SYNTHESIS AND CHARACTERISATION OF TRANSITION METAL COMPLEXES OF SCHIFF BASES DERIVED FROM DITHIOCARBAZATE AND APPLICATION OF THESE COMPLEXES AS CYCLOHEXANE OXIDATION CATALYSTS

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ALI AHMED QAID ALSHAHERI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

June 2019

DEDICATIONS

To my beloved father. It is your wish, thus I insisted to achieve it. To my beloved mother, who endured my absent. Her prayers for me have not stopped. To all my family members for their unconditional love and support.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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Schiff bases and their complexes are excellent applicants' compounds that are synthesized by the condensation of a primary amino compound with either ketones or aldehydes for a variety of industrial applications. They can act as catalysts for the catalytic oxidation of organic compounds. In recent years, most of the researches in oxidation catalysis focused on how to employ the metal complexes-catalyzed oxidation of organic compounds. Schiff base complexes of transition metals can be used for catalytic oxidation of hydrocarbons in the presence of tert‑butyl hydroperoxide (TBHP) and hydrogen peroxide. The oxidation of cyclohexane is an important oxidation reaction for industrial applications as their products, cyclohexanone and cyclohexanol, also known as K-A oil, are very important to produce nylon 6,6 and nylon-6 polymers, plastics, lubricant additives, fibers and are intermediate for pharmaceuticals. In this work, dithiocarbazate Schiff bases derived from the condensation of S-methyldithiocabazate (SMDTC) and S-benzyldithiocarbazate (SBDTC) with 2,6-diacetylpyridine (diAP) and 1,3-diacetylbenzene (diAC), and their Ni(II), Cu(II), Fe(II), Co(II), Mn(II) and Zn(II) complexes have been successfully synthesized in ethanol and characterized using several physico-chemical techniques. The oxidation of cyclohexane by the prepared metal complexes as catalysts has been investigated in the presence of tert‑butylhydroperoxide (TBHP) and hydrogen peroxide as the oxidative source under mild conditions at 4h, 70°C, 0.09 mmol catalyst and 20 mmol oxidant. The product was analyzed using gas chromatography and it was found that cyclohexanol and cyclohexanone are the main products for the oxidation reaction. All the transition metal complexes of the Schiff bases showed activity toward oxidation of cyclohexane. Time of the reaction, temperature, and the concentration of TBHP and catalyst influenced the selectivity and conversion of the cyclohexane oxidation. The complex, [CuSBdiAC], was found to be the most active catalyst with ~50% conversion.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

SINTESIS DAN PENCIRIAN BAGI KOMPLEKS LOGAM PERALIHAN BES SCHIFF YANG TERDIRI DARIPADA DITIOKARBAZAT DAN APLIKASI BAGI KOMPLEKS INI SEBAGAI PEMANGKIN PENGOKSIDAAN SIKLOHEKSANA

Oleh

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Bes Schiff dan kompleksnya adalah sebatian yang sangat baik yang disintesis oleh kondensasi sebatian utama amino dengan keton atau aldehid untuk pelbagai aplikasi perindustrian. Kompleks logam peralihan bes Schiff telah memainkan peranan penting dalam kimia koordinatan. Kaedah sintesis dan kestabilan terma yang sesuai bagi kompleks bes Schiff telah banyak menyumbang untuk aplikasi mereka yang mungkin dalam pemangkinan, biologi, fotonik dan perubatan. Kompleks bes Schiff boleh memainkan peranan sebagai mangkin untuk pengoksidaan bermangkin sebatian organik. Dalam tahun-tahun kebelakangan ini, sebahagian besar penyelidikan dalam pemangkinan pengoksidaan memberi tumpuan kepada cara menggunakan mangin kompleks logam dalam pengoksidaan sebatian organik. Kompleks logam bes Schiff boleh digunakan untuk pengoksidaan bermangkin hidrokarbon dengan kehadiran tert-butil hidroperoksida (TBHP) dan hidrogen peroksida. Pengoksidaan sikloheksana adalah tindak balas pengoksidaan penting bagi aplikasi perindustrian. Sikloheksanon dan sikloheksanol, yang juga dikenali sebagai minyak KA, adalah produk yang sangat penting untuk penghasilan nilon 6,6 dan nilon-6 polimer, plastik, bahan tambahan pelincir dan gentian serta industri racun serangga dan perantaraan untuk farmaseutikal menggunakan pengoksidaan sikloheksana oleh kompleks logam peralihan sebagai mangkin dengan penukaran dan pemilihan yang sangat rendah. Hasil daripada kestabilan ikatan C-H menyebabkan tenaga pengaktifan yang lebih tinggi diperlukan untuk melaksanakan tindak balas ini pada suhu dan tekanan yang lebih tinggi. Di dalam projek in, bes Schiff ditiokarbazat terbitan dari kondesasi S-metilditiokarbazat (SMDTC) dan S-benzilditiokarbazat (SBDTC) dengan asetofenon, 2-asetilpiridina, 2,6-diasetilpiridina dan 1,3-diasetilbenzena bersama dengan kompleks Cu(II), Fe(II), Co(II), Mn(II) dan Zn(II) mereka telah berjaya disintesis dalam etanol dan dicirikan menggunakan beberapa teknik fizik-kimia. Pengoksidaan sikloheksana oleh kompleks logam yang disediakan sebagai mangkin telah dijalankan dengan kehadiran tert-

