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REVIEW ARTICLE

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## REVIEW ON: THIOLATE BASED METAL COMPLEXES AS POTENTIAL ADVANCED MATERIAL FOR ELECTRONIC INDUSTRIAL AND BIO-MEDICAL APPLICATIONS

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### ABSTRACT

The dithiolene ligands and their complexes are well recognized by researchers. The diverse bonding behavior and versatility in chemical nature resulting from electronically delocalized pathway for electrons in the structure of these complexes. These specific features make them very promising raw molecular compounds synthesis of wide variety of magnetically and electronically important materials. Several main group, alkali metal and transition metal complexes with conductive, semi conductive and super conductive properties have been reported. In material science the applications of metal dithiolates cover the wide spread fields like special magnetic behavior (ferromagnetism, ferrimagnetism etc). In this article the semiconductor behavior of monometallic and homo metallic, hetero bimetallic as well as poly-metallic complexes have been reviewed. The conventional semiconductor molecular materials are used in semiconductor industries. They have some limitations regarding temperature resistance, high frequency and high voltage energy harvesting applications. The metal dithiolates have potency to solve these issues and they can be used as wide band semiconductor compounds. The wide band gap complexes also have appreciable importance in defence research, signal systems, vehicle development etc. There are several biologically important and medicinal complexes have also been reported. The present paper deals with the chemistry of metal dithiolates including bonding patterns, synthetic strategy and multifarious applications in electronics and biomedical fields.

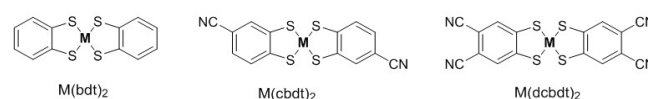
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## INTRODUCTION

Dithiolene complexes of transition metal have garnered significant attention for nearly four decades and have emerged as crucial entities in diverse scientific disciplines such as material science, analytical chemistry, catalysis, and biological systems [1]. These complexes exhibit variable oxidation states, influenced by the kind of ligand nature, which actively participate in redox processes. The transition metal itself and its oxidation state dictate the magnetic properties of these complexes, leading to a variety of magnetic moments. Consequently, their versatility in oxidation states and magnetic behaviours have positioned them as preferred building materials with the development of advanced materials with both magnetic and conducting functionalities. These structural characteristics facilitate enhanced solid-state intermolecular interactions. Researchers have focussed their attention on exploring novel complexes featuring diverse ligands having multiple oxidation states, as the redox processes Manuel Almeida et al [2] studied ligand 4,5-dicyanobenzene-1,2-dithiolate (dcbdt), for which they have reported a series of complexes  $[M(dcbdt)_2]_z$  where  $M = Au, Pt, Ni, Pd, Cu, Co$ , and Fe.

These complexes exhibit a range of ( $z = 0.4, 1, 2$ ), [2]. This type of complexes have got attention due to their potential as highly electrically conductive compounds [3]. Some complexes show partially oxidized form  $z = 0.4$ , exemplified by  $[n-Bu_4N]_2[M(dcbdt)_2]_z$  where ( $M = Ni, Au, Cu$ ), which exhibit interesting range of electrical conductance in solid state [4]. The several other complexes have also been synthesized using substituted derivatives of (cbdt) and it's iron complexes [5]. These complexes hold promise for optical and electronic applications [6] and as engineered materials with intriguing specific magnetic features [7]. The substituted cyanodithio complexes have proven effective in forming chains of heterometallic compounds via coordination of cyano group with the other metals [8]. The detail designing and characterization of  $[M(cbdt)_2]$  where ( $M = Cu, Ni, Au, Pd, Co, Zn$ ) with analogs containing unsubstituted benzene dithiolate (bdt) and (dcbdt) ligands have been reported (Fig.1).



