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Synthesis and Characterization of Silica Supported Nickel Catalysts

تخليق وتوصيف حفازات النيكل المدعمة بالسلكا

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إستهـــلال

قال تعا لى :

بِسۡمِ ٱللَّهِ ٱلرَّحۡمَٰنِ ٱلرَّحِيمِ

أقرأ بأسمِ رَبكَ الَّذِى خَلَقَ ﴿1﴾ خَلَقَ الإنسان من عَلقِ ﴿2﴾ إقرَأ ورَبّك الأكرم
﴿3﴾ الذّي عَلّم بالقلم ﴿4﴾ علم الإِنسانَ مالم يعلم ﴿5﴾

الايا ت 1 -5)((صدق الله العظيم))   سورة العلق

Dedication

I dedicate this work to

To my The Souls of my parents

Wife,

Brothers,

Sisters

sSon

Daughter

and

Friends

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Praise to Allah Almighty for giving me the health to accomplish this work My appreciation is due to my supervisor professor Dr Mohammed Elmubarak Osman for persistent encouragements, continuous support, advice and indispensable, help throughout this work I am especially indebted to my Co supervisor Dr Mohammed Suleiman Ali for his encouragement, suggestion and guidance has always been appreciated. Especial thanks of department of chemistry Sudan University of Science and Technology. I thank to Regional forensic Laboratories Directorate. To help me and analysis the samples

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Abstract

A series of $Ni\backslash SiO_2$ catalysts were synthesized by heating different Concentration of $NiCl_2$ impregnation SiO_2 (at load 1 %, 3%, 5 % 8 % and 10%, respectively) at 110, 300, and 500 C The nature of surface species upon drying, and thermal treatment were characterized using different microscopic techniques such as XRD, EDX-SEM spectroscopy .The obtained result showed that there is a significant difference in the microstructure of the catalyst in different Concentration applied for pure silica and $NiCl_2$ calcinated at 300 C 500 C, showed that the characteristic peak of silica is shifted to different Θ position from 15 °, 22 ° ,55°, to Θ equal 28 °, 35 °, 38 ° ,44 ° 55 ° ,65 ° 75 ° and 80°. While in concentration of silica supported nickel at 1%, 3%, 5%, 8% and 10% nickel chloride impregnated silica at 300 C and 500 C the back scattering peak, shifted from the concentration increased and anew peak was appeared. for the nickel chloride supported silica at 8% and calcinations at 300 C and 500 C showed the same shifted pattern from Θ equal 25 ° 30 ° 35 °, 45 ° 55 °, 65 .° at 500 C and equal 75 ° at 300C to 28 ° 30 ° 33 °, 38 ° 40 °, 42 °,46 °,50 . °,65 ° 75 °,and 80.° Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) of pure silica calcinated at 300 C and 500 C showed the map like flower shape is white and brown color in concentration of

silica supported nickel at 1%, 3%, 5%, 8% and 10% nickel chloride impregnated silica at 300 C and 500 C the new phase was appeared and dominated as the concentration increased for increase concentration level. And the shape of maps changes to be black, brown, red and green. The results indicate that following (the drying step and calcinated 300 C and 500 C). The Nickel occurs in many detectable forms. $NiCl_2$ particle and Ni – O surfaces. Calcinations appear to transform the $NiCl_2$ (Ni (OH) s CL) (NiOCL) and NiO surface species.

المستخلص

يتناول هذا البحث دراسة سلسلة من حفازات النيكل احادية الفلز والمدعمة على اكسيد السيلكون والمحضرة بطريقة التشرب المتزامن عند معالجات حرارية عند 110م^{0} و300م^{0} وكذلك 500م^{0} وقد تم توظيف طريقة من طرق الاستقصاء الفيزوكيميائي (حيود الاشعة السينية) للتعرف على طبيعة هذه الحفازات استخدمت تراكيز مختلفة من كلوريد النيكل المدعمة بالسلكا وكلسنته عند درجة حرارة 300 م و 500م عند تحليل سلكا النقية بواسطة الاشعة السينية يظهر سيتا في الاتي 15 ،22 ،55 ، 58، ويظهرفي 28 ، 35 ،38 ،44. 55، 65 ، 75 .و 80 اخذت 1% 3% 5% 8% و10% من كلوريد النيكل المدعمة بالسلكا وتم تحليلها بالاشعة السينية كانت نتائجها مختلفة يظهر سيتا عند 37 و نجد إختلاف في قيم سيتا عند300م سيتا يساوي 25، 30 ، 35، 45 ، 55 ،65 و 75 أما عند حرارة 500 م . سيتا يظهر عند 28، 30 ،33 ، 38 ،40، 42، 46، 50، 65,75 و80 . أما عند استخدام جهاز مسح المجهر الالكتروني والاشعة السينية .استخدمت سلكا النقي عند درجة حرارة 300 و500 يظهراشكال يشبه زهور لها الوان مختلفة ابيض ، بني ، اخضر، احمر . تم اخذ تراكيز مختلفة من كلوريد النيكل المدعمة بالسلكا وكلسنته عند درجة حرارة 300 و 500 اخذت 1% 3% 5% 8% و10% من كلوريد النيكل المدعمة بالسلكا وتم تحليلها بجهاز مطيافية تشتت الطاقة بالاشعة السينية وكانت نتائجها مختلفة . نجد ان مستوى التحميل يزيد بزيادة التراكيز استخدمت السلكا النقية عند درجة حرارة 300 و500 وتكوين متراكبات أيونية مختلفة من كلوريد النيكل ومع إجراء الكلسنة يتحول كلوريد النيكل الي كل من هيدروكسي كلوريد النيكل أكسي كلوريد النيكل وفي النهاية يكون اكسيد النيكل على السطح.

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Chapter one

I Introduction and literature Review

1.1 General introduction

Transition metals and metal oxides supported on oxides matrices are among the most widely used materials in heterogeneous catalysis, *at* laboratory and industrial levels. The technique of loading a catalyst active phase on inert support is a means of obtaining the catalyst in a highly active form. The technique facilitates the dispersion of the active phase on the support and, thus, increasing the accessible surface area catalyst finds application in various fields such as fuel cells, oil refining chemical processing exhaust system, etc. The fundamental concept of heterogeneous catalysis is that the catalytic substance has a prominent surface catalytic activity which is termed as the active site of the catalyst. Typically, most industrial catalysts are of nanometer- scale metal particles with many active Centers supported on inert porous metal oxide substance (Satterfield ,1980). Metal oxide such as silica alumina and magnesia are the most widely used supports .The main purpose of the support is to disperse and stabilize the active centers; the support itself is usually catalytically in active .These supported catalysts are often referred to as high surface area catalysts. A Catalyst cannot change the ultimate equilibrium determined by thermodynamics but it can change the kinetics of the chemical reaction Preparation methods, the composition of active metals and the conditions employed given the behavior of the catalysts which is described in Global terms of activity, selectivity and deactivation. Activity is expressed in units of amount of product made in the reactor per unit time and per unit of reactor volume (Bull Korean 2014).

The selectivity is a measure of the extent of which it accelerates the reaction to form one or more of desired products that are usually intermediates instead of those formed by reaction at lowest free energy (M .Boudart, et al.,1997) .Activity and selectivity usually varies with pressure, temperature, reactant composition, extent of conversion, as well as, with the nature of the catalyst .On the other hand, a catalyst many deactivate for a wide variety of factors such as poisoning fowling and sintering (Tohji,Kazuyuk 2020).

The best performance of a heterogeneous catalyst is achieved by varying the preparation procedures, the operating conditions and the catalysts constituents. The catalyst constituents vary from monometallic to multi metallic system and typically contain promoters, poisons, etc. Bimetallic supported catalysts have attracted major attention because of their enhanced catalytic activity and selectivity compared to monometallic supported catalysts, catalyst. Substance that can cause a change in the rate of chemical reaction without itself .Being consumed in the reaction, the changing of reaction rate by use of catalyst is called catalysis. Substances that increase the rate of reaction are called positive catalysts or, simply, catalysts, while substances that decrease the rate of reaction are called negative catalysts or inhibitors (Bron, Gates1992).Heterogeneous catalysis is a chemistry term which describes catalysis where the catalyst is in a different phase (i, e. solid, liquid and gas, but also oil and water) to the reactor. Heterogeneous catalysts provide a surface the chemical reaction to take place on .In order for the reaction to occur, one or more of the reactants must diffuse to the catalyst surface and adsorb to it, after reaction, the products must adsorb from the solid surface .Frequently, this transport of reactants and products from one phase to another plays a dominant role in limiting the reaction rate, understanding these transport phenomena and surface chemistry such as dispersion is an important area of heterogeneous catalyst research. Catalyst surface area may also be considered .Malodorous silicates, for example , have found utility as catalysts because their surface areas may be in excess of 1000m2/g, which increases the probability that a reactant molecule in solution will come in contact with the catalyst surface and adsorb. If diffusion rates are not taken into account, the reaction rates for various reactions on surfaces depend solely on the rate constants and reaction concentrations. A symmetric heterogeneous catalyst can be used to synthesize enantiomerically pure compounds using chital heterogeneous catalysis. The field is of great industrial and environmental importance, it has attracted two Nobel prizes for living Longmuir in 1932 and Gerhard Erik in 2007). Most of the chemical reactions in industry and biology are catalytic, and many chemists and chemical, engineers work to understand and apply catalysis. Catalysis is involved at some stage of the processing of a large fraction of good manufactured in the United States. The value of these product s approaches a trillion dollars annually, more than gross national products of all but a few countries in the world. Catalysis is the key to the efficiency of chemical conversations. An age of increasing limited energy and raw materials and concern for the in environments, its needed more and more. The technological needs are matched by the scientific opportunities; new techniques are bringing rapid progress in the understanding of molecular details of the working of catalysts (Bond C Serious 1987).Catalysts work by changing the activation energy for a reaction, i.e., the minimum energy needed for the reaction to occur. This is accomplished by providing a new mechanism or reaction path through which the reaction can proceed. When the new reaction path has lower activation energy, the reaction rate is increased and the reaction is said to be catalyzed. If the activation energy for the new path is higher, the reaction rate is decreases and the reaction is said to be inhibited .Inhibitors provide an interesting challenge to the chemist. For example, because oxygen is an inhibitor of free-radical reactions, many of which are important in the synthesis of polymers, such reaction must be performed in an oxygen- free environment, e.g., under a blanket of nitrogen gas. In some reactions one of the reactions products is a catalyst for the reaction; this phenomenon is called self- catalysis or auto catalysis. An example is the reaction of permanganate with oxalic acid to from carbon dioxide and manganese Ion, in which the manganese Ion, acts as an auto catalyst. Such reactions are potentially dangerous, since rate may increase to the point of explosion. Some substances that are not themselves catalysts, increase the activity of a catalyst when added to some reaction; such substances are called promoters Alumina, the reaction of hydrogen and nitrogen to form ammonia Substances that react with catalysts to reduce or eliminate their effect are called poisons in usual case, the catalyst is in crystallineform(Max,Greger2015).

1 .1 Types of Catalysis

There are two types of catalysis Homogenous catalysts and Heterogeneous Catalysts

1 .1 .1 Homogenous Catalyst

When the catalyst and the reactant are in the same phase, the process is known as homogenous catalysis. May consist entirely of gases, or both the catalyst and reaction are in solution phase. For instance, nitric oxide gas catalysis the combination of moist sulphure dioxide and oxygen in the lead chamber process for the manufacture of sulphuric acid .Homogenous catalysis in solutions is more common. The most importance class of such reaction is those catalyzed by the presence of acids and base, i.e. acid –base catalysis. However parts from this class friedlscraft reaction provide and other example of homogenous catalysis in solutions, the whole reaction is supported to occur in the presence of $Al\ Cl_3$ catalyst. Some other Lewis acid catalysts are also involved in this reaction i.e. $AlBr_3$, BF_3 the acidic strength of the most common Lewis (Marrch, 1992Acids decreases in the following order $BX > AlX_3 > FeX_3 > Ga\ X_3$…..e t However the reactivity decreases in the order $F > Cl > Br > I$

An excess of aromatic or solvent i.e. dichloromethane. Olefins alcohols can also be employed in place of alky halide

1 .1 .2 Heterogeneous Catalysts

Heterogeneous Catalysts act in a different phase than the reactants. Most

Heterogeneous Catalysts are solids that act on substrates in a liquid or gaseous reaction mixture, Diverse mechanisms for reactions on surfaces are known, depending on how the adsorption takes place, The total surfaces area of solid has an important effect on the reaction rate . The smaller the catalyst particle size, the larger the surfaces area for a given mass of particles .A heterogeneous Catalysts has active sites. Which are the atoms or crystal face where the reaction actually occurs, Depending on the mechanism, the active sites may be a planar exposed metal surface, a crystal edge with imperfect metal valence or a complicated combination of the low? Thus, not only most of the volume, but also must of the surface of heterogeneous Catalysts may be catalytically inactive. Finding out the nature of the active sites requires technically challenging research. Thus, empirical research for Finding out new metal combination for catalysis continues (Spessard etal, 1997) .For example, in the Haber process, finely divided iron serves as a catalyst for the synthesis of ammonia from nitrogen and hydrogen. The reacting gases adsorb onto active sites on the iron particles. Once physically adsorbed, the reagents undergo chemisorptions that dissociation into adsorbed atomic species, and new bonds between the resulting fragments form in part due to their close proximity, in this way the particularly strong triple bond in nitrogen is broken, which would be extremely uncommon in the gas phase due to its high activation

Energy, Thus the activation energy of the overall reaction is lowered and the rate of

Reaction increases. Another place where a Heterogeneous Catalysts applied is in the oxidation of sulfur dioxide on vanadium (v) oxide for the production of sulfuric acid Heterogeneous Catalysts are typically supported which means that the catalyst is dispersed on a second material that enhances the effectiveness or minimizes their cost. Supports prevent or reduce agglomeration and sintering of the small catalyst particles. Exposing more surface area, thus catalysts have higher specific activity on spread to increase the surface area more often the supports and the catalyst interact, affecting the catalytic reaction. Supports are porous materials with a high surface area most commonly alumina zeolites or various kind of activated carbon Specialized support include silicon dioxide, titanium dioxide, calcium carbonate