butilhidroperoksida (TBHP) dan hidrogen peroksida sebagai sumber oksidaan di bawah keadaan sederhana pada 4h, 70ºC, 0.09 mmol mangkin dan 20 mmol pengoksida. Produk ini dianalisis dengan menggunakan kromatografi gas dan didapati bahawa sikloheksanol dan sikloheksanon adalah produk utama untuk tindak balas pengoksidaan tersebut. Semua kompleks logam peralihan bes Schiff mempunyai aktiviti terhadap pengoksidaan sikloheksana, kompleks bes Schiff yang diperoleh daripada 2,6-diasetilpiridina dan 1,3-disetilbenzena menunjukkan aktiviti tertinggi semasa kajian saringan terhadap pengoksidaan sikloheksana. Hasil kajian menunjukkan bahawa masa tindak balas, suhu, dan kepekatan TBHP dan pemangkin mempengaruhi selektiviti dan penukaran pengoksidaan sikloheksana.

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LIST OF ABBREVIATIONS

BINAP	Binaphthyl Schiff base complexes (Organophosphorus compound 2,2'- bis(diphenylphosphino)-1,1'-binaphthyl)
CHNS	Carbon, hydrogen, nitrogen and sulfur
Cy.	Cyclohexyl radical
CyO•	cyclooxyl radical
CyOO•	cycloperoxyl radical
DTC	Dithiocarbazate
KA	Ketone and alcohol
ROO•	cyclohexyl peroxyl radical
ROOH	Cyclohexyl hydroperoxide
SBDAC	Benzyl 2-(1-phenylethylidene)hydrazinecarbodithioate
SBDAP	Benzyl 2-(1-(pyridin-2-yl)ethylidene)hydrazinecarbodithioate
SBdiAC	1,3-diacetylbenzylbis(S-benzyldithiocarbazate)
SBdiAP	2,6-diacetylpyridinebis(S-benzyldithiocarbazate
SMDAC	Methyl 2-(1-phenylethylidene) hydrazinecarbodithioate
SMDAP	Methyl 2-(1-(pyridin-2-yl)ethylidene)hydrazinecarbodithioate
SMdiAC	1,3-diacetylmethylbis(S-methyldithiocarbazate)
SMdiAP	2,6-diacetylpyridinebis(S-methyldithiocarbazate)
TBHP	Tert-butyl hydroperoxide
TMSCN	Trimethylsilycynide
FT-IR	Fourier transformed infrared
NMR	Nuclear magnetic resonance
Cy-OH	Cyclohexanol
Cy=O	Cyclohexanone
TMS	Tetramethylsilane

UV-VIS	Ultraviolet-Visible
DMSO	Dimethyl sulfoxide
MSB	Magnetic susceptibility balance
GC	Gas chromatography
IS	Internal standard
TGA	Thermogravimetric analysis
LMCT	Ligand metal charge transfer
INT	Internal charge transfer

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Schiff bases contain the azomethine (imine) group with a general formula (RHC=N-R'), where R and R' are alkyl, aryl, cycloalkyl or heterocyclic. Scheme 1.1 shows the general formation of Schiff bases by the primary amine condensation with an active carbonyl compound (aldehyde or ketone) in different solvents, and in different reaction conditions with the elimination of water molecules.

