxidation of an aromatic alcohol to its corresponding aldehyde was carried out using a mixture of acetonitrile (5 mL), alcohol (0.5 mL) and Mn(III) complex (0.01 g), followed by the addition of 0.002 mol oxidant dissolved in 5 mL of water. The reaction was stirred at room temperature using a magnetic stirrer and a 1 mL aliquot was withdrawn after 10 min for analysis using a UV-1900 series UV-Visible spectrophotometer.

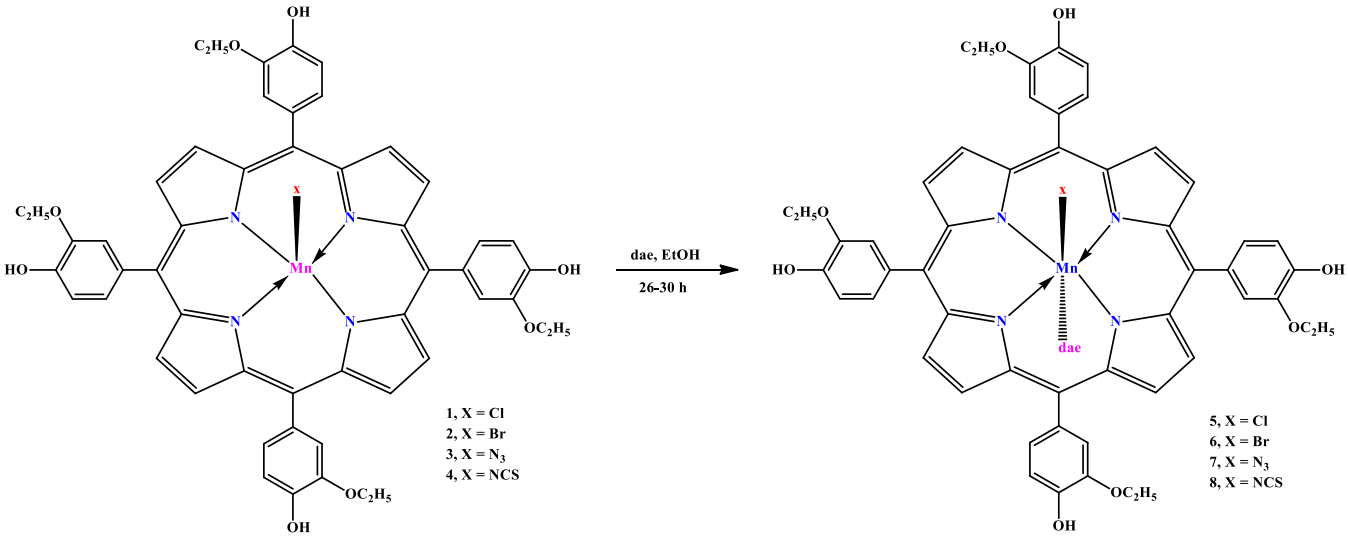
## RESULTS AND DISCUSSION

All synthesized manganese(III) porphyrin complexes were characterized by different spectroscopic techniques. The Mn(III) complexes are readily soluble in common solvents like ethanol, acetone, acetonitrile and chloroform. The physical properties and analytical data of these complexes are shown in Table-1.

**Electronic spectral studies:** An intense B (Soret) band at ~400 nm and several weaker Q bands at ~480-700 nm are generally shown by the normal metalloporphyrin spectrum. These spectral absorptions are due to aromatic porphyrin ligand's π-π* transitions [28,29]. The electronic absorption spectra of Mn(II) porphyrin complexes were obtained in $CHCl_3$, B band and Q bands of these complexes are shown in Table-2. [Mn(TEHPP)X] complexes exhibit four Q bands. After the coordination with 1,2-diaminoethane, two bands (out of these 4 bands) disappear. Further, red shift and blue shift are shown by B bands and Q bands of [Mn(TEHPP)X(dae)] complexes respectively, while comparing with the spectra of their parent complexes *i.e.* [Mn(THMPP)X]. Such wavelength shifts are due to the axial ligation in these complexes [30].