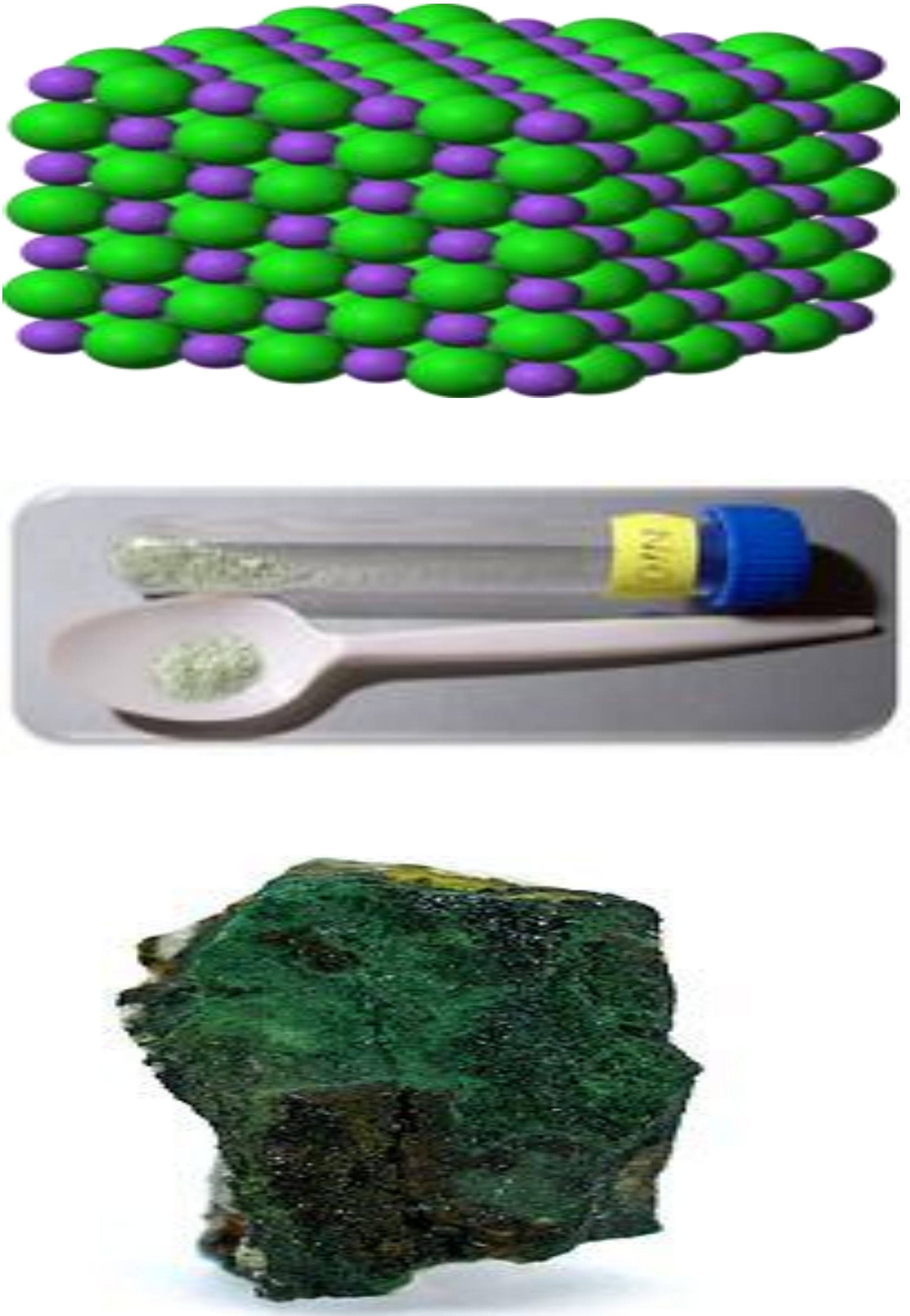


Fig1:1 Sample of nickel oxide

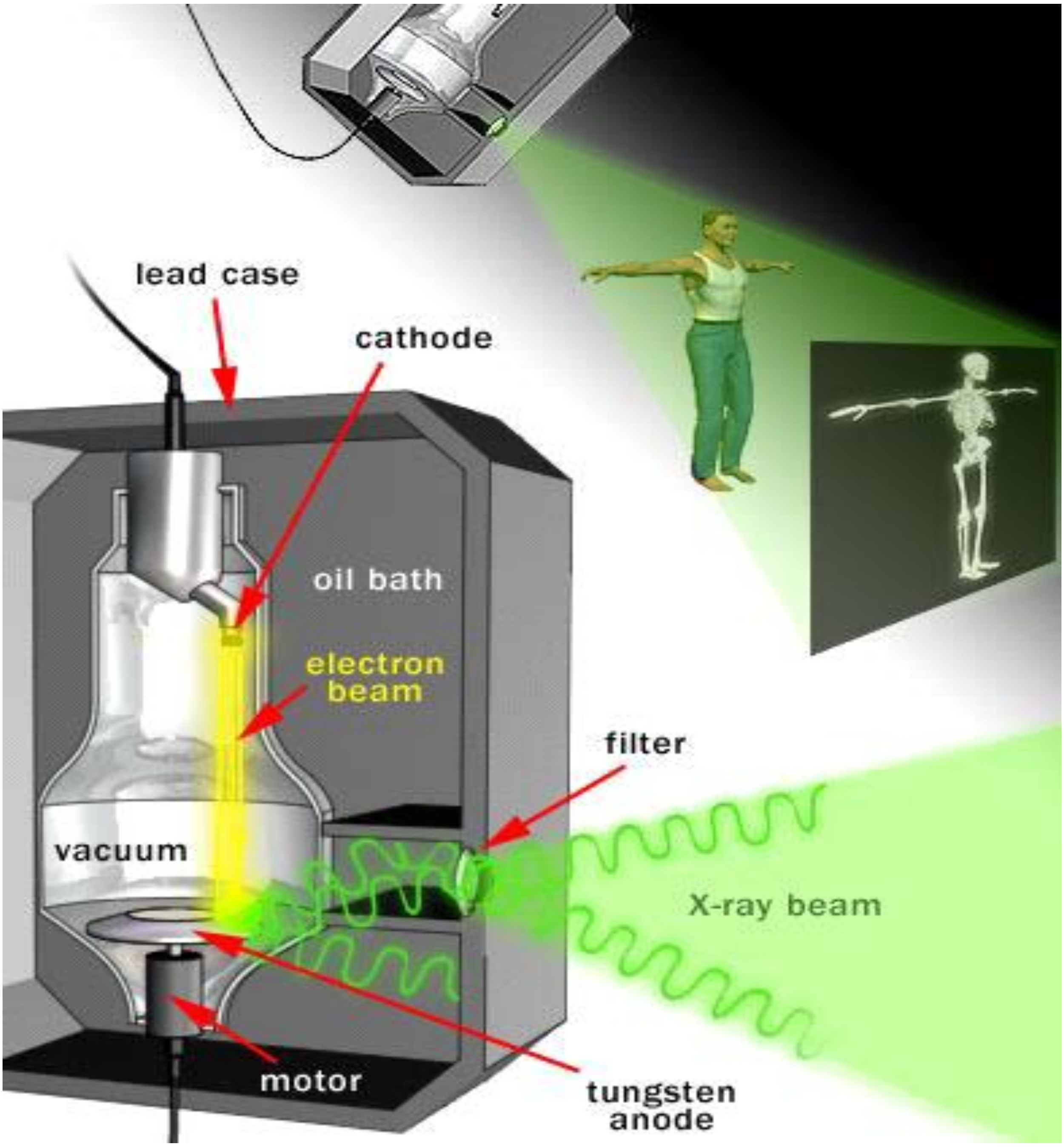


Fig 1: 2 X R D spectrometer

Fig 1:3 Technical operation

1.1.3 Example of Heterogeneous Catalytic Reaction Process

The catalytic process in which the reactants and the catalyst are in different phases is known as heterogeneous catalysis.

Adsorption theory of heterogeneous catalysis explained in two ways.

(i) The reactants in gaseous state or in solutions are adsorbed on the surface of the solid catalyst on the increase in concentration of the reactants on the surface increases the rate of reaction Adsorption being an exothermic process, the heat of adsorption is utilized in enhancing the rate of the reaction.

(ii) The catalytic activity is localized on the surface five steps:

(i) Diffusion of reactants to the surface of the catalyst.

(ii) Adsorption of reactant molecules on the surface of the catalyst.

(iii) Occurrence of chemical reaction on the catalyst’s surface through formation of an intermediate.

(iv) Desorption of reaction products from the catalyst surface, and thereby, making the surface available again for more reaction to occur.

(v) Diffusion of reaction products away from the catalyst’s surface.

The surface of the catalyst unlike the inner part of the bulk has free valencies which provide the seat for chemical forces of attraction. When a gas comes in contact with such a surface, its molecules are held up there due to lose chemical combination. If different molecules are adsorbed side by side, they may react with each other resulting in the formation of new molecules. Thus, formed molecules may evaporate leaving the surface for the fresh reactant molecules.

*product molecules must follow the same track in the reverse direction to return to gas phase. The term green catalytic processes has been used frequently in recent years implying that chemical processes may be made environmentally being by taking advantage of the possible high yields and selectivity for the target products , with little or no unwanted side products and also often high energy efficiency. Heterogeneous catalysts have distinct advantages of easy recovery. And recycling over the catalysts. Some of distinc features of homogeneous and Heterogeneous catalysts are summarized in Table (1.1)

Table (1.1) Advantages and Disadvantages of Homogeneous and Heterogeneous Catalysts

Advantages of homogeneous catalysts	Advantages of Heterogeneous catalysts
1- Efficient heat transfer	1-continuous processing
2 Mild reaction condition	2-facile separating of catalysts and products
3 high activity and selectivity 4-No mass transfer limitation	3-Elimination of corrosion problems
Disadvantages of homogeneous catalysts	Disadvantages of Heterogeneous catalysts
1-Toxic was water after catalyst recycling 2-Contamination of product with catalyst 3-High cost due to catalyst losses (Noble metal complexes) 4- Not readily adopted to a continuous process	1-heat transfer problems 2-low selectivity 3-diffusion within catalyst- temperature control of highly exothermic reactions 4-Harsh reaction condition 5-Mass transfer limitations 6-High mechanical stability required
Example $CaCO3 \xrightarrow{Ni} CaO + CO2$	Example 1 $C2H4+H2 \xrightarrow{Ni} C2H6$
$Mg\ CO3 \xrightarrow{NiO} MgO + CO2$	$H_{2+}O_2 \xrightarrow{NiO} 2\ H_2O$

1 .1 .4 Catalysis and Supported Catalysts

The reactants and products being in one or more liquid phase. The catalytic reaction occurs at the surface of the solid and, ideally, its rate is proportional to the area of the catalyst .The effective surface area of an active metal oxide catalyst can be increased by supporting it on a less active oxide of higher surface area **(**Yuxaim 1977**).**The nature and properties of the support materials are, of course a critical determining factor of the reaction activity of the metal species. In fact, the presence of a carrier may stabilize a particular electronic state of such active components. This fact plays an important role in the catalytic behavior. Of equal Importance, is the understanding of the effects due to the method of catalyst preparation and to subsequent methods used in calcination and heat treatment .In addition, the activities of these supported catalysts in their respective systems are controlled by the concentration level, the oxidation and dispersion state or aggregation of the active metal oxide on the support (Schuit,G,C1951) for understanding the behavior of supported metal catalysts we need to know the degree of dispersion of the metal .The parameter can be expressed as specific metal surface area or the average metal crystallite size. Hydrogen chemisorptions have proved a useful tool to the determine the surface area of supported platinum and nickel But it has hitherto not been possible to apply this technique to supported palladium, because hydrogen tends to be absorbed ,Palladium catalyst are well known and are, widely, used as the most efficient catalysts of selective hydrogenation of organic compounds, and in particular alkynes, other group VIII metal Ni, Ru, or Pt are also active and selective, but less so than Pd.The important aspects of the catalytic hydrogenation of unsaturated hydro – carbons on metals have been extensively discussed by Board and wells and by Quito and Bond.

1 .1 .5 Supports and Surface Chemistry

Both silica (SiO2) and α– alumina (α– Al_2O_3) have been, widely, used as carriers in different catalytic systems

(Jogannatnas 1981). As it has been mentioned, the nature and properties of the support material are critical, determining factors for the reaction activity of the supported species. Many muresligation , have been published on alumina. Silica has also received great attention regarding its preparation, and characterizations of surface hydroxyls (Linson, B, and G1960).

1 .1. 6 Silica Supported

Silica (SiO_2) is an amorphous solid that can be made by heating which can be made by a acidifying a dilute solution of sodium silicate (Na_2 SiO_3) and alloying sufficient time for the silica in solution to polymerize.The hydrogen is mm in diameter made up of a network of interconnected covalently bonded Si tetrahedral. Drying of the hydrogen by heating leaves open space constituting a pore network and internal surface area of perhaps 500 m2/g. The surface is that of the aggregated primary particles, which are n on crystalline (hence the terms silica gel). In comparison with surfaces of most metal oxides, the silica oxide surface is nearly inert. The most reactive groups are – OH groups called Solano groups) that terminate the primary particles these are weakly acidic comparable to alcohols. The bulk may be entirely, terminated, by – OH groups, which can be removed, be dehydrogenation. Two types of Solano groups are, usually, distinguished, isolated groups and neighboring (vicinal) groups that may be hydrogen bonded to each other. Fully hydrated samples, heated in air "i.e. calcited " at temperatures < 200 C also contain germinal groups, Si $(OH)_2$ complete removal of Silone groups requires temperatures greater than $700C$. The a product sites present after dehydrogenation at $600\ C -\ 800C$ have been suggested to be primary highly strained Si – O – Si linkages (Lier,R,K1962).

Table 1:2The Physical Properties of silica

Molecular formula	SiO_2
Molar Mass	60 g/mol
Appearance	White solid
Melting point	1700 C
Density	2.65$g/cm3$
Solubility in water	in soluble
Solubility	soluble in diethyl

1.1.7 Catalysis on metal Surface

Molecules are activated by metal atoms in metal surfaces, in the way that they are activated by metal atoms in metal. For a reactant to be activated, it must be bonded to the metal, which requires coordinative instauration (Heteu, 2003,.)..Metal surfaces have a marked advantage over metal complexes in (e.g. by treatment under vacuum to remove adsorbents) without loss of their structures, metal complexes and clusters, on the other hand,. The compounds require stabilizing laggards, solid metals do, and the laggards that stabilize a surface metal atom are the neighboring metal atoms in the bulk and surface. Therefore, reactivity of metal surface is distinct from that of complexes and clusters of the same metal and not easily predicted, many metals have catalytic activity for many reactions. In practice, catalytically important metals include most of the transition metals that are important in metal complexes catalysis, platinum and nickel group metals find especially wide use, even though they are expensive. Metal surfaces offer many opportunities for catalysis that are not offered by metal complexes. Because the metals are robust, they can be efficiently used at temperatures that are too the stability of most metal complexes in solution or anchored to supports. Metal surfaces can be used with varying degrees of coverage and can be applied as catalysts with a wide variety of reactants, the surface may be able to accommodate and facilitate all the reactions, as many as hundreds of different compounds may be converted, simultaneously, on metals surface catalyst for conversion of petroleum fractions. One of the important concepts of metal catalysis is postulate of active centers (Taylor;s 1925). Taylor inferred that surfaces are not uniform and that only, a minority of specific sites may be active for a particular catalytic reaction under a particular set of conditions. Experiments with selective poisons have typically shown that only a small fraction of the accessible surface is catalytically active. Experiments with well – defined single – crystal surfaces having various crystal faces and various densities have shown, unequivocally, that catalytic activity for some reaction depends strongly on the coordination of a metal center and that, in some instances, only a very small fraction of metal atoms.

1.1.8 Catalysis by supported metals

Metal catalysts are used in the form of powders or screens (gauzes) in industrial processes, but most are used in the form of highly dispersed aggregates on supports. One reason why highly dispraised metals are preferred is that many of the industrial catalytic metals are very expensive, and it is efficient to have most of the metal exposed at a surface and accessible to reactants. Typical supports are robust porous solids, including metal oxides, such as alumina, silica and carbon

1.1. 9 Nickel Element

Nickel (Ni) has a relative atomic number 28, atomic weight 58. 62, valences “2” 3” and electronic captivity 1, 88, it is electronic Configuration is [Ar] 3d, 4s and have partially filled 3d shells either in ground state of the free atom or in one. The concentration of Nickel in water as reported by the national water quality net work in the U S A ranges from 1 ----70mg|liter our knowledge of the possible effects of small quantities of. Nickel, including food and water is very limited and much more research is needed other toxic elements and their compounds are found in natural and polluted water, usually in very small concentrations they include Barium, Beryllium, Cobalt, Molybdenum, Tin, Uranium, Vanadium, and may other insufficient data is available to assess the heath significance of their presence in water. Supported – Nickel catalysts find. Wide, applications in a number of industrial processes such as hydrogenation, hydro cracking and steam reforming. Deactivation of nickel catalysts due to sintering and compound formation during high temperature reactions is a major problem (Satter fild 1980).

The activity of the supported catalyst largely depends on the nature of the carrier used, the active species involved in the reaction may by the metal or metal oxide. Even though the concept of metal support interactions is used in explaining the variation in catalyst characteristics and activity, the nature of the interaction which occurs in the preparation process and during the thermal treatment, and also the type of nickel species present, are still not clear .The phase of silica and the calcinations temperature of catalyst and support do play a major role in influencing the support metal interaction. The presence of metal is reported to name a pronounced effect on the phase transformation and surface area of the support. When the Ni is dispersed on $\mathrm{Si}O_2$, the bonding is intermediate between the Ionic bonding of NiO and the covalent bonding of $\mathrm{Si}O_2$.

Catalysis is influenced by distribution of reactions and catalyst, between phases and by geometric effects associated with groups bonded to catalyst molecular and surface

1.1.10 the Surface Chemistry of Nickel

Nickel may be present in the bulk of silica and simultaneously present as Ni element on the surface of this support. A vast range of techniques has been used in attempts to elucidate the structure and distribution of the supported species and identify and quantify the active sites.

In an innumerous amount of literature concerning moth of the aspect of their performance .These studies have significantly advanced our understanding of supported Ni – oxide catalysts, but a complete molecular picture of these complex and important catalytic material has not emerged. Moreover, the states of nickel that can simultaneously by present in the supported nickel oxide phase have led to much confusion in the literature. Among the debated characteristics, two have been at the focus of a continuing controversy (Houall and Delmont, 2020).

These are.

1- The physico - chemical Identify of the nickel oxide surface species.

2- The oxidation state of nickel responsible for their catalytic activity.

In general, nickel – oxide species gain stability when " dispersed " on a support through "bonding " to the surface hydroxyls, Silica has a low

1.1.11 Catalysts and Industry

Since the first industrial catalytic process was introduced in 1875 to oxidize SO_2 to SO_3 in the manufacture of sulphuic acid, the use of catalysts has been invaluable in developing new products in food chemistry, organic fine chemicals, heavy inorganic chemicals and the petroleum industry. Industrial catalysts should, usually, have all of the following properties or at least on optimum compromise of them (Rymer,Bridge and Tomlis1971).

1- High and stable activity
2- Thermal and mechanical stability
3- Selectivity towards the desired product
4- Low cost.

In some instances these conditions can be met by a new catalyst. The cost of changing the plant to accept and run on this new catalyst can in the short term be quite unacceptable. This will mean that new catalyst, although superior, will not be used. Therefore, there is an added condition that a new catalyst must fulfill in order to replace an existing catalyst, it must be able by its additional properties, to overcome industrial and historical inertia.