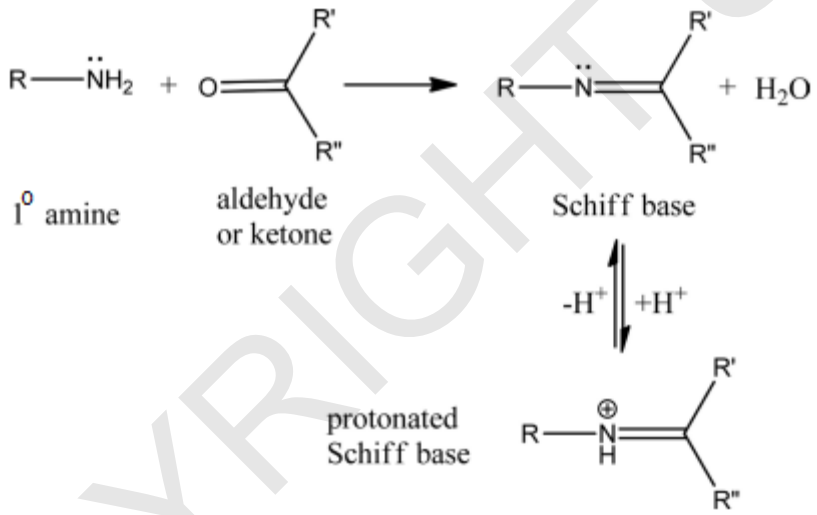

Figure 1.1 : General Formation of Schiff bases (Abu-Dief, A. M., & Mohamed, I. M. A., 2015)

The methanol and ethanol are the most used solvents for the Schiff base preparation. The formation of Schiff base occurs either in refluxing or at room temperature conditions (Bhagat, et al., 2013).

In recent years, the easy preparation of Schiff bases and their production of complexes with almost all metal ions which can be stabilized them in different oxidation states (Kumar, et al., 2010). Schiff bases play a significant role in the coordination chemistry development. Basically, this study concerned with metal complexes chemistry as their physical and chemical properties give rise to a wide range of applications in many scientific areas. Schiff bases can stabilize a lot of various metals in different oxidation state. Schiff bases influence the performance of metals in many different useful catalytic transformations.

Several Schiff base complexes have shown excellent catalytic activity in different reactions at high temperature (>100 ºC) and in the presence of moisture. In the last few years, many applications of these complexes have been carried out in homogeneous and heterogeneous catalytic reactions. Schiff base complexes of transition metal complexes are effective catalysts in both heterogeneous and homogeneous reactions, such as oxidation reaction (Abu-Dief, A. M., & Mohamed, I. M. A., 2015), reduction of ketone to alcohol (Tingting, et al., 2013; Himeda, et al., 2003), alkylation of allylic substrates (Brunel, et al., 1998; Brunner, et al., 2003), carbonylation of alcohols and alkenes at low pressure to produce α-arylpropionic acid and their esters (Greb, et al., 2004; Jedlicka, et al., 1998; Paul, 2000; Wan, et al., 1998), Heck reaction (Beletskaya & Cheprakov, 2000; Bhanage, et al., 2003; Iyer, et al., 2004) etc. The activity of metal complexes differs according to the metal ions, type of ligands and coordination sites. Selective oxidation is the most important functionalization saturated hydrocarbons to ketones and alcohol especially the selective oxidation of cyclohexane because of its important in the industry for example; adipic acid, nylon-6 and nylon-6,6 (Sun, et al., 2016; Wang, et al., 2015). Among the catalytic process, the organic oxidation material is strongly exothermic. Therefore, when the free reaction enthalpy ΔH° is more negative, the activation energy separation will be smaller for producing of the desired product versus the production of other products. Generally, the activation energy, $E_{a,}$ to produce a desired hydrocarbon oxidation product is higher than that for the following products. Therefore, the selective oxidation of hydrocarbons to suitable oxygenates pose a great challenge.

There are a lot of hydrocarbons in the constituents of the natural gas and oil, thus, the usage of the hydrocarbons in reactions was the way for several economic industries. Selective oxidation is the most significant functionalization of hydrocarbons. In the liquid phase, different oxidants such as; hydrogen peroxide, tert-butyl hydroperoxide (TBHP) and others have been used for oxidation of cycloaliphatic and aliphatic compounds to produce suitable alcohol and ketones under tough conditions like high temperature and pressure due to the difficulty activate the C-H bonds (Crabtree, 1995; Shul ’pin, 2016).