**Scheme-I:** Schematic diagram for synthesis of complexes **1-4**




**Scheme-II:** Schematic diagram for synthesis of complexes **5-8**

TABLE-1
DATA OF PHYSICAL PROPERTIES AND ELEMENTAL ANALYSIS FOR Mn(III) PORPHYRINS

| Complex | m.f. | Colour | Yield (%) | Elemental analyses (%): Found (calcd.) | | | | | | | | Molar conductivity ($\Omega^{-1}$ cm$^2$ mol$^{-1}$) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | C | H | N | O | S | Cl | Br | Mn | |
| **5** | $C_{54}H_{52}ClMnN_6O_8$ | Black | 79.25 | 64.64 (64.60) | 5.22 (5.24) | 8.38 (8.35) | 12.76 (12.79) | – | 3.53 (3.51) | – | 5.48 (5.52) | 11 |
| **6** | $C_{54}H_{52}BrMnN_6O_8$ | Black | 74.40 | 61.90 (58.90) | 5.00 (6.00) | 8.02 (10.02) | 12.21 (11.49) | – | – | 7.63 (8.05) | 5.24 (5.56) | 10 |
| **7** | $C_{54}H_{52}MnN_9O_8$ | Black | 78.20 | 64.22 (60.20) | 5.19 (4.99) | 12.48 (15.70) | 12.67 (14.89) | – | – | – | 5.44 (4.44) | 8 |
| **8** | $C_{55}H_{52}MnN_7O_8S$ | Black | 79.60 | 64.38 (59.40) | 5.11 (7.09) | 9.56 (8.56) | 12.47 (15.97) | 3.12 (3.52) | – | – | 5.35 (5.45) | 12 |

TABLE-2
Mn(III) PORPHYRINS' UV-VIS
SPECTROSCOPIC DATA IN CHLOROFORM

| Complex | λ (nm) | |
|---|---|---|
| | Soret (B) band | Q bands |
| [Mn(TEHPP)Cl(dae)] (**5**) | 475 | 575, 620 |
| [Mn(TEHPP)Br(dae)] (**6**) | 478 | 572, 620 |
| [Mn(TEHPP)$N_3$(dae)] (**7**) | 475 | 575, 620 |
| [Mn(TEHPP)NCS(dae)] (**8**) | 478 | 572, 620 |

TABLE-3
CALCULATED VALUES OF LIGAND FIELD PARAMETERS

| Complex | 10Dq (cm$^{-1}$) | B (cm$^{-1}$) | B |
|---|---|---|---|
| [Mn(TEHPP)Cl(dae)] | 21,053 | 780 | 0.68 |
| [Mn(TEHPP)Br(dae)] | 20,920 | 775 | 0.68 |
| [Mn(TEHPP)$N_3$(dae)] | 21,053 | 780 | 0.68 |
| [Mn(TEHPP)NCS(dae)] | 20,920 | 775 | 0.68 |

The ligand field parameters *i.e.* 10 Dq, B and β have also been calculated and the values are reported in Table-3. $\lambda_{max}$ values at maximum absorbance are utilized to calculate 10 Dq. Racah inter-electronic repulsion parameter (B) was calculated utilizing a Tanabe-Sugano diagram whereas following formula was used to calculate the β values:

$$\beta = \frac{B_{complex}}{B_{free\ ion}}$$

where B free ion for $Mn^{3+}$ = 1140 cm$^{-1}$ [31]. The value of β (covalency factor) is less than 1, which suggests the covalent nature of M-L bond in all the Mn(III) porphyrin complexes.