1.1.12 Alumina

Aluminum oxide is a chemical compound of Aluminum and oxygen with the chemical formula $\mathrm{A}l_2O_3$ It is the most commonly occurring of several aluminum oxides, and specifically identified as aluminum (III) oxide. It is commonly called alumina, and may also be called oxide, aloxite, or launder depending on particular forms or applications It occurs naturally in its crystalline polymorphic phase $\alpha\, Al_2O_3$ as the mineral corundum, varieties which forms the precious gemstones ruby and sapphire A_2O_3. is significant in its use to produce aluminum metal, as an abrasive owing to its hardness, and as refractory material owing to its high melting point (Zumdahl, 2009) .Corundum is the most common, naturally, occurring crystalline form of aluminum oxide. Rubies and sapphire are gem-quality forms of corundum. Which one their characteristic colors to trace impurities,. Sapphires come in different colors given by various other impurities, such as iron and titanium Al_2O_3, is an electrical insulator but has a relatively high thermal conductivity for a ceramic material aluminum oxide is insoluble in water In its most commonly occurring crystalline form, called corundum or α aluminum oxide , its hardness makes it suitable for use as an abrasive and as a component in cutting tools. Aluminum oxide is responsible for the resistance of metallic aluminum to weathering; metallic aluminum is very reactive with atmospheric oxygen, and thin passivation layer of aluminum oxide. Forms on any exposed aluminum surface

1 .1 .13 Alumina – Supported Nickel Catalysts

Are extensively used in various oleo – and petrochemical processes, including de-aromatization of commercial solvents and oils, hydrogenation of gasoline " per gas ", olefins, edible oils, and osmotic compounds. A new generation of hydrogenation catalysts has been developed which combines a high specific nickel surface area of high reducibility indicating a high dispersion and a limited, but effective, metal support interaction. A hi ghly selective hydrogenation catalyst with limited hydrogenation potential for the conversion of di–olefin hydrocarbons into mono – olefin hydrocarbon.

Table 1:3 The Physical Properties of Alumina

Molecular formula	$Al_2\ O_3$
Molar Mass	102 g/mol
Appearance	White solid
Melting point	660 and 1220 C
Density	2.7g/cm3
Solubility in water	in soluble
Solubility	in soluble in diethyl

1 .1 .14 Heavy Metals Catalyst

Metals such as Pt, Pd, Ru, Rh, Os, Co and Ni have been used in the reduction of

hydrogenation of olefins and carbonyl compounds in organic syntheses. The metal catalysts are prepared in the form of, not only metal particles dispersed on supporting materials but also porous bulk metals. Porous metals such as Nickel and Cobalt prepared by dissolution of Al from Ni –Al and Co- Al are frequently used as catalysts for liquid phase hydrogenation (Pfoels , Beek 2019).

Ni- MgO catalysts, which are prepared using nickel nitrate, magnesium Reduced fine nickel powders are also effective as bulk metals for catalytic hydrogenation (Wang.H X Chem 1997)

Nitrate and citric acid, have high nickel metal surface areas and efficient catalytic activities for several gas phase reactions.

In our preliminary work, have Ni –Mgo catalyst with high Ni surface area to the liquid – phase hydrogenation of cyclohexan one to cyclone hexanes, and found that the Ni- MgO catalysts with high Ni contents ranging from 60 to 80 wt% exhibited reaction rate higher than those of rainy nickel catalysts which had pores smaller than these of the nickel MgO. It has been reported that hydrogenation rate in the liquid. Phase sometimes has no correlation with metal surface area to Raney nickel (Freel.J.pieters 1970). Speculated that the diffusion is in the mesopore region probably cause the reaction rate.

1.1.15 Supported NiO and WO3 Catalysts are known for Catalyzing

Different important reactions, and NiO decomposition .WO_3, Al_2O_3 for propone metathesis WO_3\SiO for olefin disproportion (Bailey1969) and WO_3 for the reduction of NiO in combustion flue gases. Supported NiO-WO_3 samples deserve attention as they are precursors of the widely, used, hydride sulfurization (HDS), hydrogenation and hydro cracking catalysts. Although under HDS reaction conditions the catalysts are being changed to sulfide to a rage extent, it is relevant to study their oxide precursors as it has been reported referee that there are some direct relation –ships between the properties of the oxide precursor and the corresponding sulfide catalysts .It is expected that hydrogenation and hydro cracking reaction will become important due to the need to produce clean fossil fuels heavy petroleum fractions and coal.

1.1,16 Synthesis and Characterization of NiO

The nickel oxide NiO became one of the most important transition metal oxides. However most of The nickel Oxide NiO: Became one of the most important transition metal oxides . However most of these applications require particles with a small size distribution. With the volume effect, the quantum size effect and the surface effect NiO nanoparticles are expected to possess many improved properties and even more attractive applications than those of bulk-sized NiO particles. For example .NiO nanostructures are P- type semiconductors with particular magnetic such as super paramagnetic. Superantiferromagnetic, and ferromagnetic order depending on the particle size, particle shape and synthesis route, where as bulk- sized NiO is an ant ferromagnetic insulator with an eel temperature.

Table 1:4 The Physical Properties of nickel oxide NiO

Molecular formula	NiO
Molar Mass	74,62 g/mol
Appearance	black to brown powder
Melting point	1,955 C
Density	6,67.$g/cm3$
Solubility in water	in soluble
Solubility	soluble in ammonium chloride

The present study is to investigate the effects of these different It is found that the work already surveyed and checked in the literature on silica-supported is confined to that prepared mainly by impregnation on the carrier with soluble nickel salt. It is worth mentioning that the preparation of supported catalyst by coating method, developed by zaki el, is believed to result in a more extensive two-dimensional distribution of the catalyst precursor on the support surface, as compared to the catalysts obtained via the conventional impregnation method (Nakayama1997), In accordance, this study adopted this method for the catalyst preparation as far as the catalyst precursor is concerned; only one catalyst precursor was used. The impregnation has been carried out from only one aqueous solution containing Ni cations from nickel chloride (NiCl_2) via impregnation method. Silica supporting, carrier was used, where the concentration was varied between 1% up to 10% besides the calcinations at 500 C (encompassing commercial conditions), the catalysts were also cal cited at 300 C to follow the changes that may occur on the surface. X-Ray technique is the one of physico- Chemical methods which plied two characterize the catalyst materials

I .1.17 Transimmion Electron Microscope (SEM–EDX) Studies

Transimmion electron microscope (TEM) uses transmitted and diffracted electrons. TEM instrument is, similar to an optical microscope in one replaced by electromagnetic lenses in TEM, a primary electron beam of high energy and high intensity passes through a condenser to produce parallel rays which impinge on sample. As the attenuation of the beam depends on the density and the thickness the transmitted electrons from a two-dimensitional projection of the sample mass, which is subsequently magnified by the electron optics to produce also called bright field image. The dark field image is obtained from the diffracted electron beams, which are slightly from the transmitted beam. Typical operating condition of a TEM instrument is 100 – 200 KeV electrons, mbar vacuum .0 .5nm.(Bron,1992,. Sons, New York pail 2,310)

1.1.18 Scanning Electron Microscope (SEM – EDX) Studies

Scanning electron microscope (SEM) is carried out by scanning a narrow electron beam over sample surface and detecting the yield of either secondary or backscattered electron as a function of the position of the primary beam. Contrast is caused by orientation, parts of the surface facing the detector appearing brighter than parts of the surface with their surface normal pointing away from the detector. The secondary electrons have mostly low energies in approximate range 5 – 50 KeV. And originate from surface region of the sample. Backscattered electrons come from deeper regions and carry information of the composition of the sample, because heavy electrons are more efficiently scattered and appear brighter in the image (Burwell,(200),.Adam, catal ,26351)

1.1.19 Metal Powders

The methods of making unsupported metals in forms suitable for use as catalysts in gas-phase reaction is a very widely used, method is reduction of a suitable compound of the metal in hydrogen is, eminently, the most successful, for it is readily purified and leaves no undesirable residues on the surface as. For example, carbon monoxide and formic acid. The nature and source of the compound of the metal is important, it is customary with the noble metals of group viii to start with chloride, since is the form in which they are normally obtained, reduction after induction period in which the metallic nuclei are formed, may are possible at room temperature but in any even the use of temperatures much over 200 ℃ is unwise by reason of the low sintering temperature of these metals. In the later cases the physical nature of the oxide depends on the condition of the precipitation and hydration or decomposition of the compounds, the nature of the metal ultimately formed in the reduction is determined by the physical what of the parent compound. But the furthermore the condition of the reduction affect the final product. Temperature certainly affects the surface area, while other possibly relevant variables are (i) the flow- rate and the purity of hydrogen (ii) The Temperature (iii) the length of the time the process is continued. If the metal has to be transferred to the reaction vessel, it is most desirable that the material be cooled to room Temperature before exposure to air , because of the pyrophoric tendency of such preparation it is better to reduce the oxide in metallic powder so prepared have been successfully freed from adsorbed hydrogen by prolonged thermal desorption. An entirely different method of preparing metal powders is the thermal decomposition under vacuum of metal salts of organic compound .An interesting study of cobalt formed in the decomposition of organic cobalt salts has recently be published(Ellision,1988,.Diakun,catal 46).

1.1.20 Skeletal Metals

These are prepared from binary alloy catalyzed and second metal easily soluble in suitable, reagentsraney nickel is obtained by treating a 50.50 nickel. Aluminum alloy with concentrated solution of sodium hydroxide until most or all of the aluminum has dissolved. this form of nickel possesses Avery open or skeletal structure and thus presents a high surface area .It is, widely, used in preparative organic chemistry .and it is case of preparation is attractive .The preparations and use of skeletal iron, copper and cobalt and .has been descry bed but there are generally less active than nickel

1.1.21 Adsorption on Solid Surfaces

For simplicity, the formation of a surfaces by fracture of a crystal of a covalent solid, any metal In this covalent bonds between atoms are broken, and so each surfaces atom must process one or more free valencies.The number and a types of these valencies depend on the bonding between atoms in the bulk solid and the angle that the fracture wake through the crystal. An atom in the new surface is clearly in rather unusual position in. It is coordination number is smaller than for atoms within the bulk of the solid (McMaster, Hall1981). One form of Adsorption occurs as a result of a molecule interaction with these free valences, this may be regarded as a chemical reaction because there is an arrangement, sometimes drastic, of the electrons within the molecule. The type of adsorption, therefore, termed chemical adsorption or chemisorptions.

A precisely analogous situation occurs of ionic solid chemisorptions on such surfaces attempts to rectify this situation, and in consequence it is often ionic or at least highly polar in character. Molecules whose electrons are not easily polarizable are only weakly adsorbed on ionic solids. The other form of adsorption, which recognized to occur at the surfaces of solids, is due to Vander Waals forces such as existing between molecules themselves in the liquid state. These include electrostatic attraction in the case of molecules with permanent dipole moment, and induced dipolar attraction with readily polarizaple molecules, dispersion force caused by slight fluctuations in electron density are the only forces of attraction between non-polar atoms and molecules. When these forces are exerted between an atom or molecules and surface, there is physical attraction of the molecules; this is termed physical adsorption or less often. The strength of physical adsorption with clearly related to the observable physical properties of the adsorbing is species. Thus for example, the adsorption of inert gases and of nitrogen and hydrogen will be weak and detectable only at low temperature, when as, in the cases of say water and benzene, adsorption will be stronger and observable 393K chemisorptions and physical adsorption are usually distinguishable from each other without any difficulty.

Table 1:5 distinguishing between chemisorptions and physical adsorption

Criterion	Chemisorptions	Physical adsorption
Enthalpy of adsorption	40- 800 Kj mole	8 -mole20Kj
Activation energy, E	Usually small	Zero
Temperature of occurrence	Depends of E but usually low	Depends on boiling point, but usually low
Number of layers	Not more than one	More than one

adsorbed		possible

The molar enthalpy of adsorption is the heat liberated when amole of a substance is transferred from the gaseous to the adsorbed is state. For chemisorptions, values are in the range that is typical of chemical reactions and generally fall between 40 and 800 Kj/ mole. For physical adsorption, values are usually in the range found for enthalpies of liquefaction or vaporization and are typically less than 20 Kj/ mole. .Higher values are sometimes observed, especially when a higher polar molecule such as water adsorbs on an ionic solid in such case it may be difficult to define the kind of adsorption occurring. The new bonds formed in Chemisorptions at a metal surface are always dipolar to some degree because of electro negativity difference between. This produces a slight increase or decrease in number of condition electron on in the solid, which may be sensed by measuring the change in electric conduction consequent upon adsorption. This is, easily, done in the case of adsorption on metal Wires. Physical adsorption produces no electrical effects (Hall,Heteroge 2009).Chemisorptions leads at most monolayer coverage of the available surface. With Physical adsorption, several adsorbed layers may be formed, especially, near the normal boiling point of the adsorbing substances. The rate of Physical adsorption is always as is the condition of a vapor on the surface of it is own liquid, because there is no activation energy. The process of Chemisorptions, on the other hand, has activation energy.

1.1.22 Catalysts preparation methods

Solid catalyzed reaction involve, the interaction between substrate molecules on the surface of the active material hence, the most efficient catalysts should have a high population of active centers on the surface exposed to the reaction medium. Very fine metal particles that have the high ratio of surface to bulk atoms needed for good catalyst activity however, these fine particles easily sinter under reaction condition. Hence, the most common way of minimizing metal catalyst sintering is to disperse the active metal component over a porous and thermally stable, support. Some methods of catalysts preparation are co-precipitation, deposition, impregnation, incipient wetness and ion exchange. Essentially most of the preparation methods for the support metal catalysts involving major step namely, co-precipitation.(Gossner,1979,.Mizera, chem.9837).

1.1.23 Co - precipitation

It involves the addition of a precipitating agent to a solution containing both a support and catalyst precursors. The resulting precipitate contains species, either as single or multiple phases, from which the active component and the support material are eventually processed. The initial step in the preparation of co-precipitated catalyst is the reaction between solution of two or more metal salts and a base, generally a hydroxide, alkali carbonate or bicarbonate. The resulting precipitate may contain not only the insoluble hydroxides and carbonates, but also a mixed metal compound if the solubility equilibrium is favorable. Even if the formation of a mixed metal compound is not favorable, some of the support material is usually trapped in the active metal precipitate. This causes the formation of large crystals of the active metal compound. Smaller crystals are easier to reduce and generate more finely divided metal particles. One of the problems associated with the preparation of co- precipitated catalyst is the formation of vapor liquid interface inside the pores of the solid during drying and calcination of the precipitated catalyst precursor. Use of solvents with high surface tension.e.g water resulting in particle collapse of the solid gives low surface area material. The problems can be overcome using a solvent having surface tension lower that water. Hence, a well known (Sol-gel) method is adapted in which the alcoholic solution of the active metal and support precursors are used precipitation is initiated by the addition of stoichiometeric amount of water. For example, alcoholic solution of tetraethoxysilane and palladium ammonium chloride with ammonia gives a gelatinous Pd-SiO2 precipitate which is dried at 353-373K and then heated to 723K this on reduction in H_2 gives a high dispersed Pd\Si O_2 catalyst. Another commercially used Copper-cremate or Adkins is also prepared by co- precipitation method which involves the addition of Copper nitrate solution to a solution of ammonium dichromate in ammonia giving a precipitate of Copper dichromate. This precipitate is clacinated and washed with acetic acid to give the cooper cremate catalyst.

Another problem associated with co- precipitation method is that, relatively large amount of active metal is retained inside the particles of the support and therefore remains unavailable for the reaction. This problem can be overcome by a sequential, precipitation procedure.

1.1.24 Deposition

Deposition is process in which the active component or precursor is added to a separately prepared carrier or support. This is preferred because (i) during co- precipitation, solid catalyst contains small metal particles trapped throughout the porous oxide support with effective use of the metal catalyst, (ii) it is difficult to form inset support with a prescribed surface area and pore structure. Deposition can be accomplished in two ways.

1.1.25 precipitation-deposition

In this procedure, a precipitating agent is added slowly to a well stirred suspension of a support in a solution of the metal precursor. For example slow addition of sodium hydroxide to suspension of alumina in a nickel chloride solution gives Ni|Al_2O_3 catalyst. Alternatively, aqueous hydrolysis of urea librates the base for homogeneous precipitation in an aqueous solution of metal precursor and the support, after isolation the supported precipitate is washed, dried and to produce support oxide which is then reduced to give the active hydrogenation catalyst. Precipitation- deposition method is used to produce a variety of a commercial catalyst e.g. Nickel on silica, alumina, magnesium, titanium, thorium, ceria, and also precious metal like palladium (Deutscmann, 2009,.Olaf ,2,3).