**Fig 1.** Structure of  $[M(bdt)_2]$ ,  $[M(cbdt)_2]$  and  $[M(dcbdt)_2]$  [ $M = Ni, Zn, Co, Cu, Au \& Pd$ ]

Dithio ligands have garnered significant interest in the development of novel conducting materials for deployment in the semiconductor industry [9-10]. Exhibiting both mono- and bidentate characteristics, these ligands feature two donor sulphur atoms [11]. The coordination capabilities of these ligands manifest diverse facets of bonding and structural behaviour. Numerous metal complexes have been documented with these ligands, encompassing homoleptic and heteroleptic dithio carbamates [12], tri-thiocarbonates [13], xanthates, thio-xanthates,  $\pi$ -conjugated dithiolenes [14], and ligands incorporating heterocyclic substitutions in  $\pi$ -conjugated dithiolene systems. Prominent examples of 1,1-dithio ligands include dithio carbamates, xanthates, thio xanthates, dithiophosphates, isomalenonitriledithiolates, 1,1-dithio oxalates, and 1,1-dithio oxalate, and 1,1-dicarboethoxy-ethylene-2,2-dithiolates. Meanwhile,  $\text{dmit}^{2-}$  (1,3-dithiole-2-thione-4,5-dithiolate),  $\text{MNT}^{2-}$  (1,2-dicyano ethylene-1,2-dithiolate or maleonitriledithiolate), and  $\text{edt}^{2-}$  (ethane-1,2-dithiolate) stand out as widely employed 1,2-dithio ligands. The versatile binding affinities of these ligands have facilitated the expansion of their complexes from main group metals to transition metal complexes. These complexes exhibit diverse applications in the realm of material science, encompassing semiconducting industrial applications [15], non-linear optical fields [16], catalyst chemistry, the extraction of metal from contaminated samples [17], high-pressure lubricants, chromophore materials for (DSSC) dye-sensitized solar and the identification of metals in selected samples. Dithio metal complexes demonstrate extensive utility in the realms of biology and medicinal fields [18-19]. Particularly, dithio ligands derived from heterocyclic compounds are prominently featured in antibacterial [20-21], antifungal, and insecticidal research pertaining to dithio metal coordination complexes. The bonding characteristics of dithio ligands and their complexes are of paramount importance, serving as determinants for their functional response in the context of metal complexes. In essence, metal dithio ligand compounds encapsulate a comprehensive spectrum of molecular-level engineering within metal-ligand chemistry, tailored to diverse application in targeted fields.

## LITERATURE REVIEW

The chemistry of metal complexes is very rich. Voluminous literature related to the synthesis, characterization and application study have been collected. The keen interest of the scientist in this field is due to multifarious applications of various metal ligand complexes including monometallic [22], homo-bimetallic [23], hetero-bimetallic [24] and even poly-metallic [25] complexes, complex salts, polymers etc. several research groups have reported the molecular semiconductors, superconductors, non-linear optical materials, photo-cell sensitizers, catalysts, sensors, biologically and medicinally as well as agricultural field related (insecticide, pesticides etc) important compounds. The metal dithiolene ligands e.g.  $\text{MNT}^{2-}$  (maleo-nitrile dithiolate [26]) and  $\text{dmit}^{2-}$  (1,3-dithiole-2-thione-4,5-dithiolate [27]) have undergone a wide expansion in their chemistry for about three to four decades. The charge transfer salts of BEDT (Bis ethylene dithio) -TTF [28] (tetra-thia-fulvalene) and TTF (tetra thio fulvalene) show different conducting behaviour like metallic, semiconducting and superconducting features. The sulphur based dithio ligand complexes have also recognized to exhibit luminescent properties, NLO behaviours, unusual magnetism. Work on organic charge transfer complexes involving TCQD [29], chloranil [30] and p-phenyl diamine [31] with semiconducting and superconducting properties are also reported. The Fig.2 show the structure of ligand TCQD, Chloranil, TTF and p-phenyl diamine.