**FT- IR spectral studies:** The main absorption bands of FT-IR spectra for all the newly synthesized Mn(III) porphyrin complexes are reported in Table-4. Stretching frequencies in the range of 1400-1380 cm$^{-1}$ and at 1678 cm$^{-1}$ correspond to C=N and C=C bonds of pyrrole rings. A medium peak at 2041 cm$^{-1}$ suggests coordination of $N_3^-$ [32]. The characteristic peak at 2049 cm$^{-1}$ confirms the coordination of $NCS^-$ [33]. The (Mn–N) was characterized by the presence of an absorption at ~431, 424 cm$^{-1}$ for all the Mn(III) complexes [34,35]. The occurrence of broad bands at 3504, 3520 cm$^{-1}$ indicates the presence of O-H bonds. The asymmetric $NH_2$ stretching frequency for ethylenediamine occurs at 3440-3250 cm$^{-1}$, while in [Zn(dae)$Cl_2$] it shifts to 3290-3230 cm$^{-1}$, indicating the chelation of ethylenediamine (dae) to the metal center [36]. While in the case of these Mn(III) porphyrins ($NH_2$) appears at 3404 cm$^{-1}$ showing the presence of a free amine group and at 3373 cm$^{-1}$ corresponding to the amine group that has coordinated with the metal ion. Characteristic bands of diamines appear between 1400-700 cm$^{-1}$. A strong band at 1335 cm$^{-1}$ has been assigned to ($NH_2$). The C-N stretching modes of all

TABLE-4
SELECTED VIBRATIONAL FREQUENCIES ($cm^{-1}$) IN FT-IR SPECTRA OF MANGANESE(III) PORPHYRINS WITH 1,2-DIAMINOETHANE

| [Mn(TEHPP)Cl(dae)] | [Mn(TEHPP)Br(dae)] | [Mn(TEHPP)$N_3$(dae)] | [Mn(TEHPP)NCS(dae)] | Assignment |
|---|---|---|---|---|
| 431 | 424 | 431 | 424 | ν(Mn-N) |
| 1082 | 1082 | 1082 | 1082 | ν(O-$C_2H_5$) |
| 1399 | 1384 | 1399 | 1384 | ν(C=N)$_{py}$ |
| 1678 | 1678 | 1678 | 1678 | ν(C=C)$_{py}$ |
| – | – | – | 2049 | ν(NCS) |
| – | – | 2041 | – | ν($N_3$) |
| 3504 | 3520 | 3504 | 3520 | ν(OH) |
| 3373 | 3373 | 3373 | 3373 | ν($NH_2$)$_{coordinated}$ |
| 3404 | 3404 | 3404 | 3404 | ν($NH_2$)$_{uncoordinated}$ |

uncomplexed diamines appear at 1090-1070 $cm^{-1}$ [37]. In the newly synthesized Mn(III) complexes, (C-N) band appears at 1012-1004 $cm^{-1}$, the downward shift shows the coordination of nitrogen to manganese. The $NH_2$ rocking mode is a strong band at 602-594 $cm^{-1}$. Two medium peaks at 803 $cm^{-1}$ and 873 $cm^{-1}$ indicate $CH_2$ rocking mode. The (C–C) bond has been assigned as a strong band at 1019 $cm^{-1}$ [38]. The above data indicate that 1,2-diaminoethane has coordinated with Mn(III) as a unidentate ligand.

**Magnetic susceptibility:** The magnetic moment of the newly synthesized Mn(III) porphyrin complexes ranged from 4.78 to 4.98 B.M., indicating the presence of four unpaired electrons and confirming their high-spin Mn(III) nature [39-41].

**Description of structure:** Based on the aforementioned data and accessible literature [42,43], an octahedral geometry has been proposed for Mn(III) complexes with 1,2-diaminoethane. The reported complex, [$Mn^{III}$(TEHPP)X(dae)], features a central Mn(III) ion in an octahedral coordination environment. The porphyrin, acting as a tetradentate ligand, provides four nitrogen donor atoms arranged in a square planar geometry around the metal centre. This configuration forms a robust equatorial framework due to the conjugated π-system of the porphyrin ring, which stabilizes the metal-ligand interactions. The axial positions of the octahedral geometry are occupied by $Cl^-$, $Br^-$, $NCS^-$ or $N_3^-$ and 1,2-diaminoethane. The proposed structure is presented in Fig. 1.