1.1.26 Reduction -deposition

In this procedure, a solution of a metal salt containing the support is treated with a reducing agent to give the supported reduced metal catalyst directly. Carbon supported platinum is obtained by treating aqueous suspension of carbon in palladium chloride. Pt\ C catalysts are prepared Similarly, however a small amount of palladium chloride is added to facilitate the complete reduction of chloroplatinic acid in a photo deposition procedure the semiconductor support is suspended in a metal salt solution an irradiated with a mercury lamp e.g. Pt /TiO_2 catalyst in which small platinum crystallites obtained by photo deposition are uniformly distributed on the titanium surface.(Bull,2014,. Korean vol,35).

1.1 .27 Impregnation

Impregnation is defined as a means of catalyst preparation by the adsorption of a catalyst precursor salt from solution on to a support material. This is also known as wet- Impregnation since; the pores of the support are filled with solvent before coming in contact with precursor salt. In this method, an intense interaction between the surface of the support and the salt takes place leading to the adsorption of the salt on to the support due to stirring suspension of the support on the salt solution for a long duration followed by the separation of the supported catalyst by filtration, centrifugation or evaporation. The supported salt is then calcined and reduced to the metal. Several types of catalysts are prepared by this method particularly, highly loaded supported metals. Major factors affecting the degree of Impregnation and metal distribution are, the solvent used (organic or aqueous), PH of the solution, nature of the metal salt or complex used, and presence of additives or modifiers.

1.1.28 incipient wetness

Incipient wetness, also known as dry impregnation or capillary impregnation, involves contacting a dry support with only enough solution of the Impregnate to fill the pores of the support. The volume of liquid needed to reach this stage of incipient wetness is usually determined by slowly adding Small quantities of the solvent to a well stirred weighed amount of support until the mixture turns slightly liquid. Some of catalysts were prepared

(Commercially) using is method includes Pt /SiO_2, Pt/Al_2 O_3, Ni/C, Ru/C.

The experimental condition dramatically, influences, the dispersion and. For example, in case of Ni /Al_2O_3catalysts PH and the weight loading of the metal affecting the activity of the resulting catalysts.

1.1.29 Ion exchange

In this method, the PH of the impregnation solution must be either sufficiently high or low to provide the appropriate surface potential and the adsorbent must have proper charge. The PH at which the net charge on the support is zero is known as the is electric point (IEP) of material. For a negatively charged surface, cationic species are attracted and get adsorbed. Similarly, negatively species are adsorbed on a positive surface.

1.1.30 Calcination

The main object of calcination (Thermal treatment in oxidizing atmosphere) is to stabilizewe physical and chemical properties of the catalyst or it is precursor. During calcinations, thermally unstable compounds (carbonate,. During calcinations new compounds may be formed, if the thermal treatment is carried out at high temperatures. For example, in the thermal decomposition of Cu or Ni nitrate, not only CuO but also Cu or Ni aluminates are, additionally, formed, Furthermore amorphous materials become crystalline. Various crystalline modifications undergo reversible or in reversible changes. Physical and mechanical properties and pore structures can also change. Calcinations temperatures are usually slightly higher than that of the catalyst operating temperatures. For the calcinations of powder or granulate, ratty kilns are preferably used. Smaller batches of powdered catalysts are in muffle furnaces with trays, as in the case of drying. The gases that are mainly used for heating are in direct contact with the material (Pfoels, 1995).

2.1 Objectives of the research

The aim of this work can be summarized in the following points:

- To prepare Silica supported as Nickel oxide catalyst
- To characterize the prepared catalyst using different analytical techniques such as XRD and EDS-SEM.
- To study the characterize of Nickel oxide catalyst
- To study the characterize of Nickel chloride

Materials and Methods

2.1 Materials

1 –Nickel chloride $\mathrm{NiCl_2}$ Of 99.9% purity was used as precursor.

2-Silica dioxide ($\mathrm{Si}O_2$) powder non porous material has amorphous structure and high surface area

3 –Distilled Water H_2O

2.1.1Apparatus and glassware

Dropper, Glass rods

Funnels

Watch glass

Instruments:

-X Ray-Diffraction meter model JSX-6pA

- Energy dispersive X-Ray Spectroscopy (EDS) and (SEM)

2: Methods

2.1.2 Preparation of nickel chloride solution:

The nickel coated silica catalyst was prepared by the impregnation method. The impregnation solution prepared via dissolving the precursor (NiCl2) to give the required Ni concentration 1%, 3%, 5%, 8%, and 10 %. mole\ NiCl2 in a suitable volume of distilled water about 20ml for each one to get the support. The amount of water required is arrived at by titrating a known of the support with water the support silica was sprayed slowly in to the solution (for each loading) while being continuously stirred for one hour, till a homogeneous slurry was formed the slurries of various nickel loading in the range of 1% up to 10 % mole Nicl2 as maintained above, were then deride at 110 C without washing then dried materials were then cal cited at 300 C and 500 C for 5hours.

The amount of water required is 20 ml arrived at by (titrating) a known weight of the support with water. The amount of metal salt (A) required giving a desired percentage concentration (B %) of the metal on this support is obtained by the following equation:

$$A = \frac{W \times B}{C}$$

A= the amount of metal salt

B = loading of the metal on support.

C = percentage of metal in the salt.

W = weight of support.

This method has been employed to calculate the amount of metal. Giving the nominal concentration of the metal in all the catalysts prepared. The catalyst is then dried in air at 110 C usually and at 300C and 500 C for 5 hours

$$\%\text{Ni Cl2} = \frac{58.62 \times 100}{129.62} = 45.225$$

There for amount of Nicl2 required for

1% concentration is = $\frac{4\times1}{45.225}$ = 0.0 9g

3% concentration is = $\frac{4\times3}{45.225}$ = 0.27 g

5% concentration is = $\frac{4\times5}{45.225}$ = 0.44 g

8% concentration is = $\frac{4\times8}{45.225}$ = 0.71 g

10% concentration is = $\frac{4\times10}{45.225}$ = 0.88 g

2.2.2 Characterization method (Microstructure Characterization)

Metallographic examinations were carried out on the material under consideration, to correlate the parameters affecting the mechanical properties, such as grain size and the distribution of the particles in the matrix, as well as the main phases and intermetallic phases present.

X-ray diffraction (XRD)

- The powder sample was characterized by X-ray diffraction (XRD) (Joel-rang diffract meters model JSX-6pA) to determine the composition using CuKa (l = 1.5405 Å) radiation. The average crystallite size (t) has been calculated from the line broadening using Scherrer's relation:,
- $D = 0.9\lambda/\beta \cos\theta$

Where

λ is the wavelength of X-ray

β is full width of half maximum (FWHM)

θ Is peak obtained angle

Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX**):**

Microstructures of the different deposits were characterized by scanning electron microscopy (SEM), as well as Energy Dispersive X-ray Spectroscopy (EDXS).

The specimens were coated with golden solution and were taken at room temperature by inserting the samples through the sample holder into the inside the instrument; Photomicrographs at different zones and magnifications were taken of each specimen at 30 kV, and was used to define the distribution of the particles in the matrix and on the grain boundaries. Photomicrographs of Energy dispersive X-ray spectroscopy (EDXS) at 10 kV was also used to determine the distribution of elements in the particles that are present in the matrix and on grain boundaries(Citation,Reads 2014).

Chapter three

3 -Results and discussion

3.1 Physico-chemical characterizations of catalysts

The physico-chemical characterization methods used to elucidate the structure and distribution of supported species and to identify the active sites or the surface functional groups have been recently reviewed most of these techniques have been successfully used to study nickel silica (Ni\SiO2) System,

The Energy-dispersive x-ray spectroscopy analysis of Silica Support calculate at 500C° at different concentration level such as 1% 3%, 5% 8% and 10% The Ni have two samples of the spectrum obtained for Ni oxide free SiO2 calcited

All this figures calculate at 500 C the concentration level, reproduce solely the Pattern of support silica for the series, the diffract gram groups display the Pattern of formation of new species or these species are formal from NiCl2.

One can easily observe the development of the new characteristeristic peak. Compare between tow figures

The activity and selectivity of a metal support catalyst s strongly depend upon the method of catalyst preparation employed (Delomon B.Jacops1978). In the present investigation the nature by XRD spectroscopy at every elementary step of Ni\Si$O_2$, catalyst preparation by impregnation method.

The investigation has concerned the nature of surface complexes produced upon impregnation, and calcinations, in relation to the active phase after reduction. The XRD results allowed us to distinguish two consecutive steps during the treatment of Ni|SiO2 catalyst. In the first step, and 100 C to 300 C the continuous increase in Ni – O and NiCl coordination suggest Nickel (NiOCL) s and the progressive formation of hydroxyls chlorate complex such as (Ni (OH) C l). Also in this step oxide of Nickel were absent in XRD spectrum, Nickel| SiO2,at 300C figure(3.16) at all loading level ,the NiO phase could not be detected by XRD. The spectra of unreduced catalyst appear to depend in calcinations temperature This result is in agreement with earlier work on silica supported catalyst . The band at 130 and 2 $\emptyset$ may be ascribed to be Nickel surface complex.

In the second step the temperature was raised from 300 C and 500C

The absorption band obviously shifted to transformation process eading to the formation of a new species (Ni(11) or NiO species phases are present in Silica surface. The spectra of unreduced catalyst appear to depend on the calcinations temperature. This result is in agreement with earlier work on silica supported catalysts (Scheffer,B, Armp1986). As the loading increase most of the additional Ni form the oxide phase are increase proportional with increase on metal loading. Moreover, indications are seen on the figs, for the formation of Ni –Silica phases.

Most states reporting elimination of Cl from Ni| Al_2O_3 or Ni|SiO2 catalysts indicate that water plays a role in the activation fragments.

The surface structure of silica Gel is widely accepted as consisting of hydroxyl (Sileanal) group and oxide (Silo one) linkages. Due to the interaction between Silica and water, there is conversion of Sloane to Solana surface sites. The alumina surface also has OH group but these less reactive

those of Silica (Subramaniar,1991,.Schattha, catal .69,235). towards certain. The iodide point of alumina is 7.4

(Nakayama, 1997). whereas this value is 3.1 for silica ,therefore the silica surface becomes more negative charged that alumina at alkaline PH , have studied the EXAFS of Ni\Al2O3 catalysts

made by the impregnation methods, reported that NiO is formed by calcinations for the sample with high Ni concentration.
Use EDS in different loading level

The results of Fig (3.3) and Fig (3.4) x –ray spectra of nickel chloride To change brown, red, blue and green look figures (3:13), (3:14), (3:15), (3:16), (3:17), (3; 18),(3:19), (3:20), (3:21) (3;22) and(3:23)The figure at 500℃

Impregnated silica at 1% concentration at 300 the intensity equal 350 the peak Show figures (3.1) and (3.2) x –ray spectra of nickel chloride Number is equal 22 but 1 % at500 the intensity equal 300 the peak number equal 18 Impregnated silica at 3% concentration at 300 the intensity equal 440 the peak Number is equal 19 but 3 % at 500 the intensity equal 400 the peak number is equal 16 The results of Fig (3.5) and Fig (3.6) x –ray spectra of nickel chloride impregnated silica at 5% concentration at 300 the intensity equal 720 the peak chloride impregnated silica at 8% concentration at 300 the intensity equal 900 the Number is equal 20 but 5% at500 the intensity equal 700 the peak number is equal 17The results of Fig (3.7) and Fig (3.8) x –ray spectra of nickel Peak number is equal 19 but 8% at500 the intensity equal 480 the peak Number is equal 16 The results of Fig (3.11) and Fig (3.12) x –ray spectra of nickel chloride Impregnated silica at 10% concentration at 300 the intensity equal 950 the peak Number is equal 14 but 10 % at500 the intensity equal 750 the peak Number is equal 11 the number of peaks to be more than number of peaks at 300℃. The number of peaks increasing to as calcinations temperature Decreasing. But the number of peaks decreasing as increase temperature Increasing. The intensity increasing at low temperature or at 300℃ and Decrease at high temperature or at 500 C Also at high concentration level. Silica supported Nickel oxide is formed. Nickel oxide NiO nano-particles were produced via rapid microwave Method. NiCl2 precise was obtained by slow drop wise addition, the resultedInformation of NiO nano-powder mean dimension of this powder was 30 nm according to the image analyzed by scanning electron microscope (S EM) the Morphology observed in the sample not showing any hard grains which gives the idea that size of the particle is small and has a sinle phase. X –ray diffraction (X R D) pattern revealed well–crystallized high purity nao structure of the syntheses powder. Microwave utilization increased (Wiley, 2007,. P.623).

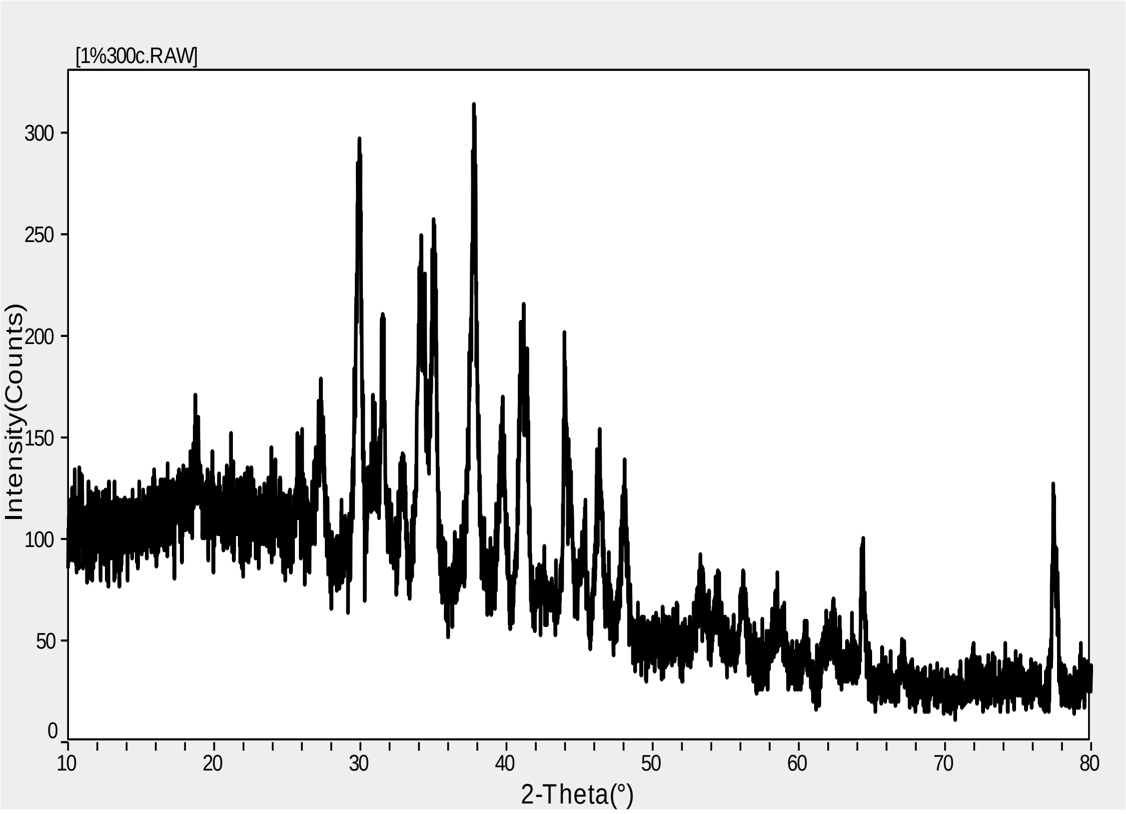


Fig (3.1) X-ray Spectra of Nickel chloride impregnated silica at 1% concentration at 300 C calcinations temperature