Annually, more than one billion tons of cyclohexanone and cyclohexanol are formed widely around the world, for the industrialization of the nylon-6 and nylon-6,6, that have been used in the manufacture of adipic acid, polyamide-6, acidulant in baking powder and caprolactam (Antony, et al., 2014; Schuchardt, et al., 2001). In the last few decades, the oxidation of cyclohexane using Schiff base metal complexes has attracted scientists to conduct research in this area. Schuchardt et al. discussed an alternate method on how to develop the industrial method of the oxidation of cyclohexane that focused on the improvement of new catalysts to oxidize cyclohexane under a suitable condition (Schuchardt, et al., 1993). The use of metal complexes as catalysts to activate hydrocarbons was improved by applying Schiff base transition metal complexes as catalysts because of their cheap and easy synthesis and their chemical and thermal stability (Chatterjee, et al., 2004; Wang, et al., 1999). These studies are indicative of Schiff base complexes as potential catalysts to influence the selectivity and yield in oxidation of cyclohexane. The use of Schiff base metal

complexes in the cyclohexane oxidation has a great interest due to their potential catalytic activity (Murahashi, et al., 2004; Parrilha, et al., 2010).

1.2 Problem Statements

The catalytic oxidation and selectivity of the hydrocarbons under mild conditions has an importance in the academic and industrial field. (Wang, et al., 2015). Oxidation reaction of organic compounds is one of the most important methods for substrate functionalization and function group transformation. Oxidation of cyclohexane is an important commercial reaction for the formation of cyclohexanone and cyclohexanol, more than one billion tons of these products are formed every year all over the world. These products have an importance in the nylone-6 and nylon-6-6 polymers manufacture. In addition, cyclohexanone and cyclohexanol are also used as solvents for varnishes and lacquers as well as homogenizers and stabilizers for soaps and synthetic detergent emulsions. Another cyclohexanone uses are in the herbicides synthesis, pharmaceuticals and insecticides. (Zhang, et al., 2014).

Today, field of catalytic oxidation is one of the most popular fields particularly in the current research and the catalytic oxidation is one of the most important to produce useful chemical compounds, but also one of the most problematic processes in chemical industry.

Most of the oxidation reactions use heavy metal-based catalysts that are expensive and produce hazardous, toxic waste, which impact on the environment, whereas Schiff base transition metal complexes are gaining more and more importance as alternative catalysts in organic compound oxidation reactions because most of them have high thermal and moisture stabilities (Parrilha, et al., 2010; Yang, et al., 2008).

cyclohexane oxidation requires severe reaction conditions such as high temperature and high pressure. These extreme conditions may lead to yield carbon dioxide and water. The best catalyst should be selective, efficient catalyst for the first step of the oxidation, not continue further with the following reactions, and stopping at the desired products. Hence it is necessary to find a new, efficient catalyst for the oxidation of cyclohexane at low temperature. The high selectivity to the products of cyclohexane oxidation is obtained only at low cyclohexane conversion due to cyclohexanol and cyclohexanone more reactive than the cyclohexane. Furthermore, the oxidation reaction conditions need high temperature and pressure ((>100 ^{o}C and > 0.9 Map) to produce cyclohexanol and cyclohexanone from oxidation reaction of cyclohexane (Zhou, et al., 2005). In industrial process the cyclohexane is first oxidized to cyclohexanoe and cyclohexanol using cobalt based homogeneous catalyst conversion and 70-80 selectivity. The other major problem for the oxidation reactions is the production of dangerous by-products and undesired compounds due to the use of certain oxidants; nitric acid is the most exploited oxidant in industry because of its low-cost. Whereas, it is responsible for emissions of nitrogen oxide; the nitric acid used as an oxidant to produce the next oxidation process of cyclohexane oxidation

which is a very unfriendly reagent (Du, et al., 2009). Mostly, a green catalyst and process of catalytic need: (a) the catalyst should be as selective as possible; (b) the rate of material converted into product is as high as possible; (c) the materials and solvents are friendly; (d) the cost of oxidation or reduction agent is as low as possible.

This work focused on the application of catalytic complexes, namely dithiocarbazate Schiff bases complexes as catalysts, in cyclohexane oxidation, which have never been tested in oxidation reactions yet.