**Computational (DFT) studies:** The computational studies were conducted on one of the newly synthesized complexes, [Mn(TEHPP)Cl(dae)], due to the similar structural pattern observed in all the Mn(III) complexes and the porphyrin-based structure, which requires several days for the structural optimization and other computational analyses. We have selected Minnesota M06L functional [44] along with the 6-31+G (d,p) basis set. The M06L functional was developed by Zhao & Truhlar [44] and provides better performance for transition metals, inorganic and organometallics. Furthermore, we have performed frequency calculations of the electronic structures of the Mn(III) porphyrin complex to obtain different modes of frequencies. Frequency calculations yielded the real and positive vibrational frequencies, indicating the stability of the complex. NBO calculations were also performed at the same level of theory to determine the highest occupied molecular orbital (HOMO), lowest unoccupied molecular orbital (LUMO) and their energy gap. All DFT calculations were carried out using the Gaussian 09 software package [45].

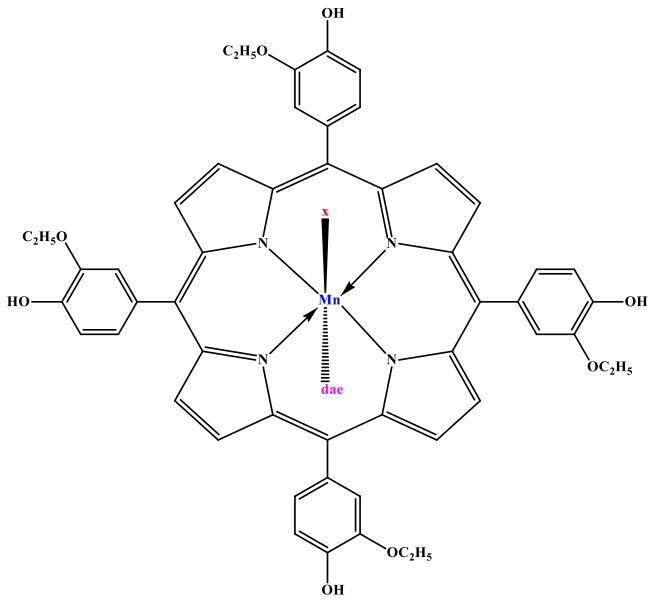



Fig. 1. Tentative structure of [Mn(TEHPP)X(dae)] complex

**Electronic structure:** Electronic structure of the [Mn(TEHPP)Cl(dae)] complex is shown in Fig. 2 along with some impotent bond parameter (Å). It is obvious from Fig. 2 that the distances of metal Mn-atom with four N-atoms are found to be 2.036, 2.067, 2.018 and 1.967 Å respectively. One Mn-N bond was found to be slightly close to other three Mn-N bond. In addition to these bonds, two other bonds with Mn-atom *i.e.* Mn-Cl bond and Mn-N bond (N-atom of

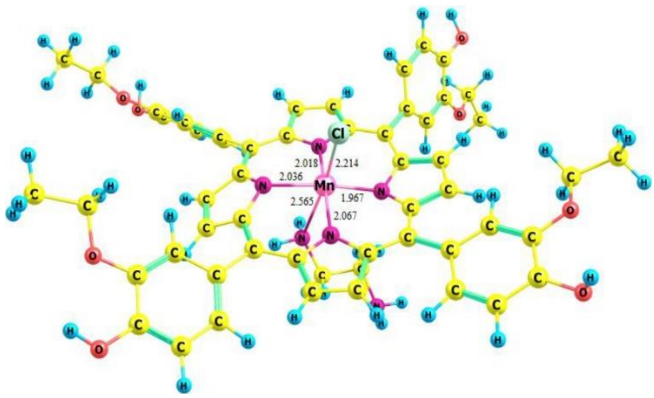



Fig. 2. Optimized structure of [Mn(TEHPP)Cl(dae)] complex at M06L/6-31+G(dp) level of theory