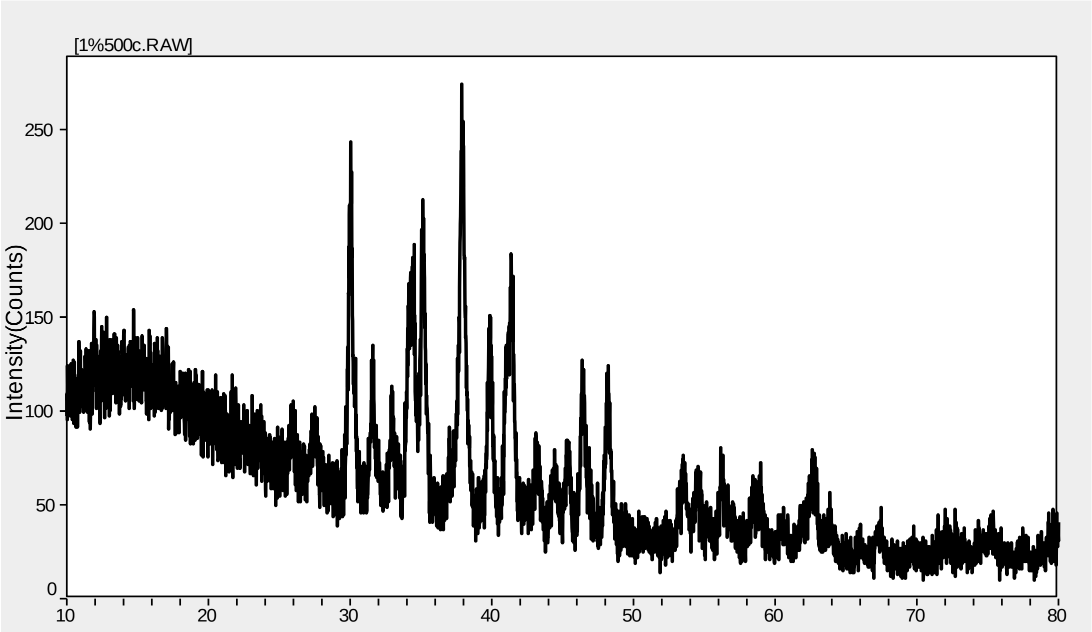


Fig (3.2) X-ray Spectra of Nickel chloride impregnated silica at 1%

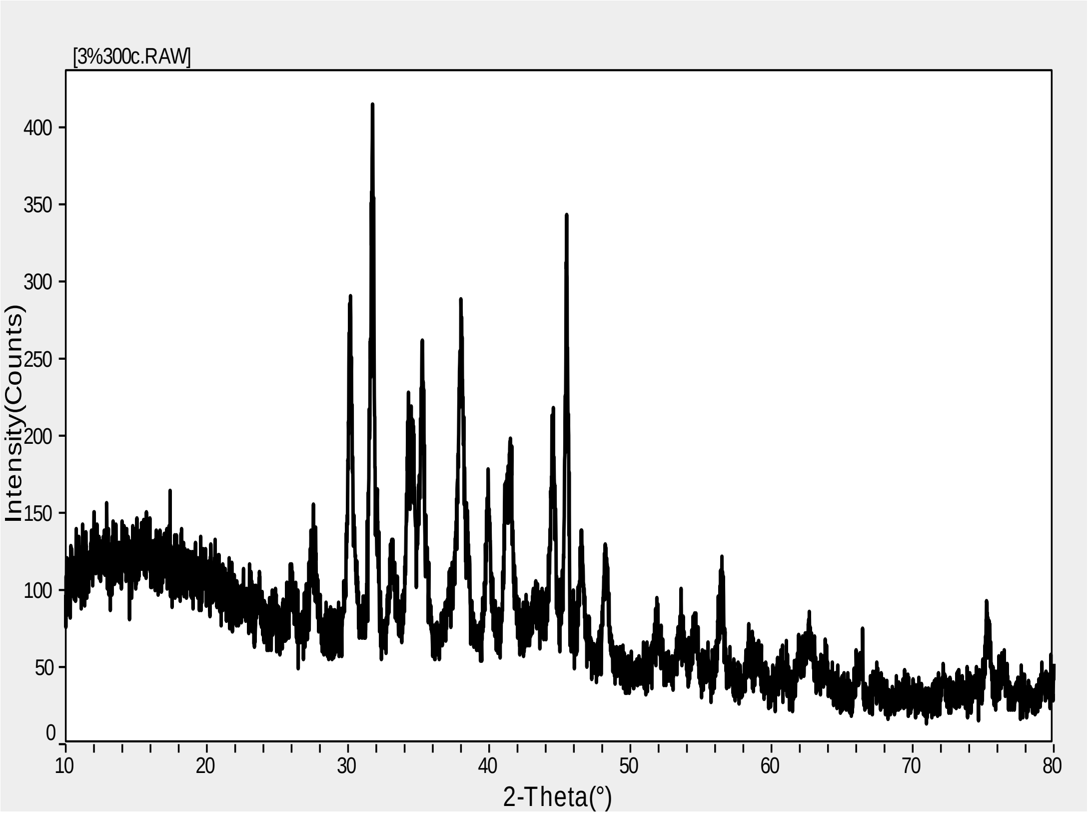


Fig (3.3) X-ray Spectra of Nickel chloride impregnated silica at 3% concentrationat300 C calcinationstemperature

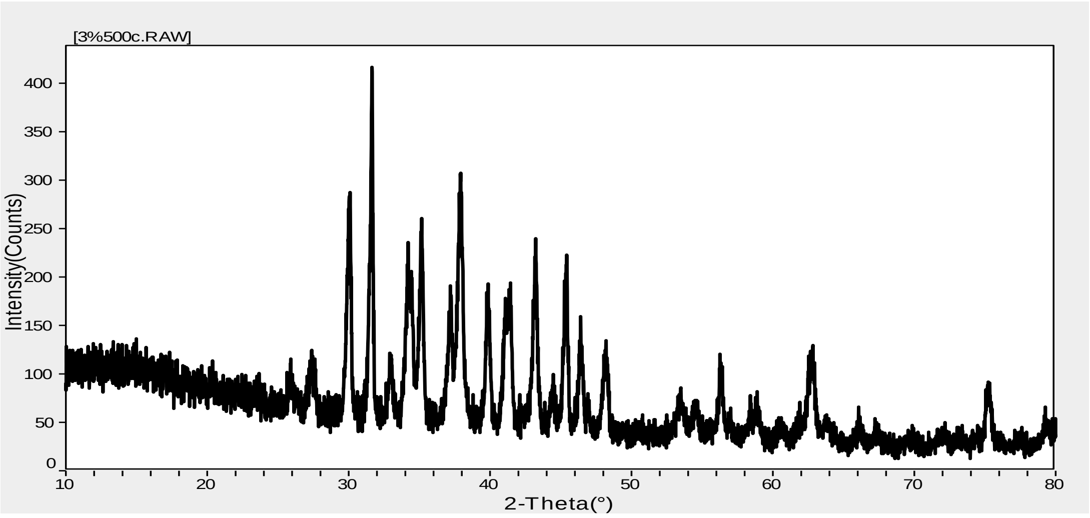


Fig (3.4) X-ray Spectra of Nickel chloride impregnated silica at 3% concentration at 500 ***C*** calcinations temperature

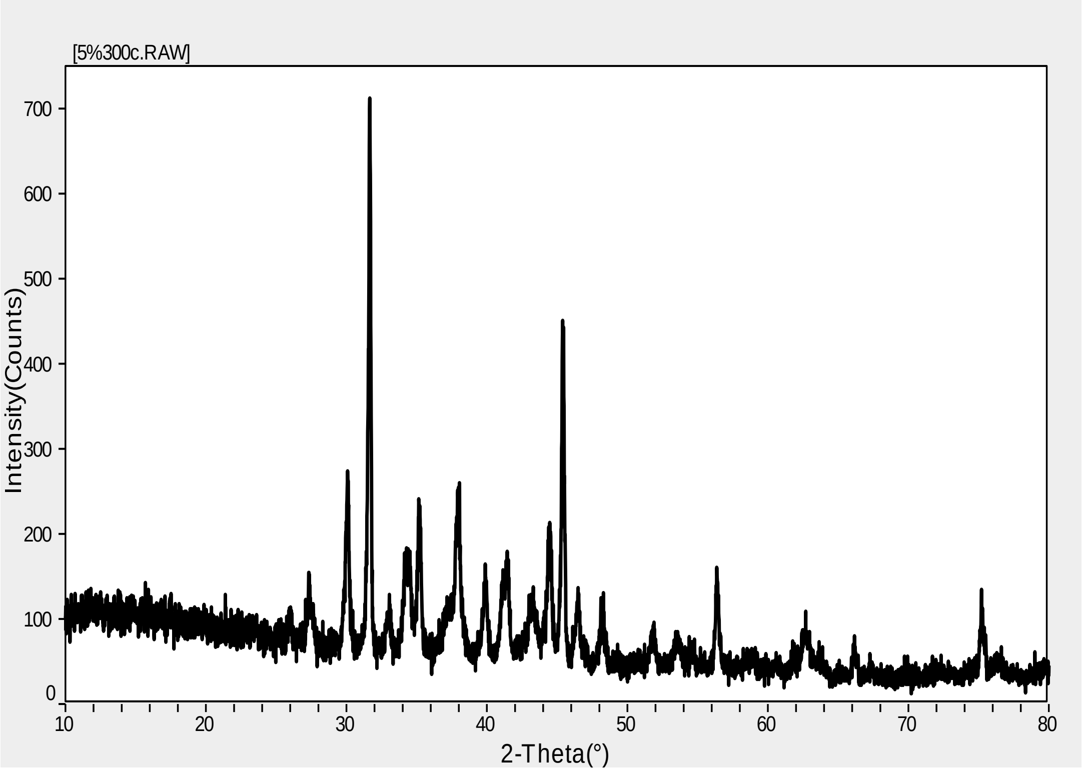


Fig (3.5) X-ray Spectra of Nickel chloride impregnated silica at 5% concentration at 300 C calcinations temperature

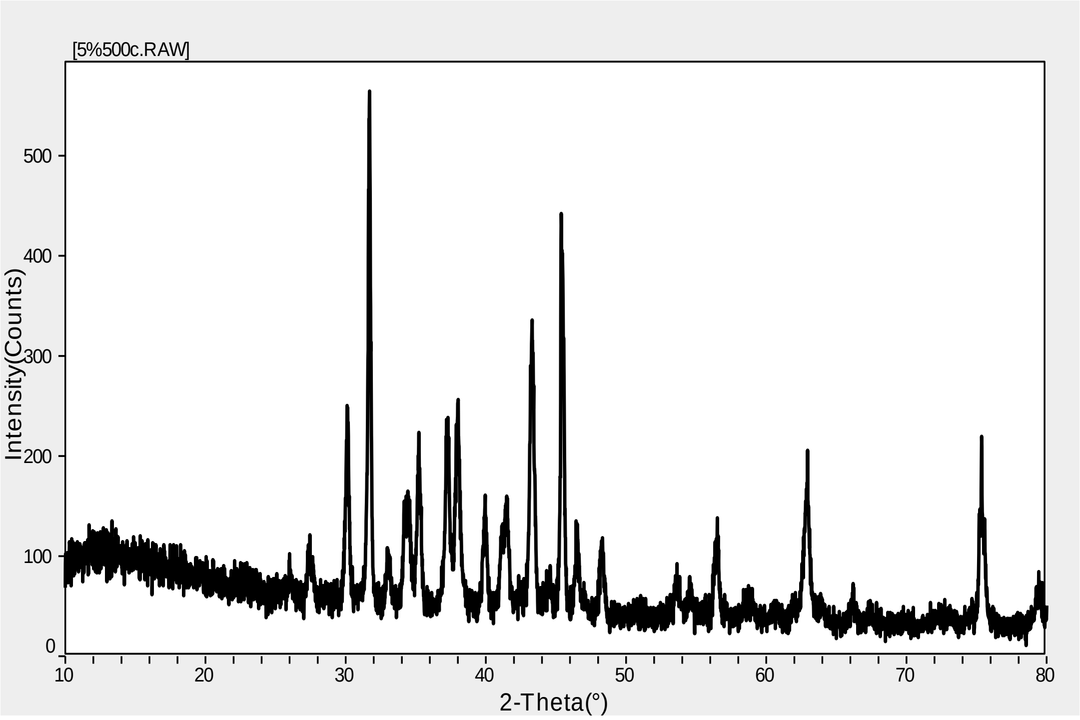


Fig (3.6) X-ray Spectra of Nickel chloride impregnated silica at 5% concentration **at 500 C calcinations temperature**

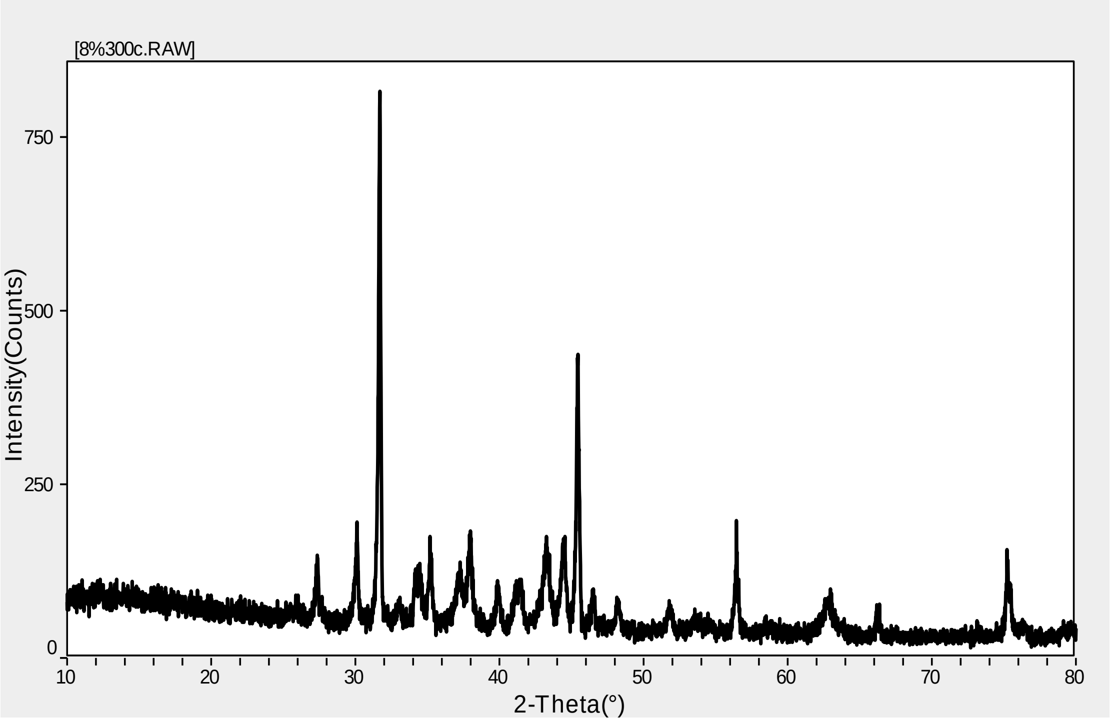


Fig (3.7) X-ray Spectra of Nickel chloride impregnated silica at 8% concentration **at 300 C calcinations temperature**

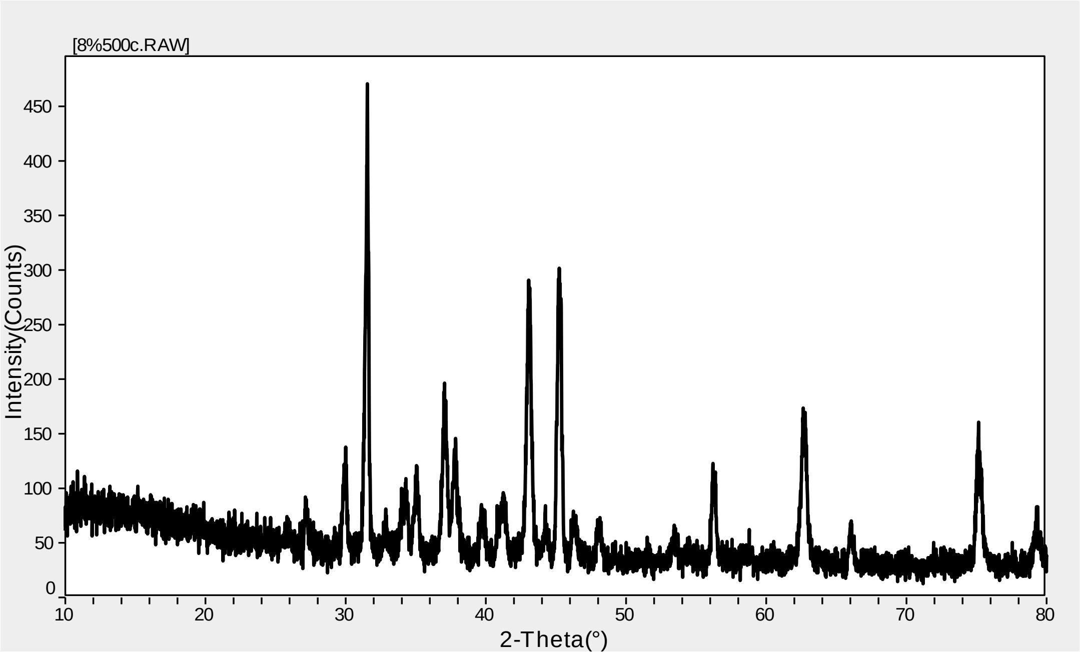


Fig (3.8) X-ray Spectra of Nickel chloride impregnated silica at 8% concentration at 500 C calcinations temperature

