# Synthesis of Iron Molybdate and Evaluation of the Methods (Review)

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Abstract:- Iron molybdate is a powerful catalyst usually used in an unsupported form and operating in an oxygen-rich atmosphere. Catalysts of iron molybdate have been used for many years for the industrial development of formaldehyde from methanol oxidation. . This paper examines the different methods used in preparing and synthetics of iron molybdate, and the differences, advantages and disadvantages of each method.

*Keywords:*- Iron molybdate, Methods, advantages and disadvantages.

#### I. INTRODUCTION

A material which increases the rate of a chemical reaction without undergoing any permanent chemical alteration on its own is known as a catalyst (Oxford dictionary), it means that another material which is itself not absorbed in the process raises the rate of a chemical reaction. By lowering the activation energy against target products without ingestion, it increases the rate of reactions. The catalyst is a kinetic effect that offers an alternate chemical mechanism by which reactants at moderate conditions are converted to products. This allows the use of lower temperatures to save electricity and building materials while reducing undesired product creation (Farrauto 2017). Figure 1. Provides a comparison of the diagrams of the reaction coordinatesor energy diagrams for catalyzed and uncatalyzed alkene hydrogenation.

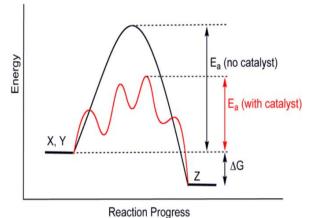


Fig. 1 by reducing the activation energy needed for the reaction to proceed, catalysis (a) speeds up a reaction (boundless)

Catalysts can be classified into two main groups, homogeneous and heterogeneous. The catalyst is in the same step as the reactants in a homogenous catalytic reaction.Usually, either in a single liquid phase or gas phase, both reactants and catalysts are. In the liquid form, the bulk of industrial homogeneous catalytic processes are carried out. Any of the essential processes are ester hydrolysis involving general acid-base catalysts, polyethylene processing with organometallic catalysts and enzyme catalyst processes, Examples of homogeneous catalytic manufacturing processes (NPTEL).In order to achieve high conversions, many homogeneous catalyst methods are highly selective and require relatively low temperatures (<200 °C). Owing to the same phase design of the catalyst and reactants, the key difficulties of homogeneous catalysts are isolation, which also suggests that distillations and expensive separation methods need to be used to eliminate the catalyst that might be destroyed in the process (Fadhel et al., 2010).

Typically, heterogeneous catalysis consists of a solid catalyst in either a liquid or gas level with the reactants. The reactants are adsorbed to the catalyst surface that binds to the catalyst.Subsequent desorption of the compounds then promotes the regeneration of the active site, active site or catalyst sites where the reaction occurs.In contrast with homogeneous catalysts, high conversions are normally performed at either higher temperatures or pressures due to the reduced concentration of active sites revealed as part of the active surface. Easy separation and reuse and stabilization are the key advantages of heterogeneous catalytic methods, which is why most industrial catalytic processes are carried out using a heterogeneous method (Ali et al., 2014).

According to researches, iron molybdate Fe2 (MoO4)3 are the most studied molybdenum (Oudghiri-Hassani, 2015). Because they are strong catalysts in general, used in an unsupported form and that work in a high oxygen environment (Yeo et al., 2016). As a catalyst for formaldehyde synthesis from methanol oxidation, iron molybdate has significant industrial applications or in the propylene oxidation phase to obtain propylene oxide (House et al., 2007). Iron molybdate has also drawn interest in alloy creation as a precursor. As an electro-catalyst for the alkaline solution evolution of O2 (Chychko et al., 2011). On iron, molybdate also deal with its application as a cathode for a rechargeable sodium ion battery or for H2S gas sensing (Sun et al., 2012). In addition, studies have shown that iron

molybdate has a promising efficacy when it used, as a photo catalysts in removing Endosulfan, Rhodamine and heptachlor dye, and its efficiency was very promising (Parveen et al., 2020).