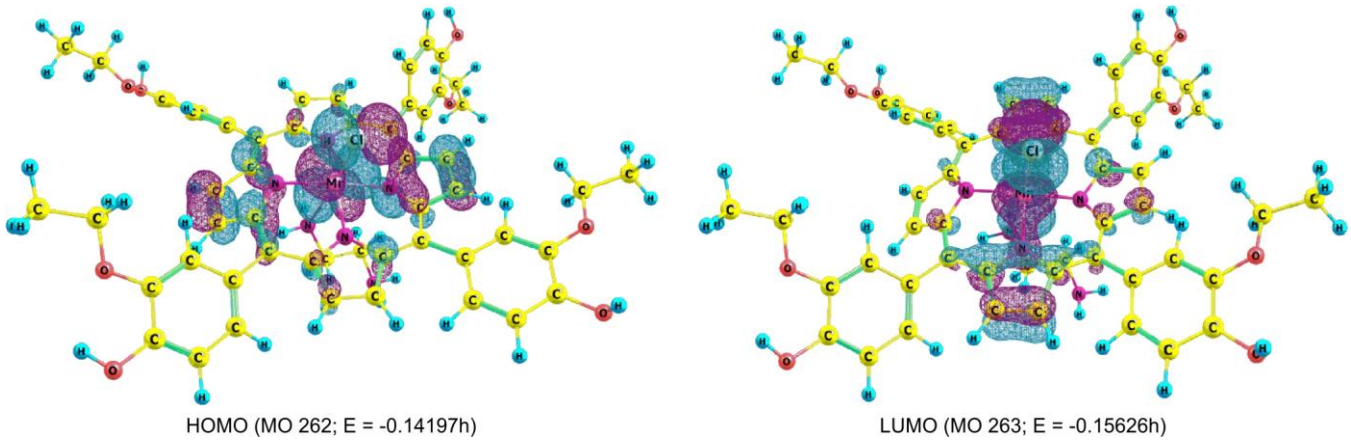
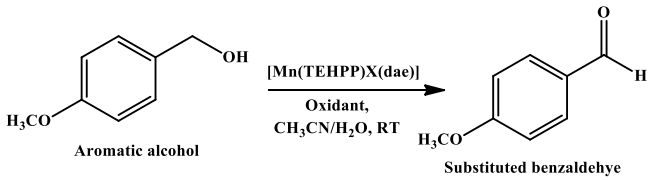



Fig. 3. HOMO and LUMO of [Mn(TEHPP)Cl(dae)]

$NH_2CH_2CH_2NH_2$) are formed which are found to be 2.214 and 2.565 Å. HOMO-LUMO isosurfaces of the complex are shown along with their energy in Fig. 3.

**Application as catalysts:** All the synthesized Mn(II) porphyrin complexes were utilized as catalysts for checking it role in the oxidation of aromatic alcohol to their corresponding aldehyde. While [$Mn^{III}$(TEHPP)X(dae)], X = $Cl^-$, $Br^-$, $NCS^-$, $N_3^-$ oxidized 4-methoxybenzyl alcohol into 4-methoxybenzaldehyde and further oxidation of aldehyde into respective carboxylic acid was not observed (**Scheme-III**).