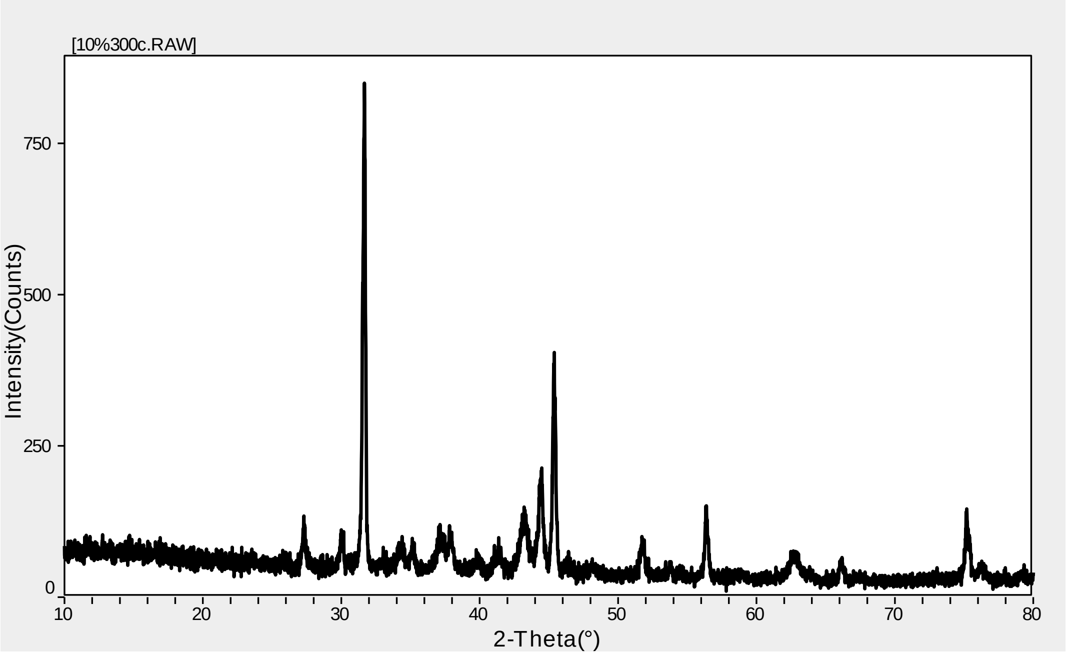


Fig (3.9) X-ray Spectra of Nickel chloride impregnated silica at 10%

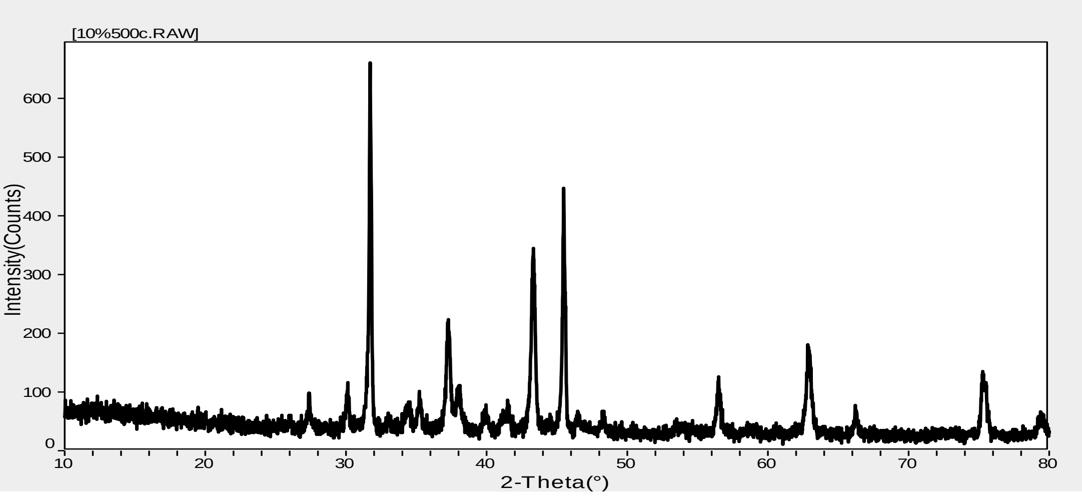


Fig (3.10) X-ray Spectra of Nickel chloride impregnated silica at 10% concentration at 50 0 C calcinations temperature

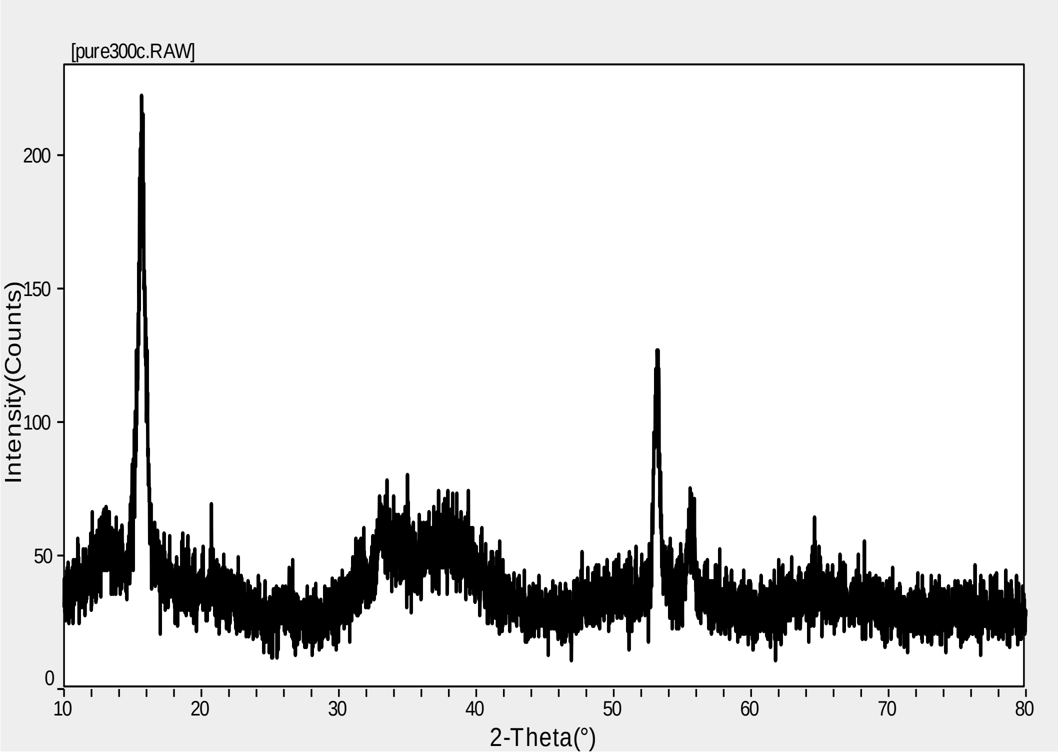


Fig (3.11) X-ray Spectra of pure silica at 300 calcinations temperature

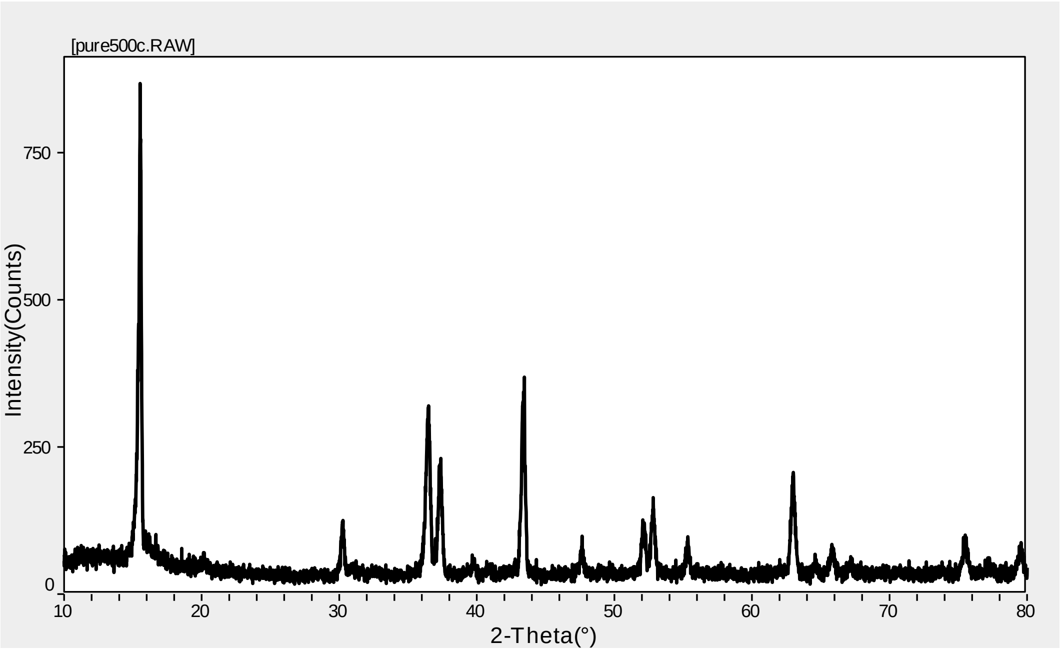


Fig (3.12) X-ray Spectra of pure silica at 500 C temperatures

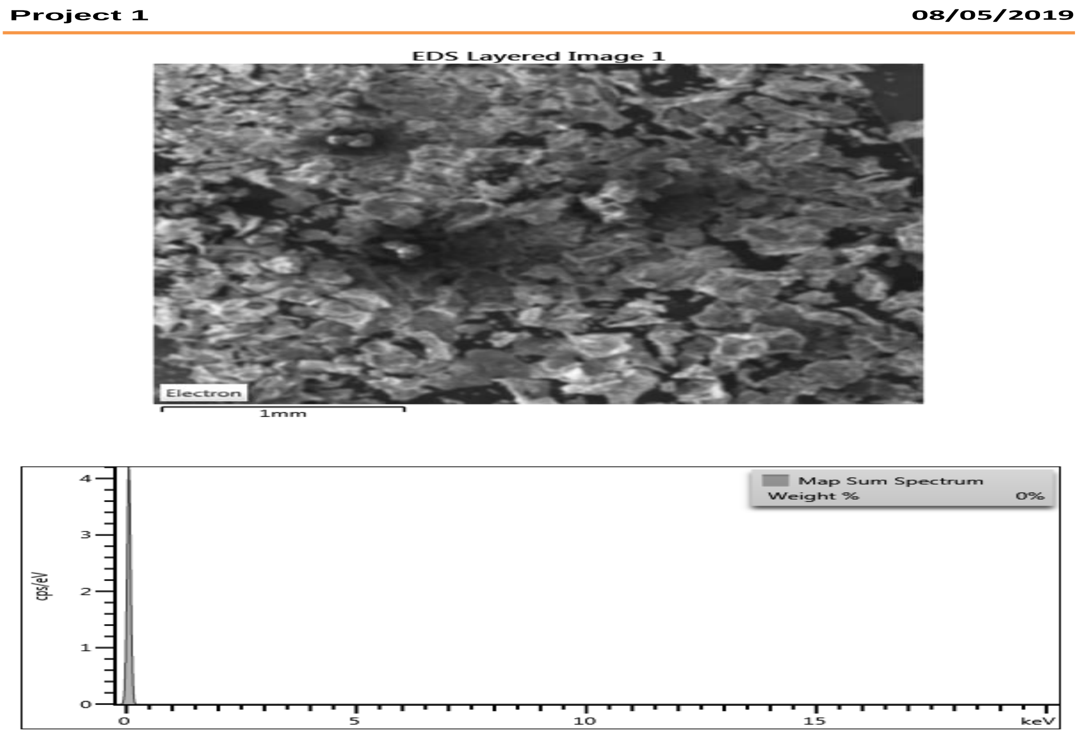


Fig (3.13) EDS and SEM analysis of nickel chloride impregnated silica at 1 % concentration at 300 C calcinations temperature

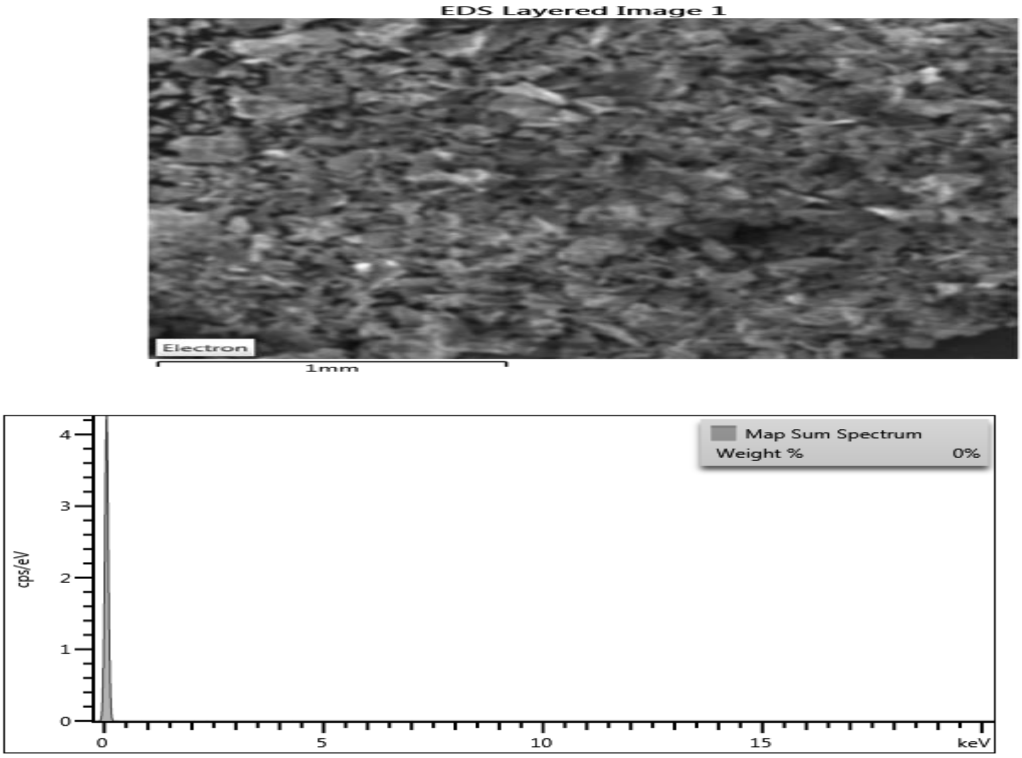


Fig (3.14) EDS and SEM analysis of nickel chloride impregnated silica at 1 % Concentration at 500 C calcinations temperature

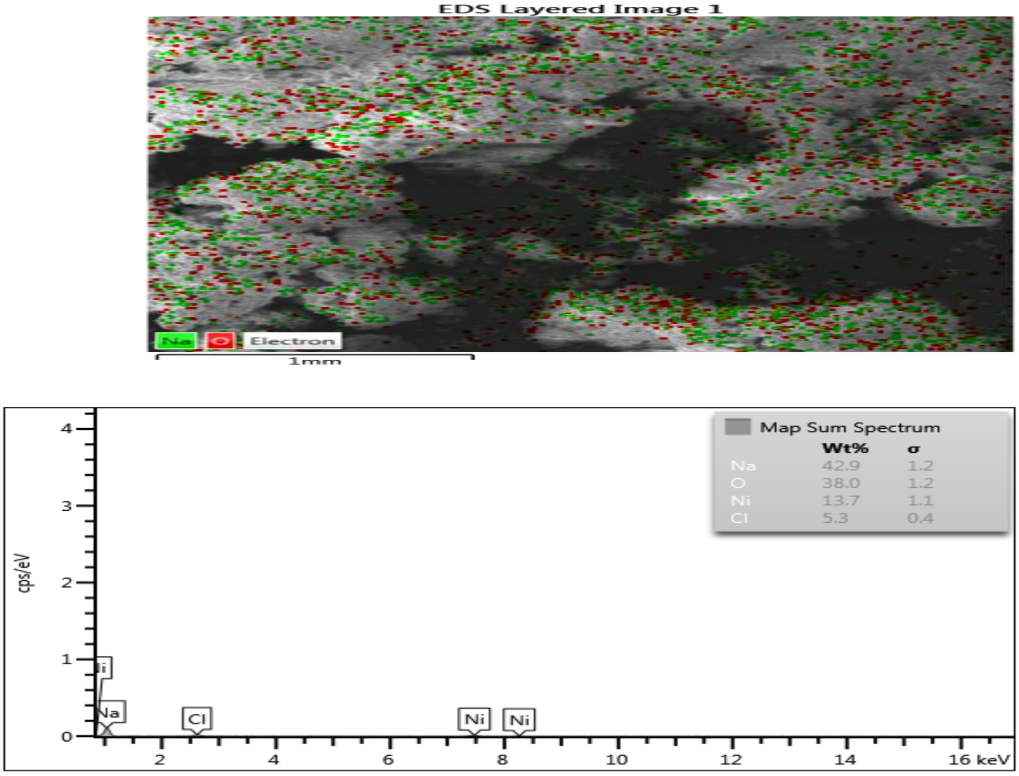


Fig (3.15) EDS and SEM analysis of nickel chloride impregnated silica at 3% Concentration at 300 C calcinations temperature