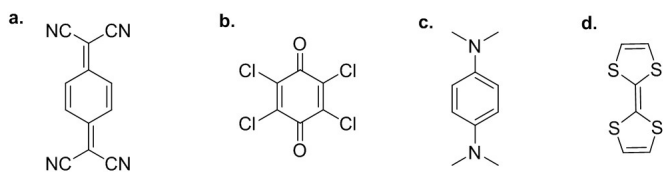


Fig. 2. (a). Tetracyanoquinodimethane (TCQD) (b).Chloranil (c).p-phenylene diamine (d). Tetra-thio-fulvalene

The complex of  $(\text{TTF})_2\text{Br}$  [32] is reported to show superconductivity property below 4.2K temperature and at 25K bar pressure. The 1,1-dithio and 1,2-dithio ligand have ability to form transition metal complexes in various oxidation states of the metal ion. Documentation of various co-ordination modes are available in literature. Like asymmetric and symmetric chelating, bridging and in few cases monodentate mode. The co-ordination polymeric compounds, bimetallic salt and mono as well as bi/poly-metallic complexes [33].

**Bonding behaviour of 1,1-dithio ligands:** In this chemical class [34], the two sulfur atoms are covalently bound to a common carbon atom within the ligand structure. These ligands exhibit a propensity for establishing stable complexes with transition metal species. Predominantly, they manifest in the formation of square planar coordination geometries in resulting complexes. The structures of 1,1-dithiolates are shown in Fig. 3.

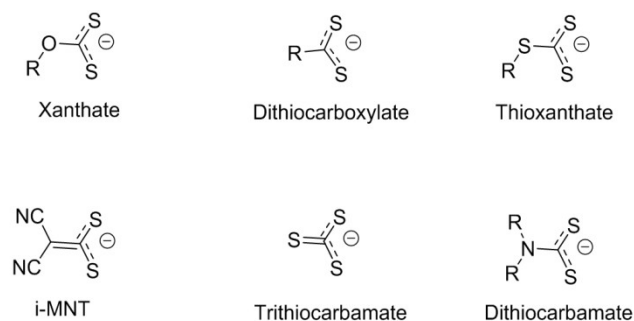


Fig. 3. Structure of 1,1-dithiolate ligand

**Bonding behaviour of 1,2-dithio ligands:** In this ligand category, the two sulfur atoms are covalently bonded to adjacent carbon atoms within the ligand molecule. The structure details of 1,2-dithiolates are represented in Fig. 4.

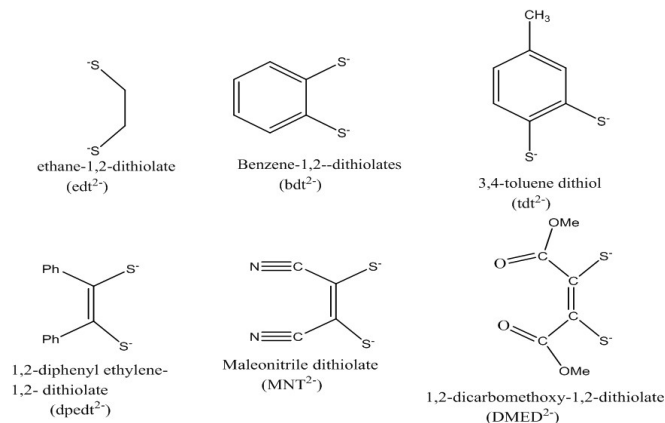


Fig. 4. Structure of 1,2-dithiolate ligand

**Bonding behaviour of 1,3-dithiolate ligands [35]:** In this category of ligands, the minimum carbons separating the two sulfur atoms in the dithio ligands. Bonding behaviour of dithioacetylacetonato complexes are shown in Fig.5.