**Scheme-III:** Aromatic alcohol's oxidation to corresponding aldehyde using [Mn(TEHPP)X(dae)]

In this conversion, study was done in the presence of three types of oxidants like $NaIO_4$, $H_2O_2$ and NaOCl, which were used as oxygen source. Out of these three oxidants, $NaIO_4$ was the most effective for the conversion reaction. In the course of oxidation, the Soret band of the complexes recedes slowly, which affirms the consumption of complexes with time. The spectral changes for reaction solution containing the complex [$Mn^{III}$(TEHPP)X(dae)] with time is shown in Fig. 4.

Product formation was assessed using UV-Visible, TLC and qualitative tests for benzaldehyde. As the Mn(III) porphyrin complex is consumed during the catalytic oxidation reaction, therefore, it cannot be reused. The results of oxidative transformations are summarized in Table-5. In the blank experiments *i.e.* same experimental conditions but in the absence of Mn(III) complex, no such spectral changes were observed. The purpose of this study was to demonstrate the involvement of the synthesized Mn(III) porphyrin complexes in the oxidation of aromatic alcohols, which was confirmed by UV-Visible spectroscopy, TLC and qualitative tests for benzaldehyde. Therefore, detailed spectral characterization of the product was not undertaken. Study suggests that Mn(III) porphyrin complexes may also act as catalytically significant catalysts like Mn (III) Schiff base complexes [46].

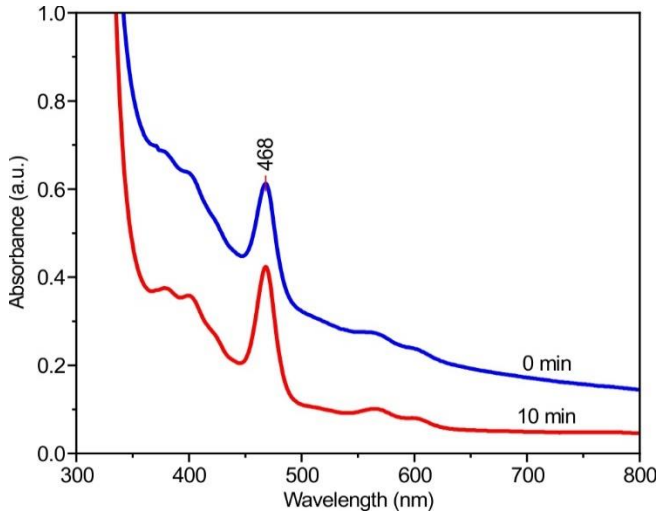



Fig. 4. UV spectra depicting the catalytic conversions

TABLE-5
RESULTS OF CATALYTIC OXIDATION REACTION

| Entry | Oxidant | Solvent | Aldehyde yield (%) after 10 min |
|---|---|---|---|
| 1 | $NaIO_4$ | $CH_3CN/H_2O$ | 40 ± 5 |
| 2 | $H_2O_2$ | $CH_3CN$ | 16 ± 5 |
| 3 | NaOCl | $CH_3CN$ | 6 ± 5 |

## Conclusion

The synthesis, characterization and catalytic properties of manganese(III) porphyrin complexes with 1,2-diaminoethane were successfully achieved. Based on physical and spectral analyses, these novel Mn(III) porphyrin complexes have been proposed to possess an octahedral structure, featuring an unusual monodentate coordination of 1,2-diaminoethane, as confirmed by IR spectral studies. The catalytic application of these complexes in the conversion of 4-methoxy benzyl alcohol into its corresponding aldehyde was also conducted. Among the three oxidants $NaIO_4$, $H_2O_2$ and NaOCl; $NaIO_4$ is the most potent oxidant which gave the maximum yield. The computational study was used to optimize the [Mn(TEHPP)Cl(dae)] structure and to get the HOMO, LUMO and energy gap. The Mn-N bond lengths were determined for all the four Mn-N bonds.

## ACKNOWLEDGEMENTS

The authors are thankful to Prof. R.K. Yadav, Department of Chemistry, Madan Mohan Malaviya University of Technology, Gorakhpur, India for providing the help in recording the spectra.

## CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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