Iron molybdate is a standard two-component selective oxidation catalyst. Iron (III) oxide and molybdenum (VI) oxide are two elements that combine to create a catalyst that supports the target product. Iron (III) oxide itself is nonselective, output water and carbon dioxide. Molybdenum trioxide is selective but poor in action (Bowker, et al. 2002). The most common sources used in the preparation of iron molybdate are iron nitrate Nano-hydrates and iron oxides as a source of iron and ammonium hepta hydrate tetrahydrateand aMolybdenum (VI) Oxides a source of molybdenum (Table 1.) because it is highly soluble and relatively cheaper, and does not contain of known catalyst poisons such as chlorine or sodium (Beale et al., 2009).

Iron precursor	Molybdenum precursor	methods	Reference
Iron(ii) sulfateFeSO4.7H2O	ammonium heptamolybdate(NH4)6Mo7O24.4H2O	Precipitation	(Nikolenko et al., 2018)
Iron(III) nitrate, or ferric nitrate, Fe(NO3)3 9H2O,	ammonium heptamolybdate	Precipitation	(Nikolenko et al., 2018)
Iron (III) nitrate nonahydrate, Fe(NO3)3 9H2O,	Ammonium molybdate, (NH4)6Mo7O24.4H2O	Precipitation	(Hummadi et al., 2009).
Iron (III) nitrate nonahydrate, Fe(NO3)3 9H2O,	Ammonium molybdate, (NH4)6Mo7O24.4H2O	evaporation	(Hummadi et al., 2009).
iron nitrate	ammonium molybdate	precipitation	(Lu et al., 2016).
Fe2O3	MoO3	precipitation	(Bowker et al., 2002)
iron nitrate Fe(NO3),.9H,O	ammonium molybdate (NH4)6 Mo7O24. 4H2o	evaporation	(Li & Huang 1999).
Fe2O3	MoO3	hydrothermal	(Gaur at al., 2020).
iron nitrate Fe(NO3)3,9H2O,	(NH4)6Mo7O24,4H2O And H <sub>2</sub> C <sub>2</sub> O <sub>4,2</sub> H <sub>2</sub> O	thermal decomposition	(Oudghiri-Hassani2015).
Fe(NO3)3·9H2O	(NH4)6Mo7O24·4H2O	thermal	(Shaheen, 2007).
Fe(NO3)3·9H2O	(NH4)6Mo7O24,4H2O and oxalic acid H2C2O4	precipitation	(Yeo et al., 2016).
iron nitrate	ammonium molybdate	precipitation	(Parveen et al., 2020)
Fe(NO3)3 .9H2O	NH4)6Mo7O24.4H2O	Precipitation	(Hassan & Mitchell 2010).
Fe(NO3)3 .9H2O	(NH4)6Mo7O24.4H2O	precipitation	(Hassan & Mitchell 2010).
Fe(NO3)3.9H2O	(NH4)6Mo7 O24	precipitation	(Soares et al., 2001).
Fe(NO3)3.9H2O	(NH4)4Mo7O24.7H2O	hydrothermal	(Beale et al., 2009).
Fe(NO3)3.9H2O	NH4)6Mo7O4.	hydrothermal	(Zhang et al., 2010)
acetylacetonate, C15H21FeO6	dioxomolybdenum, (C10H16MoO6),	solvothermal	(Kersen & Keiski 2009).
Fe(NO3)3.9H20	NH4)eMo702	precipitation	Soares et al., 1997
Fe(NO3)3.9H20 or FeCI3	NH4)eMo702	Sol-gel	Soares et al., 1997

# II. METHODS OF PREPARATION

# 2.1 Precipitation

One of the most widely used methods for preparing catalysts is the Precipitation or Co-precipitation method, and it can be used to prepare single-component catalysts or mixed or supported catalysts. In other meaning, it can be used to prepare bulk and subsidized catalysts. This process is centered on alkaline solutions and a mixture of mineral salts to produce insoluble mineral hydroxide or carbonate. The precipitation process can occur by changing conditions such as the pH level Temperature, salt concentration, and evaporation. These factors lead to gradual changes in crystal growth and aggregation. In fact, a sudden change and a high concentration will result in the aggregation of large and small particles (Soares et al., 2001).