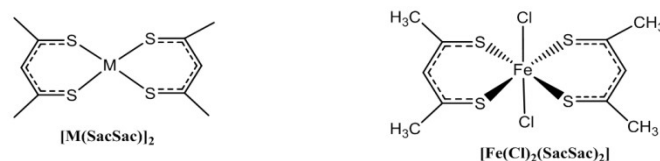


Fig. 5. Structure of 1,3-dithiolate ligand (sacsac - Dithioacetylacetonato)

**Synthesis of 1,1-dithio ligands:** 1,1-Dithiolates are typically synthesized as alkali metal salts. The synthetic pathways for several prevalent 1,1-dithiolate ligands are outlined as follows:

**Dithio-carbamate:** The synthesis of most dithiocarbamate salts is quite straightforward [36]. A variety of secondary and primary amines can react with carbon disulphide in water, using a base, to produce highly water-soluble dithiocarbamate salts. While many different bases can be employed, alkali-metal hydroxides are usually sufficient. The reactions typically occur quickly (within minutes) and can be quite exothermic, so it's advisable to perform them in an ice bath, particularly if the amine is volatile.



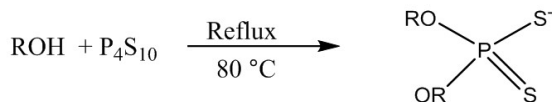
**Xanthate:** Alkoxy salt of alkali metal on reaction with carbon disulphide give alkoxy derivatives of 1,1-dithiolate named xanthate.



**Thio-xanthate:** Thio-alkoxy salt of alkali metal on reaction with carbon disulphide give thio-alkoxy derivatives of 1,1-dithiolate named thio-xanthate.



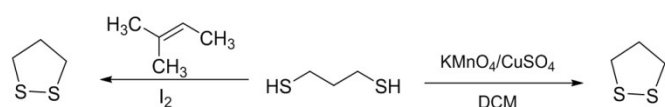
**Thio-phosphosphate:**



### Synthesis of 1,2-dithio ligands

**Preparation of MNT:** Cyanodithioformate-3-dimethylformamide  $NaS_2C.CN.3DMF$  was synthesized through the reaction of a suspension containing finely powdered dried sodium cyanide, carbon disulfide, and dimethylformamide following the procedure described by Bahr and Schleitzer [37].

**Preparation of 1,2-dithiolane [38]:** The scheme for preparation of 1,2-dithiolane is represented as follow.



**Preparation of Ethane-1,2-dithiol [39]:** Ethane-1,2-dithiol is generally synthesized on commercial scale by the reaction of dichloroethane and sodium bisulfide solution in water. In lab method synthesis involve reaction of thiourea and dibromo ethane and the product is hydrolysed to get the ethane-1,2-dithiol.

**Synthesis of Metal Complexes:** Richard Eisenberg et al. introduce three new platinum diiminedithiolate complexes, labeled I-III (Fig.6).