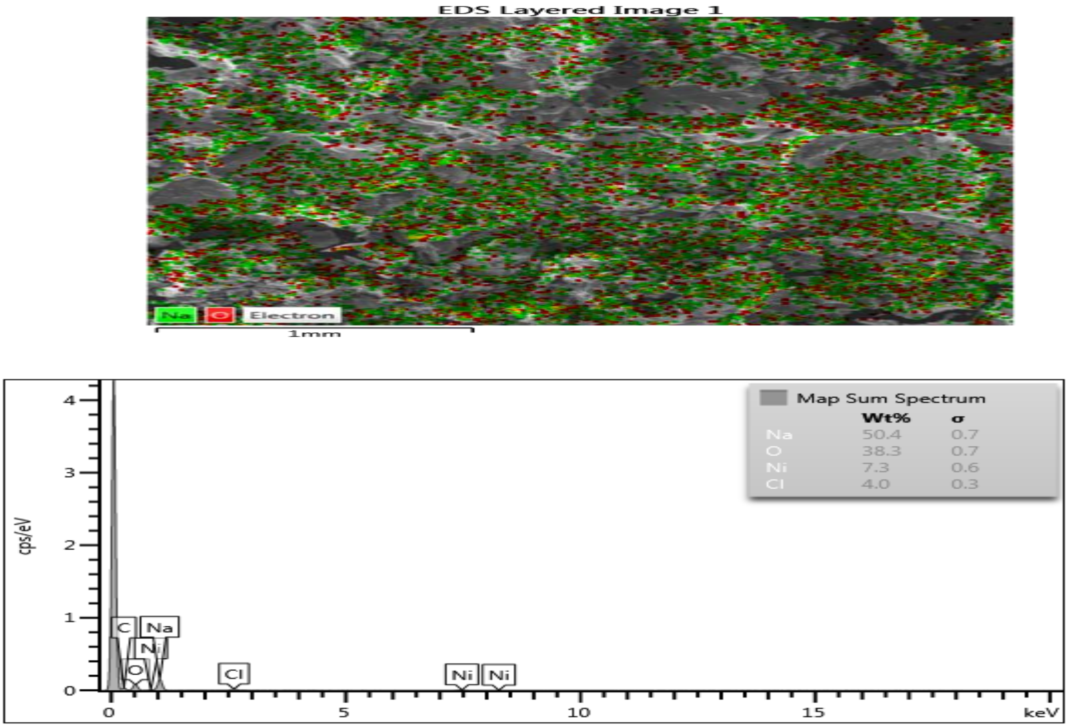


Fig (3.16) EDS and SEM analysis of nickel chloride impregnated silica at 3 % Concentration at 500 C calcinations temperature

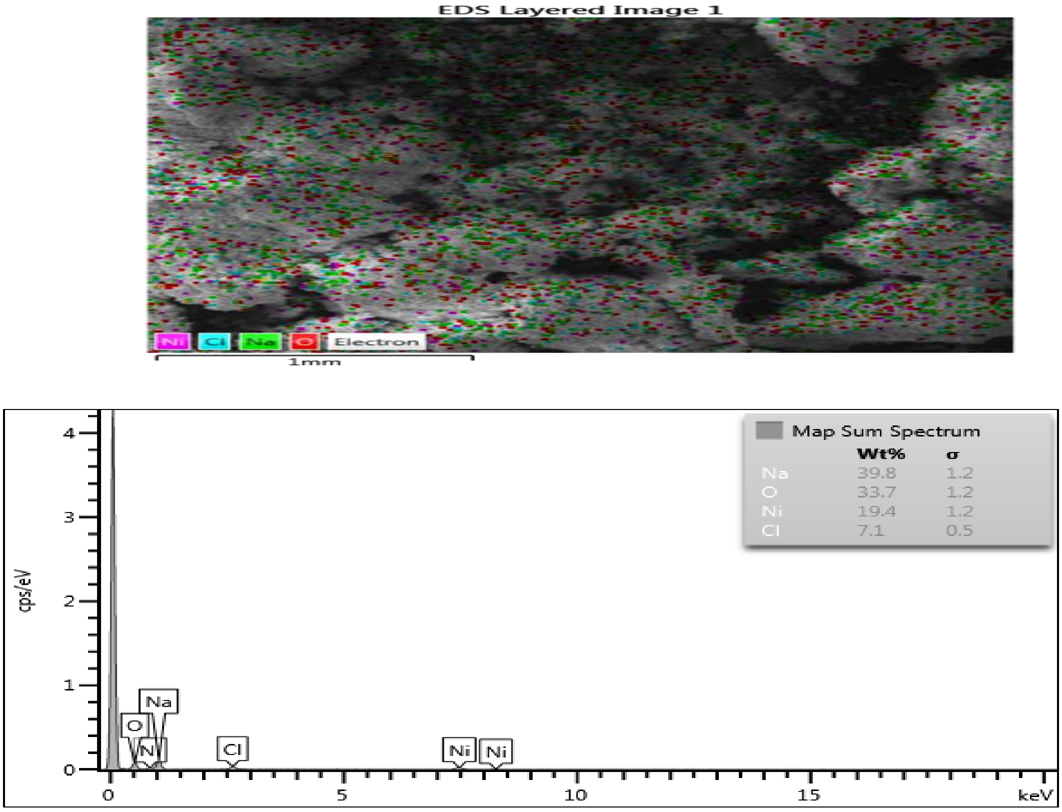


Fig (3.17) EDS and SEM analysis of nickel chloride impregnated silica at 5% Concentration at 300 C calcinations temperature

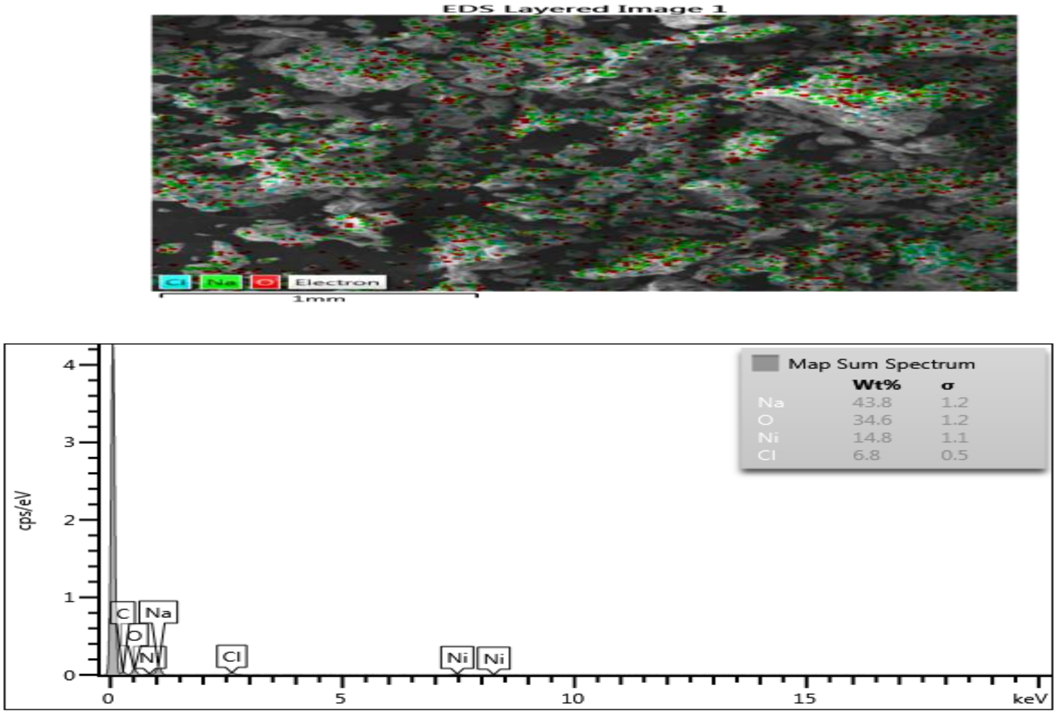


Fig (3.18) EDS and SEM analysis of nickel chloride impregnated silica at 5 % Concentration at 500 C calcinations temperature

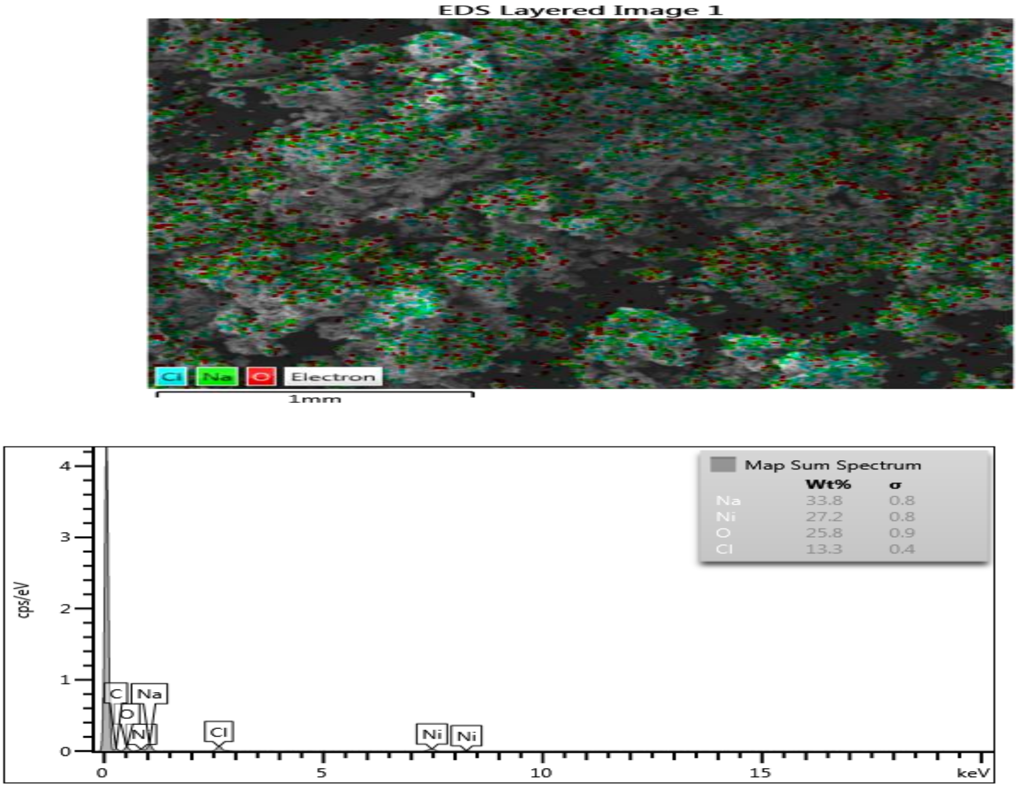


Fig (3.19) EDS and SEM analysis of nickel chloride impregnated silica at 8 % Concentration at 300 C calcinations temperature

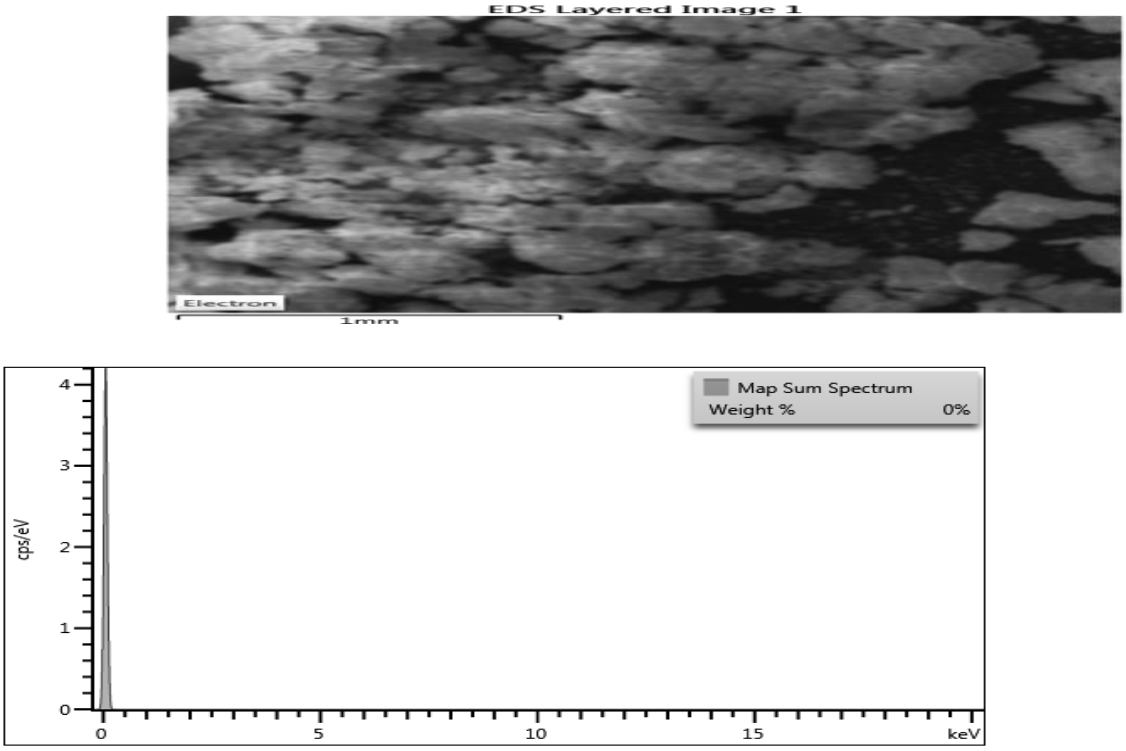


Fig (3.20) EDS and SEM analysis of nickel chloride impregnated silica at 8 % Concentration at 500 C calcinations temperature

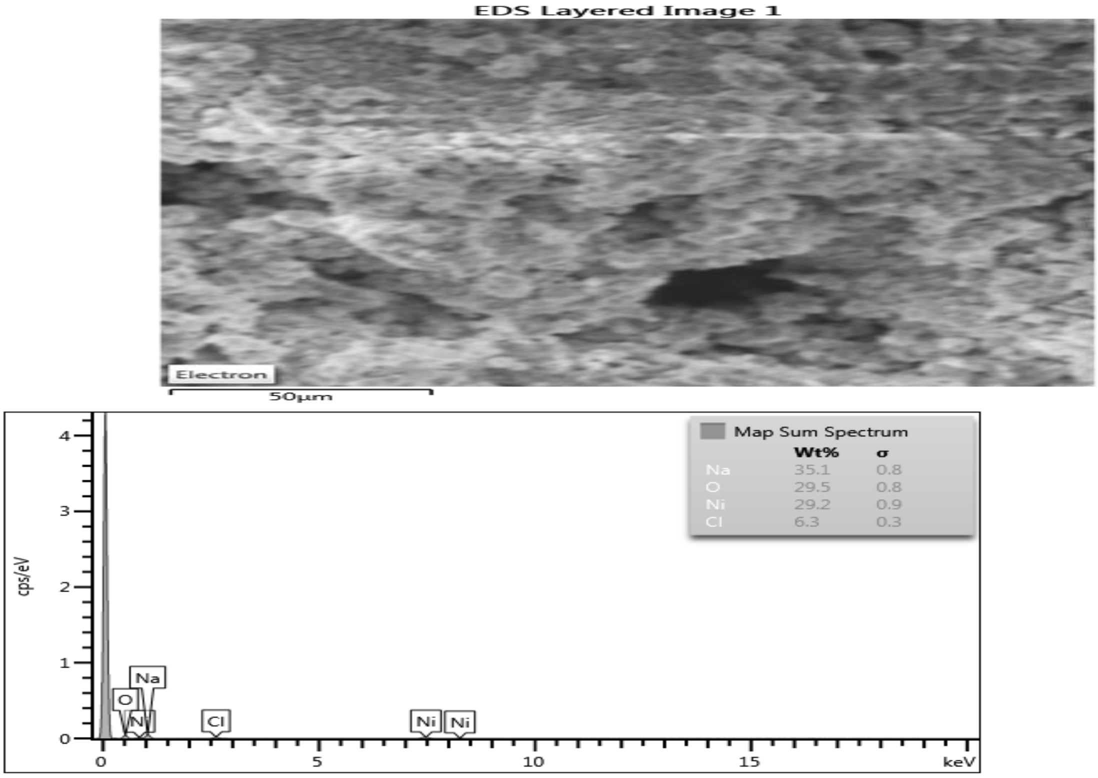


Fig (3.21) EDS and SEM analysis of nickel chloride impregnated silica at10 % concentration at 300 C calcinations temperature