#### 2.1.1 Procedures of Precipitation Method

This approach involves different steps:

1. The step of dissolution in which the precursors of active components (Nitrate is the preferable salt to chloride or sulfate) are mostly dissolved in their salt forms first in water or in a suitable medium to create a homogeneous solution.

2. Precipitation stage in which the solution is subjected to modification of the pH or evaporation to induce precipitation of certain salts. The salts may be hydrolyzed into hydroxide forms or oxides during this precipitation, so this step may be called the hydrothermal process.

3. The stage of filtration and drying in which the solid mass is then gathered and dried gradually to about the medium's boiling point. The collected dry mass is in an irregularly formed loose state; thus, this mass is first ground to powder form. To assist binding together, a binder should be added; the binder is picked so that during calcination or activation it is melted into explosive steam vapor and carbon dioxide.

4. In order to transform the salt or hydroxide form of the active components into oxides, the calcination step is carried out by reacting with air at the required temperature, such as decomposing carbonate, acetate or nitrate into oxide (Deraz, 2018; Hummadi et al., 2009; Hassan & Mitchell 2010).

(Lu, 2016), Confirm that for the synthesis of the heterocyclic Fe2 (MoO4) 3 catalyst to activate persulfate for Rhodamine B degradation the following steps were followed: First, 5 mL of 1 M ammonium hydroxide was applied to the 100 mL 0.005 M ammonium molybdate solution, and then iron titrate (50 mL, 0.0465 M) was added to the mixed solution under intense agitation at room temperature ( $25 \degree$  C) for 20 minutes, and the solution continued to be vigorously agitated for 2 hours and centrifuged for 20 minutes. The extracted precipitate was washed several times with double-distilled water and then dried for 12 h at 80 ° C. Finally, the dry solid was calcinated in the ambient air for 2 h at 550 ° C ( $2\degree$  C / min rate).

#### 2.1.2. Advantage and disadvantage

The precipitation method has several advantages, including that the reaction temperature is relatively low, the precision of particle size is uniform, the agglomerated particles is weak, the uniformity of the components distribution as well as their low cost, its cost is low. In terms of reaction movement, it is not controlled. The reaction conditions strongly influence the solid phase nucleation and growth processes that depend on the precipitation reactions. Particle morphology of synthesized solids is uncontrolled and has a wide particle size distribution and metal ions do not fully precipitate and agglomerate (Deraz 2018).

# **2.2. Solvothermal synthesis**

Solvothermal synthesis is commonly characterized as a chemical reaction at temperatures above the boiling point and pressures above one bar, taking place in a solvent. In Solvothermal synthesis, the medium used can range from water (hydrothermal) to alcohol or some other organic or inorganic solvent (Walton, 2002). To obtain the target substance through the specific process, the specified temperature (supercritical and subcritical) requires the required reactions (De mazeau 2008).

Whether the chemical composition of the solvent is (aqueous or non-aqueous), the word Solvothermal can be used (De mazeau 2011). In certain papers, the word "hydrothermal" is commonly used to describe all forms of synthesis that occur in a closed vessel with regulated temperature and pressure (Nunes et al., 2019). The breakdown of the chemical reagents and the processing of the products by crystallization are enabled by temperature and pressure conditions. A one-step reaction route to complex materials is given by this technique. The use of solvents also has a high degree of diffusivity, which increases the stability of dissolved ions and facilitates the mixing of reagents (Yoshimura & Byrappa 2008).

Aqueous solutions of simple salts such as nitrates, acetates or metal chlorides are also precursors used in the synthesis of inorganic compounds. This method takes advantage of a change in the basic properties of water and other solvents used, by increasing the pressure and temperature. Important properties such as ionic product, density, thermal conductivity, viscosity, heat capacity, and dielectric constant are all highly dependent on both pressure and temperature, and by adjusting the synthesis parameters, specific Solvent properties can be obtained 'Many of the properties such as density, viscosity, ionic product, thermal conductivity, thermal capacitance and dielectric constant depend heavily on temperature and pressure. The specific properties of the solvents can be obtained by adjusting the synthesis factors (Nunes et al., 2019).