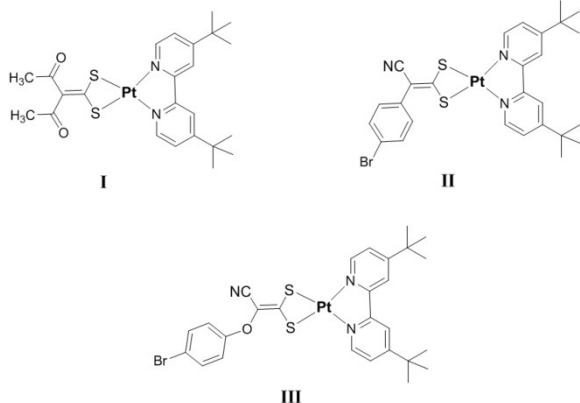


Fig. 6. Structure of Pt(diamine) complexes I, II & III

The ligands involved in designing of these complexes are 1,1-dithiolates created by reacting active methylene compounds and carbon disulfide in basic medium. The structure of these platinum complexes with diamine and thio ligand have been reported. The dithiolate ligand in complex-I has been studied with gold and thallium(I)[40]. In his work the synthesis of mono-, di-, and tetranuclear complexes of platinum(II), palladium(II), and silver(I) using the same ligand has also been reported[41]. The dithiolate groups in complexes I-III offer exciting possibilities for connecting to donor components in devices designed for photoinduced charge separation. Several new platinum (II) complexes with di-tert-butylbipyridine (dtbbpy) and 1,1-dithiolates have been created and studied (Fig.7). The 1,1-dithiolates include: 2,2-diacetylene-1,1-dithiolate, 2-cyano-2-p-bromophenylethylene-1,1-dithiolate, p-bromophenyl-2-cyano-3,3-dithiolatoacrylate[42-43].

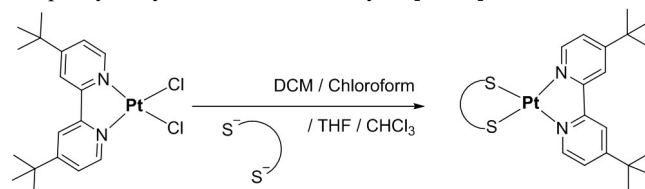


Fig. 7. Scheme for the preparation of Platinum (II) complexes

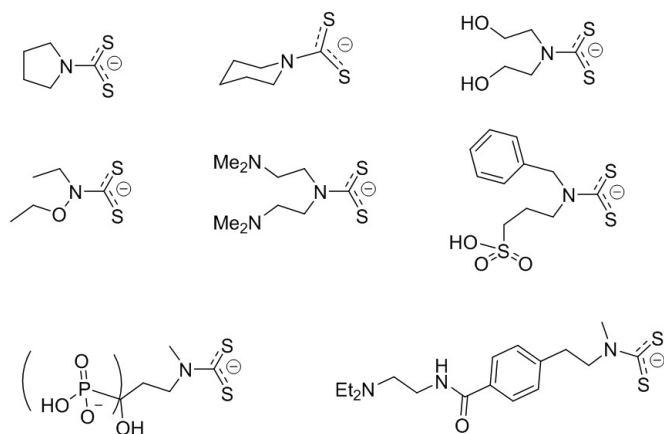
### Application of Metal Complexes of Dithio-Ligands

**Application as Superconductors Compounds:** The well-known ligand BEDT-TTF [bis-(ethylene dithio) tetra-thiofulvalene] forms complexes with thiocyanato ligands that exhibit a range of interesting properties [44]. For example, H. Urayama et al. reported [45] the synthesis of  $K-(BEDT-TTF)_2[Cu(NCS)_2]$ , while M. Oshima et al. and H. Mori et al [46]. synthesized and characterized complexes of the form  $(BEDT-TTF)_2X[M(NCS)_4]$ , where  $X = NH_4^+$ ,  $Tl^+$ , or an alkali metal, and  $M = Co(II)$ ,  $Cd(II)$ , or  $Hg(II)$ . Additionally, complexes such as  $(BEDT-TTF)_2[Cr(NCS)_4(NH_3)_2]$  have been prepared and reported to display metallic to superconducting behaviour. Superconductivity has also been observed in complexes with the general formula  $[M(dmit)_2]^n$ , where  $M = Ni$  or  $Pd$ , and  $0 \leq n \leq 2$ , with examples like  $[Ni(dmit)_2]$  [TTF] demonstrating superconducting properties [47-51].