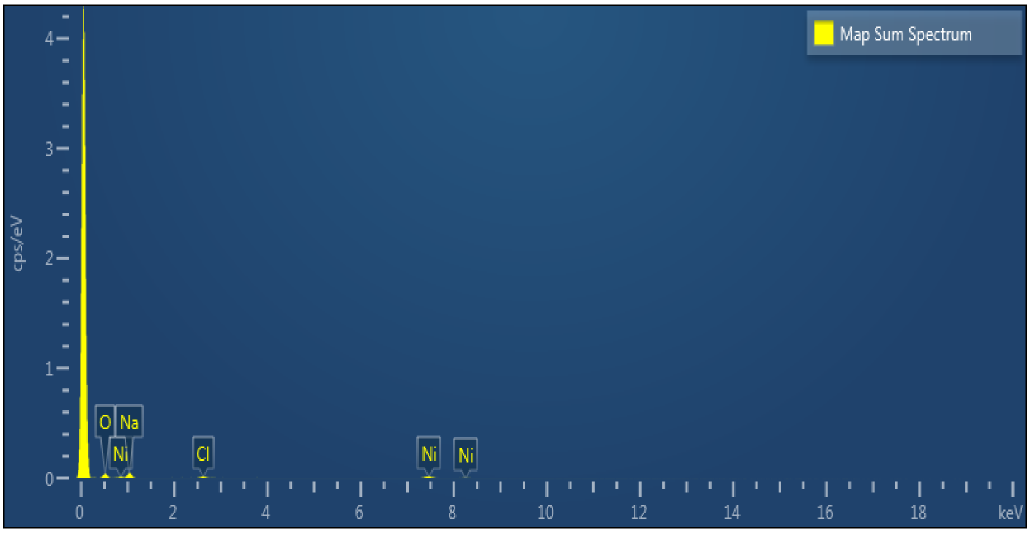
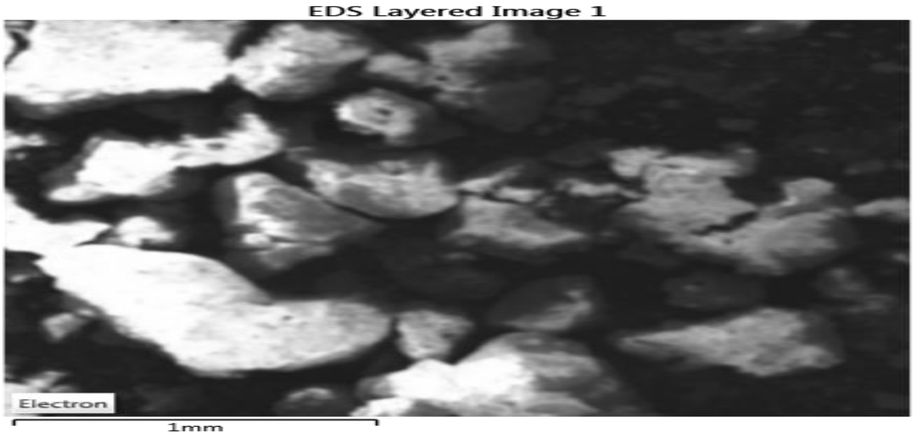
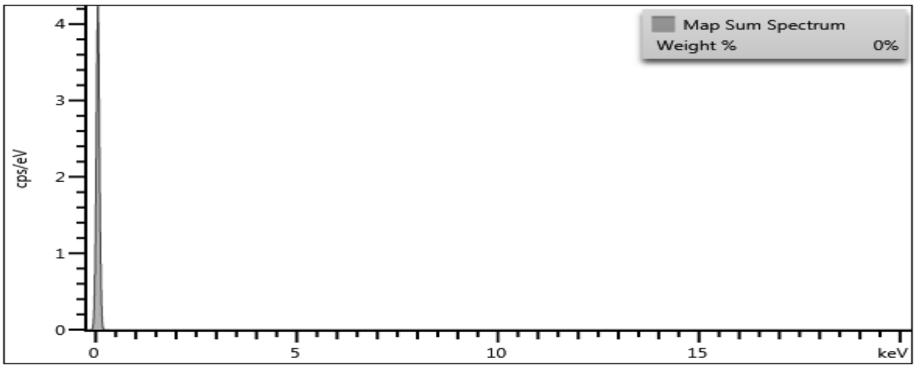


Fig (3.22) SEM analysis of nickel chloride impregnated silica at10 %Concentration at 500 C calcinations temperature





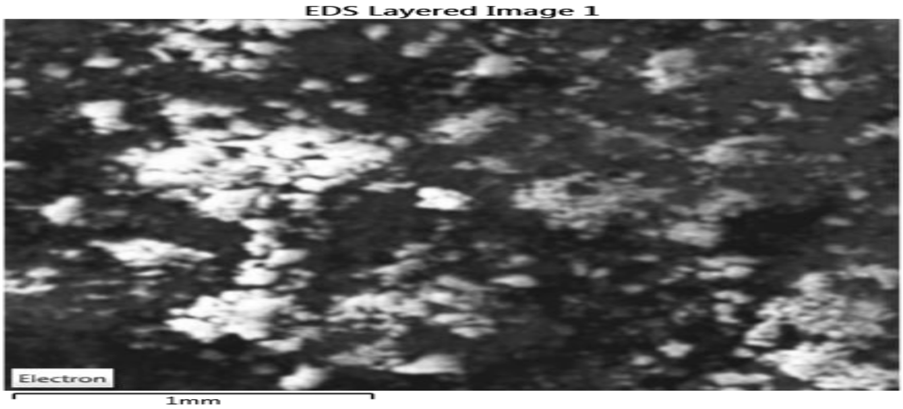


Fig (3.23) pure Silica at 300 C analysis by EDS and SEM

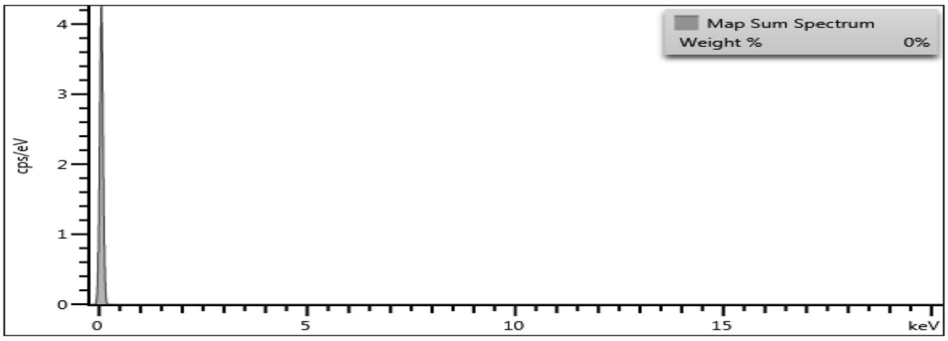


Fig (3.24) pure Silica at 500℃ analysis by EDS Scanning Electron Microscopy SEM

3.2 Results

3.2.1 Support (SiO_2)

The X-Ray diffraction analysis of Silica support calcite at 300 C and 500 C are shown in fig (3.4) for one and two samples, the spectrum obtained for Ni free SiO2 (calcite), Ni= zero. Two absorption peaks located around 20 – 10 and 24A. The EDS analysis of Silica support calcite at 300 C and 500 C are shown in Fig (3:13) and (3:14) map sum spectrum weight = 0 and other like fig (3:15), (3:16), (3:17) , (3:18) (3:19) and (3:.21) map sum spectrum weight % have .different number of Na , O , Ni , Cl but the (3: 20) ,(3:22) and (3:23) map sum spectrum weight % = 0

3.2.2 Silica Supported catalysis

The difference groups of Ni\SiO2. Catalyst series calcite at 300 C and 500 C are shown in fig (3.5) and (3.16) the samples calcite at 300 ℃ and calcite at 500 ℃ the concentration level, reproduce solely the pattern of the support (Bowker, 2002). For the series, the diffract a grows display the pattern of formation of new species (NiOCL) or (Ni (OCl) S these species are formal from NiCl2. Upon increasing to calcinations temperature to 500℃ are easily observe the development of the characterize peaks of Ni-oxide species. These peaks at20 and are more intensive and obvious with increasing concentration level from 3% to 10% Ni on support (Lines, 2008,).

3 .3 Discussion

The activity and selectivity of a metal support catalysts strongly depend upon the method of catalyst preparation employed (Bowker,2002,.). In the present investigation the nature by XRD spectroscopy at every elementary step of Ni\Si$O_2$, catalyst preparation by impregnation method.

The investigation has concerned the nature of surface complexes produced upon impregnation, and calcinations, in relation to the active phase after reduction. The XRD results allowed us to distinguish two consecutive steps during the treatment of Ni|SiO2 catalyst. In the first step, and 110 C to 300 C the continuous increase in Ni – O and NiCl coordination suggest Nickel (NiOCL) s and the progressive formation of hydroxyls chlorate complex such as (Ni (OH) C l). Also in this step oxide of Nickel were absent in XRD spectrum, Nickel| SiO2,at 300C figure(3.16) at all concentration level ,the NiO phase could not be detected by XRD. The spectra of unreduced catalyst appear to depend in calcinations temperature (Freel, 1997.).

This result is in agreement with earlier work on silica supported catalyst . The band at (130) and 2 ∅ may be ascribed to be Nickel surface complex.

In the second step the temperature was raised from 300℃ to 500℃, the absorption band obviously shifted to transformation process leading to the formation of a new species (Ni(11) or NiO species phases are present in Silica surface. The spectra of unreduced catalyst appear to depend on the calcinations temperature. This result is in agreement with earlier work on silica supported catalysts (Fujitani, 2000). As the concentration increase most of the additional Ni form the oxide phase are increase proportional with increase on metal loading. Moreover, indications are seen on the figs, for the formation of Ni –Silica phases.

Most states reporting elimination of Cl from Ni|Al2O3 or Ni|SiO2 catalysts indicate that water plays a role in the activation fragments.

The surface structure of silica Gel is widely accepted as consisting of hydroxyl (Sileanal) group and oxide (Silo one) linkages. Due to the interaction between Silica and water, there is conversion of Sloane to Solana surface sites. The alumina surface also has OH group but these less reactive Those of Silica , towards certain. The iodide point of alumina is 7.4

,where as this value is 3.1 for silica , therefore the silica surface becomes more negative charged that alumina at alkaline PH have studied the EXAFS of Ni\Al2O3 catalysts made by the impregnation methods, reported that NiO is formed by calcinations for the sample with high Ni concentration.8% and 10% the colure in different concentration level like 1%, 3% 5%To change brown, red, blue and green look figures (3:13), (3:14), (3:15), (3:16), (3:17), (3; 18),(3:19), (3:20), (3:21) (3;22) and(3:23)The figure at 500 C Number is equal 20 but 5% at500 the intensity equal 700 the peak number is equal 17The results of Fig (3.7) and Fig (3.8) x –ray spectra of nick Show figures (3.1) and (3.2) x –ray spectra of nickel chloride

Impregnated silica at 1% concentration at 300 the intensity equal 350 the peak Number is equal 22 but 1 % at500 the intensity equal 300 the peak number equal 18 The results of Fig (3.3) and Fig (3.4) x –ray spectra of nickel chloride Impregnated silica at 3% concentration at 300 the intensity equal 440 the peak Number is equal 19 but 3 % at 500 the intensity equal 400 the peak number is equal 16

The results of Fig (3.5) and Fig (3.6) x –ray spectra of nickel chloride impregnated silica at 5% concentration at 300 the intensity equal 720 the pea el chloride impregnated silica at 8% concentration at 300 the intensity equal 900 the peak number is equal 19 but 8% at500 the intensity equal 480 the peak Number is 16equal The results of Fig (3.11) and Fig (3.12) x –ray spectra of nickel Nickel oxide is formed chloride (Barthoco,2019).

Impregnated silica at 10% concentration at 300 the intensity equal 950 the peak Number is equal 14 but 10 % at500 the intensity equal 750 the peak Number is equal 11 the number of peaks to be more than number of peaks at 300℃. The number of peaks increasing to as calcinations temperature Decreasing. But the number of peaks decreasing as increase temperature Increasing. The intensity increasing at low temperature or at 300℃ and Decrease at high temperature or at 500 C Also at high concentration level. Silica supported.

Nickel oxide NiO nano-particles were produced via rapid microwave Method. NiCl2 precise was obtained by slow drop wise addition, the resultedInformation of NiO nano-powder mean dimension of this powder was 30 nm according to the image analyzed by scanning electron microscope (S EM) the Morphology observed in the sample not showing any hard grains which gives the idea that size of the particle is small and has a sinle phase.

X –ray diffraction (X R D) pattern revealed well–crystallized high purity nao structure of the syntheses powder. Microwave utilization increased

Homogeneity and decreased the mean particle size of the produced NiO powder The morphology and structure of the nickel oxide ,NiO were investigated by scanning electron microscopy (SEM) and energy ray disuses' x-ray spectroscopy (EDS). The results of scanning electron microscopy (SEM) show the fig(3.23) and fig(3.24) pure silica at 300℃ and 500 C the ship of NiO in the flower like areas and with increasing loading levels while a results of SEM images fig(3.17) and fig(3.18) displayed the NiO dominantates in the flower like areas and increase in peaks with increasing concentration. The Scanning Electron Microscope and Energy dispersive X –ray Spectrometry (SEM, EDS) the result is different. Different load $NiCl_2$ impregnation SiO_2 (1 %, 3%, 5 % 8 % and 10% respectively) in air at 110, 300, and 500 C . The nature of surface species upon drying, and thermal treatment of $NiCl_2$ Use different concentration of nickel chloride supported silica and calcinations at 300 C and 500 C were characterized using different analytical techniques such as X-Ray diffraction (X R D), Scanning Electron microscope (SEM) and Energy dispersive X-ray (EDX). The result obtained showed that there is a great difference in the microstructure of the catalyst in order of the different load applied e.g. XRD (fig 3.11) and Fig (3.12) for the pure silica calcinations at 300 C and500 C showed the characteristic peak of silica is different in fig (3.11) is ∅equal 15 °, 22, ° 55 °, 58 ° the observed as (260), (110), (80) and (65). but in the fig (3.12) peaks ∅ is equal 15 ° .28 °, 35 °, 38 ° ,44, ° 55 ° ,65 ° 75.° and.80° there are observed as (780) (98) (280) (250) (400)(80) (110)and (84) .°

While in concentration of silica supported nickel at 1%, 3%, 5%, 8% and 10% nickel chloride impregnated silica at 300^0Cand500^0C the back scattering of this peak, or distention appeared as the concentration increased and anew peak was appeared such as peak at figs (3.1) (3.2), (3.3),(3.4) (3.5) (3.6) (3.7) (3.8) (3.9) and (3.10).e.g. The peak at 37. Shift to 42, the nickel plane appear at. For example fig(3.7) and fig(3.8) for the nickel chloride support silica 8% and calcinations at 300 C and500 C showed the characteristic peak of nickel is different in fig (3.7) ∅ is equal 25 , ° 30 , ° .35 °, 45 ° 55 °, 65 ° and 75 °. The observed as (125), (180), (780),(500),(240)(100) and (170). In fig (3.8) ∅ is equal 28.° 30.° .33 °, 38 , ° 40 °, 42 , °,46 °,50 °,65 ° ,75 °,and 80.° The observed as (100), (140), (450),(200),(180)(245) (220)(190) (184) and (90).

But for other technique (SEM) in the fig (3.23) and Fig (3.24) for the silica pure calcinations at 300 C and500 C showed the characteristic. The map like flower ,shape and color is white and born While in concentration of silica supported nickel at 1%, 3%, 5%, 8% and 10% nickel chloride impregnated silica at 300 C and500 C the new phase was appeared and dominated as the concentration increased for increase concentration loading level. And the shape of maps changes to be black, born, red and green. The results indicate that following (the drying step and calcinations 300 C and 500 C) .in air the Nickel occurs in many detectable forms. $NiCl_2$ particle and Ni – O surfaces. The calcinations appears to transform the $NiCl_2$ particles into the (Ni (OH) CL) s, (NiOCL) s and NiO surface species

Appearance of some particle is in spherical shape and some are rode shape, it is observed the particle are highly applemerated and they areEssentially particle, nanoparticles the SEM picture indicates the Size of Polycrystalline particles, the observation of some larger nanoparticle may be Attitude to the fact that NiO nanoparticles have tendency to applomerate due to their high surface energy and high surface tension of ultrafine vnanoparticles the fine particle size result in a large surface area that in Nan particles catalytic activity so we can conclude that the prepared nickel oxide NiO particles are in nanometer range. The average diameter of theParticles observed from SEM analysis are 97 nm. Which is large than the diameter particles from X-ray diffraction(X R D). (Barnes2021,catl 11,324).

3.4 Conclusions

From the presented and discussed results, the following conclusions can be drawn.

Nickel nanoparticles Synthesized using different method, involves characterization of silica Supported Nickel oxide catalyst using X-Ray diffraction. The Study aimed to know the affect of the loading level and calcinations temperatures. The catalyst was prepared by impregnation of silica with aqueous solution of Nickel chloride to from past. Then the past was dried at 110 C for 17hrs the dried past Clacined at 300 C and 500 C for 5 hrs. The prepared products were characterized by X- Ray diffraction. The results of this analysis indicated that increasing of loading level and calcinations temperature appears to transform the NiCl2 particles into the (Ni (OH)Cl)s, (NiOCl)s and NiO Surface species.

3.5 Recommendation

- To use electron microscope Spectra (E M S) in characterization
- To use another Carrier materials for example Alumina
- To use this catalyst in to reactions like oxidation redaction reaction

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