Solvothermal synthesis is normally carried out in an autoclave reaction vessel. The autoclaves are composed of a heavy metal, such as steel, to withstand the strain produced during the reaction. In recent years, thermal-solvent synthesis with the help of microwave radiation has been increasingly used to develop minerals (Gonçalves et al., 2016). Compared to traditional Solvothermal synthesis, solvothermal synthesis aided by microwave radiation offers certain benefits. In a very short period, it makes a full reaction (only a few minutes are required for certain nanostructures). Since microwave, radiation specifically converts energy into the reactive species found in the solution, supporting transformations that cannot be accomplished with traditional solvothermal heating (Pimentel at el., 2016).

#### 2.2.1. Principal parameters for solvothermal reactions

The parameters of thermodynamics, the parameters of chemistry. These factors are pressure, temperature, and reaction time. The (T <400  $^{\circ}$  C) is the temperate temperature for the development of dissolved thermal reactions. In the main cases, pressure and temperature improve solubility and increase these factors lead to enhanced concentration of precursors in the solvent and then the growth process (Demazeau, 2008).Two related parameters can be taken into account: nature of the solvent and the nature of the reagents. It is important to appropriate the chemical structure of the precursors to that of the target materials. Furthermore, the precursor concentration seems to play a role in regulating the structure of Nano crystallites arising from the solventermal phase (Wang et al., 2006).

The physico-chemical properties of the solvent chosen can play a significant role in the structural shape of the final material orientation.During solvothermal reactions, the reaction processes caused are depending on the physicochemical properties of the solvent (Demazeau, 2008).In solvothermal processes, the pH value may have different functions for: Preparation of complex components, elaboration of nano-composites and stability of various structural forms, regulation of crystallite size and morphology: by adjusting the decomposition of one reactant and by altering the additive's ionization equilibrium (Demazeau, 2011).

(Kersen & Keiski 2009), underlined to produce b-FeMoO4 particles by solvothermal chemistry, Bis (acetylacetonate) dioxomolybdenum (C10H16MoO6) and Iron acetylacetonate, (C15H21FeO6) are used. The ingredients are mixed and the mixture is placed in an autoclave and it is heated to 270 ° C under pressure from 90 to 100 bar for a period of 2 hours. Under the nitrogen flow, the contents inside the autoclave are cooled to room temperature. Then, at 500 ° C, the contents particles are heated, resulting in the formation of Fe2 (MoO4)3 with the required atomic ratio of Mo / Fe. All heat treatment takes place within 2 hours.

Several studies have been conducted according to this method (Gaur et al., 2020; Parveen et al., 2020; Bowker et al., 2015; Beale et al., 2009). In addition to that, some studies have used Oxalate (Oudghiri-Hassani 2015; Yeo et al., 2016; Oudghiri-Hassani 2015). Oxalic acid plays two roles: as an iron and molybdenum complexing ligand and as a nitrate anion and molybdenum reduction.

(Kersen & Keiski 2009), underlined to produce b-FeMoO4 particles by solvothermal chemistry, Bis (acetylacetonate) dioxomolybdenum (C10H16MoO6) and Iron acetylacetonate, (C15H21FeO6) are used. The ingredients are mixed and the mixture is placed in an autoclave and it is heated to 270 °C under pressure from 90 to 100 bar for a period of 2 hours. Under the nitrogen flow, the contents inside the autoclave are cooled to room temperature. Then, at 500 °C, the contents particles are heated, resulting in the formation of Fe2 (MoO4)3 with the required atomic ratio of Mo / Fe. All heat treatment takes place within 2 hours.

# 2.2.2. Advantage and disadvantage

The advantages of this method is easy and precise, shape, size, and distribution, crystalline of final product through adjusting the parameters. But it needs expensive autoclaves, during reaction process it have safety problem and it is impossible to observe reaction process.

# 2.3.1. Impregnation Method

This is the process by which a certain amount of solution containing an active phase precursor is touched by a solid (support or other active solid phase) which is then dried to eliminate the impregnated solvent. Thus, to prepare assisted and mixed catalysts, this process can be used (De Jong 2009). Recently, several studies have been performed

to generate selective iron molybdate catalysts by promoting MoO3 via impregnation and subsequent annealing processes on Fe2O3. Any of these have been recruited as discussed previously, model catalyst surfaces for the inquiry into the active site of iron molybdate structures (Pudge 2017).