**Application as semiconductor compounds:** A lot of research has been done on the semiconducting properties of dithio metal complexes, bi-metallic salts, and coordination polymer compounds, using both 1,1- and 1,2-dithio ligands. N. Singh [52] and colleagues have published extensive work on synthesis of various compounds such as carbamates, xanthates, dithiophosphonates, trithiocarbonates, and several heterocyclic derivatives of dithiolate compounds, including i-MNT<sup>2-</sup>, MNT<sup>2-</sup>, DED<sup>2-</sup>, and ecda<sup>2-</sup>. These complexes have been shown to exhibit semiconducting properties. Nitrogen and sulfur-containing ligands, such as 1,10-phenanthroline, ethylenediamine, and iso-thiocyanates, are commonly used to create mixed-ligand mono- and poly-metallic complexes with different coordination structures. One example includes heterometallic complex and complex salts derived from bis-(1-ethoxy carbonyl-1-cyanoethylene-2,2-dithiolate) malonate(II) ions, with general formulas like  $VO[M(ecda)_2]$  and  $[n-Pr_4N]_2[M(ecda)_2]$  where M can be Cu(II), Ni(II), Zn(II), or Cd(II). These complexes show paramagnetism, with quenching at room temperature suggesting strong antiferromagnetism. All of these compounds display semiconducting behaviour, with conductivity values ( $\sigma_{300}$ ) ranging from  $10^{-2}$  to  $10^{-11}$  S/cm. Li Qu et al [53]. described a straightforward Ni-bis(dithiolene) complex that serves as an outstanding n-type semiconductor molecular material for FETs (field-effect transistors). R. Ghosh et al. reported [54] the electrical conductivity in solid state as well as the designing and structural characterization of bis-(1,2-diphenyl ethylene-1,2-dithiolate) nickel(II). Similarly, C. Paulmann et al [55]. Studied metal complexes with dithiolate ligands such as 5,6-dihydro-1,4-dithiin-2,3-dithiolate (dddt<sup>2-</sup>) and dihydro-1,4,6-trithio-2,3-dithiolate. N. Robertson [56] and his team studied crystalline materials to explore their conducting and magnetic

behaviour, focusing on metal-based complexes with 1,2-dithiolene ligands. Many compounds have been created using dithiolene ligands, with their main structures extended by units like TTF or heterocyclic groups to understand how these changes affect conductivity and magnetic behaviour in solid form. E.A.M. Geary [57] and his group developed platinum dithiolate-based dyes for DSSC applications.

**Application in Bio-medicinal Fields:** Dithiolate metal complexes, also find extensive applications in biological fields. V. Milacic et al [58] reported that pyrrolidine dithiocarbamates of Zn(II) and Cu(II) they have potency to inhibit proteasomal functioning in tumor cells. Mansouri-Torshizi et al [59] studied anticancer activity of diiminePt(II) and Pd(II) dithiocarbamates. Dithiocarbamate-organotin complexes have also been explored for their biological activities. Sharon J. N. Burgmayer [60] highlighted the role of metal dithiolene systems in catalysis. M.K. Singh [61] and his team synthesized transition metal dithiolates, showing their luminescent and antifungal properties. Oliveira et al [62] investigated dithiocarbamates as potential drugs against trypanosomatid infections, emphasizing their applications in controlling Chagas disease, leishmaniasis, and African trypanosomiasis. Olsen and his group [63] demonstrated hydrogen activation using biomimetic diiron dithiolates. A. Das and his team explored [64] the reactivity and catalytic processes of these systems in biological contexts. The various dithio carbamates have multifarious properties in biological studies, the pyrrolidine derivative of carbamates are popular [65], while the piperidine derivative offers more flexibility for adjusting steric and electronic properties due to the availability of many substituted versions. The 2-hydroxyethyl derivative is commonly used to improve the water solubility of transition metal complexes, while the unsymmetrical is widely used in technetium radiopharmaceuticals [66]. Compounds with extra amine groups, are valuable for adjusting organic and aqueous solubility based on pH [67]. Sulfonyl-substituted derivatives, have been tested for their ability to induce cancer cell apoptosis [68]. Other innovative derivatives, such as and are useful in radiopharmaceutical applications [69].  $[\text{Au}(\text{BPYH})(\text{Cl})_2]\text{Cl}$ , and  $[\text{Au}(\text{BPYH})(\text{DMDTC})]\text{Cl}_2$ ; BPYH= Bis(pyridine) ligand, DMDTC = S-methyl-N,N-dimethyldithiocarbamate are reported as active compounds against prostate cancer [70]. The mechanism of action gold complexes are reported by Benoit Bertrand [71].