In this study, we have carried out the oxidation of cyclohexane using the oxidant hydrogen peroxide and *tert*-butylhydroperoxide. The catalysts used are iron(II), copper(II), nickel(II), cobalt(II), manganese(II) and zinc(II) complexes derived from the Schiff bases. There are many advantages for these complexes like, low cost, simple synthesis method and the catalysts give good yield and high products selectivity within a short time under mild conditions.

1.3 Scope of the study

Schiff bases are condensation products of amines and a ketone or aldehydes. The greatest advantage of Schiff bases is that they can be easily synthesized from relatively inexpensive materials. Schiff bases containing the hetero atoms like nitrogen, sulfur, and oxygen are of much importance and the metal complexes play a major role in homogenous and heterogeneous catalysis in deferent solvents. Numerous works have been reported in the study of Schiff bases from chemical and biological viewpoints. Schiff bases have an important role in biochemical reactions also as they form intermediate in the formation of medicinal compounds. Metal complexes formed with Schiff bases have found utility in many fields, like pharmaceuticals, dye stuff preparations and in corrosion and inhibition studies. Numerous works have been done to study and investigate dithiocarbazate ligands, their Schiff bases, and metal complexes. The main reason that initially stirred up researchers' interest to further pursue this field is that these compounds were found to possess antifungal and antibacterial properties. In spite of all the work done in this field, no application for the dithiocarbazate Schiff base complexes in catalysis reactions especially in oxidation reaction. According to the literature reviews, there are few reports on Schiff base complexes derives from dithiocarbazate of SBDTC and SMDTC. Hence, we have studied the dithiocarbazate Schiff base metal complexes as catalysts for cyclohexane oxidation in mild conditions in details. The general goal of this work is to develop oxidation catalyst that is high selective for oxidation of cyclohexane, with good conversion, stable, inexpensive and environmentally friendly. In particular, it was the aim of the present research to synthesize dithiocarbazate Schiff bases derived from the condensation of S-methyldithiocabazate (SMDTC) and S-benzyldithiocarbazate (SBDTC) with acetophenone, 2-acetylpyridine, 2,6-diacetylpyrdine and 1,3-diacetylbenzene, and their Ni(II), Cu(II), Fe(II), Co(II), Mn(II) and Zn(II) complexes and characterized using several techniques which are; Fourier transform infrared (FT-IR) spectroscopy, nuclear magnetic resonance (^{1}H-NMR, ^{13}C-NMR), magnetic susceptibility measurements, molar conductivity, ultraviolet visible (UV)

spectroscopy, and inductively coupled plasma spectrometry (ICP-OES), to test the prepared metal complexes as catalysts in the presence of oxidant as the oxidative source under mild conditions for cyclohexane oxidation to cyclohexanone and cyclohexanol, to analyze the reaction products using gas chromatography (GC).

1.4 Objectives

The main objectives of this study:

1. To synthesize and characterize dithiocarbazates their Schiff bases and their transition metal complexes.
2. To study the catalytic activity of the synthesized transition metal complexes of the Schiff bases in oxidation reactions of cyclohexane.
3. To optimize the parameters of the cyclohexane oxidation reaction to achieve higher conversion and selectivity.
4. The selected ligands for this study are:
1. Acetophenone Schiff base of S-benzyldithiocarbazate and S-methyldithiocarbazate (SBDAC and SMDAC).
2. Acetylpyridine Schiff base of S-benzyldithiocarbazate and S-methyldithiocarbazate (SBDAP and SMDAP).
3. 2,6-diacetylpyridine Schiff base of S-benzyldithiocarbazate and S-methyldithiocarbazate (SBdiAP and SMdiAP).
4. 1,3-diacetylbenzene Schiff base of S-benzyldithiocarbazate and S-methyldithiocarbazate (SBdiAC and SMdiAC).

In this regard, multifunctional hetero ligands comprising different donor atoms may serve as a suitable candidate for metal chelation in order to enhance the catalytic behavior of metal complexes.

Cobalt(II), copper(II), iron(II), manganese(II), nickel(II), and zinc(II) complexes of these dithiocarbazate Schiff bases were synthesized and characterized. The synthesis of dithiocarbazate complexes is successfully carried out using Schiff bases and metal ions. The catalytic activity of synthesized dithiocarbazate Schiff base metal complexes have been screened in the cyclohexane oxidation. The oxidants are not expensive. Furthermore, the reaction is carried out in environmentally desirable solvents such as acetonitrile. TBHP and HP are clean oxidants compared to other oxidants (Mardani & Golchoubian, 2006).

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