Based on the mount of solution there are two types of this method: Incipient wetness impregnation and. Wet impregnation In the Incipient wetness impregnation method volume of a sufficiently formulated solution is equal to or marginally less than the volume of the solution, than the support's pore volume or another active solid phase. The catalyst is impregnated onto the support or other active solid phase followed by a drying and calcination process. Wet impregnation: There is an excess solution used. The solid is isolated after a certain amount of time and the remaining solvent is extracted by drying. The composition of the solution of the batch will change and the release of debris will shape a mud that makes it impossible to use the solution entirely (Deraz, 2018).

# 2.3.2. Advantage and disadvantage

This approach is simpler, cheaper, and unlocks the final property Configuration and to be pre-controllable. The form of wet impregnation achieves a great benefit as opposed to Other techniques used in catalyst preparation include the fact that it is possible to prepare a coating of active matter on the surface of the catalyst. This may be due to moving the transport of the solution from a phase of capillary motion to a much slower diffusion process. However, it is more difficult to prepare a high-concentration catalyst and to distribute the surface components of the catalyst (De Jong, 2009).

# 2.4.1 Sol Gel Method

The process of sol-gel is an excellent way to synthesize nanoparticles and nanocomposites, consisting of sol preparation, sequential gelation, and removal of solvents. The method of sol-gel illustrates the chemical transformation of a device from a liquid "sol" (mostly a colloidal particle suspension) into a step of a gelatinous network "gel" with corresponding post treatment and transfer to solid oxide content (Prasad et al., 2018). The methods of sol-gel enable a unique way of designing the catalyst, since they reflect an ab initio synthesis of the final solid from well-established molecular compounds (Soares et al., 1997). This technique helps the pore shape, porosity, composition, surface polarity and crystallinity or amorphicity of metal oxides to be predefined by sufficient reagent collection, reaction and drying conditions (Schneider & Baiker 1995). The sol-gel strategy suggested by (Soares et al. 2003) in which a combination of iron and molybdenum precursors are dissolved in solution and acidified by propanoic acid to avoid precipitation, The water is removed, which leads to the formation of a gelatinous substance and it is calcined later. The calcination conditions are very important with the molybdenum surface loss with the use of airflow due to the fluctuations of the organic molins (Soares et al., 2003). Compared to related coprecipitated cases, the samples prepared by sol-gel demonstrate lower crystallinity of MoO3. This is due to the

development of amorphous MoO3 in the lattice spacing's of Fe2 (MoO4)3 instead of the crystalline MoO3 excesses found using co-precipitation instead of forming, this was meant to improve the surface area and, by implication, the reaction detected by samples of sol-gel against partial oxidation of methanol (Pudge, 2017). Unlike the precipitated sample, the use of sol-gel synthesis limited the deactivation and actually increased the formaldehyde selectivity over time. This was assumed to be attributed to MoO3's movement to the surface from the lattice spacing's. (Soares et al., 2003).

### 2.4.2 Advantage and disadvantage

The advantage of the sol-gel approach is a simple and very inexpensive method of preparing metal oxides and allows, as opposed to other preparation methods, control over the doping phase or inclusion of transition metals. It has a low temperature sintering capability, about 200-600 ° C. the disadvantage is Shrinkage after drying of a wet gel, which also results in fracturing due to the generation of high capillary stresses and therefore making it impossible to achieve large monolithic pieces; Owing to the differing reactivity of the alcoxide precursors, the preferential precipitation of a specific oxide during sol formation (in multicomponent glasses); Challenging to avoid residual porosity and expensive technique (MODAN & PLĂIAȘU 2020).

# III. CONCLUSION

There are four types of approaches to the synthesis of iron molybdate catalyst. Each technique has different principle and advantage and disadvantage. The purpose of catalyst preparation must be specified, taking into account the broad gap between the usage at the level of the laboratory and the half- or complete industrial level.

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