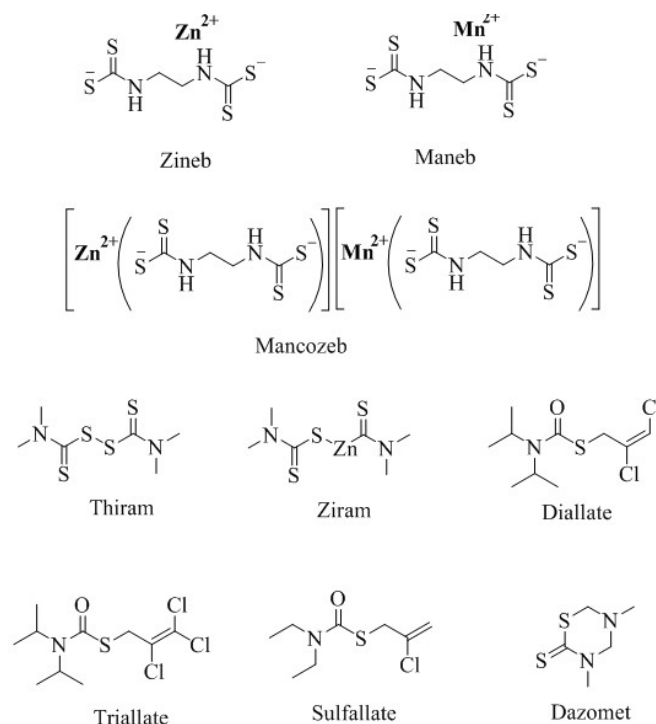


**Fig. 8. Structure of some dithio carbamates having important role in radiopharmaceuticals**

**Application in Agriculture:** Dithio carbamates are employed in combating diseases in plants and livestock. These compounds serve as pesticides, helping to prevent or eliminate plant diseases. They also aid in controlling unwanted plant growth. This section discusses several dithio carbamate compounds utilized for these purposes.

**Application as Herbicides:** Dithiocarbamate-based herbicides include compounds like propylenebis (dithiocarbamates), ethylenebis (dithiocarbamate) and dimethyldithiocarbamate. Some common examples includes Zineb, Thiram, Metiram, Disulfiram, Dazomet, Maneb and Propineb [72].

Among these few members also exhibit pesticidal behaviour. These herbicides [73] affect the growth of weeds, grasses (eg. Crabgrass, foxtail, bromegrass and cheatgrass etc) [74]. Dithiocarbamate based herbicides have also been successful in eliminating plants that produce oxidants [75] (active oxygen species). Modifying the hydrophilic and lipophilic behaviour of dithiocarbamates using some chemical substitutions like- ethyl, butyl, hexyl and ethoxy etc. This modification improves the herbicide's ability to penetrate plant cuticles, outperforming the use of regular sodium diethyldithiocarbamate. Other well-known dithiocarbamate-based herbicides include Diallate, Sulfallate, Dazomet, and Triallate. Diallate [S-(2,3-dichloroallyl)-diisopropylthiocarbamate] is very active to control weeds of monocot category [76]. (Fig.9).



**Fig. 9. Some dithiolate based herbicides**

**Application as pesticides:** Dithiocarbamate based pesticides fungicides activity for different crops in their shipping, cultivation and storage [77]. In Fig-9 the structural details are summarised. Triphenyltin N-n-butylthiocarbamate and tricyclohexyltin dithiocarbamate have been utilized as larvicides against the larvae of *Anopheles stephensi* mosquitoes and *Aedes aegypti*, [78] proving effective in controlling these mosquito species. Additionally, dithiocarbamates derived from chitin oligosaccharides have been successful in eradicating *Meloidogyne incognita*, a nematode-related disease [79]. This derived pesticide demonstrates strong activity against the nematodes and also inhibits egg hatching, which helps reduce the nematode population.

**Application in dye-sensitized solar cell:** Among various dye-sensitized solar cell sensitizers, metal based complex compounds of (dmit) 1,3-dithiole-2-thione-4,5-dithiolate ligands represent a very special category of functional materials with remarkable physical as well as chemical features [80]. In this study, the nickel complexes with this ligand have been reported with multiple electrical and molecular characteristics. The study of TD-DFT (time dependent density functional theory) based calculations reveals that the lowest energy transitions in the ultraviolet-visible-near-infrared (UV-Vis-NIR) region primarily involve the  $\pi\pi^*$  excitation between the HOMO to LUMO level. When adsorbed on the surface of  $\text{TiO}_2$ , [81] the absorption spectrum of the nickel complex experiences a minor shift towards red. In recent years, many dye sensitizers, including a variety of transition metal complexes, have been designed and tested [82-85]. Among these, Ru(II)-based sensitizers on  $\text{TiO}_2$  semiconductors are the most efficient [86-88]. However, as noted by Linfoot et al., their use has drawbacks, such as limited metal availability and potential

toxicity [89]. To address these concerns, researchers have explored complexes with more abundant and less toxic metals, such as platinum. A significant focus has been on enhancing the light-absorbing feature of Platinum based complexes. For example, Du et al and Singh et al. developed variety of Platinum based complexes having square planar spatial orientation geometry with diphosphine/diamine and dithiolate ligands, including 1-ethoxycarbonyl-1-cyanoethylene-2,2-dithiolate ( $\text{ecda}^{2-}$ ) and dithiocarbamate<sup>2-</sup>. These studies show that these complexes possess promising photophysical and electrochemical properties [90-91].

**Application as Catalyst:** Phosphane ligand complex are reported to play a vital role in several catalysis reactions. The chemistry of thiol ligands has been widely studied, showing a variety of structures with different transition metals in various oxidation states. Although the sulfur based ligand and their complexes are less explored in catalysis reactions. The metal-catalysed reactions that involve S-donor auxiliary ligands. Therefore, reactions using sulfur-containing substrates, even though they may form intermediates with M-S bonds, are not included. For instance, HDS and reactions involving thioethers, thiols, sulfoxides, disulfides, or  $\text{H}_2\text{S}$  as reagents are not discussed. Metalloenzymes with sulfur ligands are also excluded. The work on hydrogenation, hydrosilylation, hydrogen transfer, hydroformylation, isomerization, carbonylation, immobilized, catalyst reaction, polymerization, etc. have been reported.

## CONCLUSION

The present paper throw light on the chemistry, structural details, synthetic routes and wide spread application of thiolate metal based complexes. The variety of ligation behaviour of thiolate ligands insist to coordination with various metals in different mode like- bridging, non-bridging, mono-dentate, bi-dentate etc. 1,1-dithio, 1,2-dithio, 1,3-dithio ligand show variety of bonding behaviour resulting different type of coordination complexes, coordination polymers and complex salts. The synthetic methods for these ligands and their metal complexes have also been reviewed. The thiolate metal complexes have multifarious applications in material science and biological fields including semiconductors, industrial applications, as superconductor, as sensor, in catalysis, in dye sensitized solar technology, as pesticides, removal of toxic metals from biological systems, as anticancer agents etc. The versatile application of these complexes attract the researchers to investigate the chemistry of these complexes and correlate the structure and activity of these compounds in different fields. There are several application for use of these metal complexes. The deep and sincere research will open new dimensions in these fields like- novel semiconductor raw advanced molecular materials for variety of electronic applications, better chromophoric compounds for DSSC, good catalysts, better sensor compounds, potential anticancer, antimicrobials and pesticides etc.

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