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Preparation of nickel catalyst supported on alumina by impregnation were utilized in hydrogenation process

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Abstract
In this study the nickel catalyst was prepared, which had been supported on gamma alumina this characterized with high surface area. Nickel sulphate was used as a source of nickel, while the method of Impregnation adopted as the basis for the preparation. The using Impregnation route consist two-stage, because it known that the preparation of catalysts with a high content of metal in one go is not possible. In the first stage the solution is fragmented into two parts and downloads each part separately. Then the Nickel content was measured in the solution by atomic absorption device as well as the comparison was made between the prepared catalyst and commercial catalytic by scanning electron microscopy (SEM) and X-ray deflection (XRD).

Keywords: Preparation, nickel catalyst, impregnation, utilized

1. Introduction
Hydrogen now is an important alternative for the energy sources in chemistry and in industry. Usually it derived from the petroleum and natural gas [1]. then other sources found for the production of hydrogen that is fossil fuels, but when it started in the diminishing, biomass had been appeared which found more acceptability because it is inexpensive, renewable and abundant [2]. After that, reported a new method to produce the hydrogen depend on the using of glucose which could be derived from the biomass under limited conditions [3]. This process gave the glucose more interesting in that time and, considered as the only product which has a close relationship with the biomass uses [4-7]. Hydrogenation is a chemical process which consist addition of Hydrogen to other materials. This method had been used in several fields, like Oil hydrogenation and hydrogenation of Petrol [8].
Hydrogenation is an important process because it could be used to improve the petrol's residue and changed it in to an economically benefit compounds. There are two classification of Hydrogenation, distractive dehydrogenation and (non-distractive dehydrogenation [9]. Distractive dehydrogenation times called a hydrocracking, obtained by the addition of Hydrogen to the double and triple bonds which led to saturated compounds with low boiling pontes, while the non-Distractive dehydrogenation used to improve the products and to prepare some important intermediate for Industrial chemistry [10].
At present, Nickel Catalyst supported on Alumina could be used widely in Hydrogenation process [11], and it consider as the violent catalyst in most hydrogenation processes which contain converting the unsaturated alkenes to saturated compounds.
Through this study, successfully nickel catalyst which supported on gamma alumina had been prepared, using an impregnation method.

2. Experiments
2.1 Preparation of Nickel catalyst / γ alumina using acetone as a solvent
Nickel alumina catalyst with Ni loadings (20 wt%) had been prepared using impregnation method. The preparation contain, drying the Nickel sulfate (10 g) for an hour in oven, followed by dissolving it in limited amount of acetone (30 ml). The increasing of acetone could be led to loss in the loading of Nickel on the alumina. Impregnation process consist two-steps, in each step 10g of Nickel was loaded. The alumina had been impregnate in the solvent which was prepared above from dissolving Nickel sulfate in acetone for two hours with stirring, followed by filtration for alumina and leaved to drying in oven at 120 °C for three hours.

In the second step, the same operation was repeated and with the same amount of Nickel. After that the reaction mixture under go to calcination at 400 °C for two hours, cooled down the preparing catalyst and then it was subjected to a reduction process using hydrazine 30% (V/V), for 2 h followed by separating and drying.

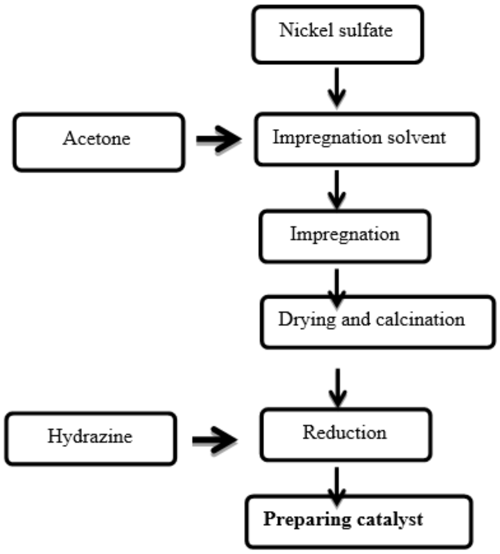


Fig 1: Diagram of steps of the catalyst preparation process

2.2 Catalyst Analysis

2.2.1. Density measurement

Alumina had been placement in a stainless steel container which has a (M*) weight. The container under go to a vibration process using a vibrator to get a homonym and be sure that had been fully, followed by a new weighed for the container and the alumina (M). After that, the volume density was calculated using the equation:

Density = (M – M*) / 100

M* = weight of empty container.

M = weight of container + weight of alumina

2.2.2. Atomic Absorption

The atomic absorption had been used to identification of nickel content which loaded on the supported catalysts in the solutions; this was resulted from the melting of nickel sulfate in acetone before and after impregnation process.

2.2.3. Catalyst Characterization

Scanning electron microscopy and X-ray diffraction had been used to determine the chemical structures (Ni loadings) of the samples, and comparing between the preparing catalysts and the commercial catalysts.

2.3. Evaluation of catalyst

Catalytic hydrogen (40 g) was loaded into stainless steel reactor (500 ml). On the other hand, p-nitrophenol 20 g was dissolved in 150 ml of ethanol then introduced in storage of p-nitrophenol as showed below. Hydrogen was passed to push the air out, followed by the heated to the required temperature. After that the p-nitrophenol was carried out and (1.6-1.7 mpa) of Hydrogen pressure was passed, cooled down and separated gas from the liquid. The liquid product analyzed by the HPLC {Agilet type) with diode array detector using ZORBAX-C18 column. This showed the percentage of conversation of p-nitrophenol to p-aminophenol.

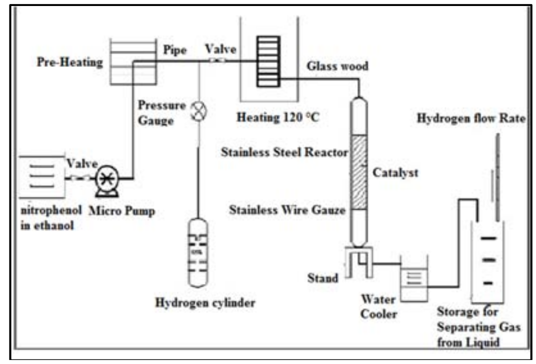


Fig 2: Drawing for evaluation of catalyst

Fig 3: HPLC Device using to evaluation of catalyst

3. Results and Discussion

Nickel catalyst, was successfully obtained through using the traditional impregnation method, but from different starting materials and conditions. This rout led to reducing the cost which is very important in industry area, moreover it depend on using available materials and mild conditions. Impregnation method had been successfully applied to loadings a Ni in 20wt % to prepare Ni/Al_2O_3 catalyst. The preparation method contains two-steps, and the required amount of Nickel sulphate which used to support of 20Wt % of Nickel on Alumina was collected through this equation:

Wt of Nickel sulfate / M.Wt =Wt of Nickel / atomic Wt

Wt of Nickel sulphate = 20 * M.Wt of Nickel sulphate / atomic Wt of Nickel

3.1. Catalyst Density

The results showed that every (9 g) of the Nickel sulfate hexa-hydrate it required to supported 20 % of Nickel on 10 g of Alumina. After catalyst preparation, the density of the alumina and the density of the catalyst were calculated and gave the following results (table 3-1):

No. of sample	Types of uses Alumina	Density (g / ml)
1	γ alumina	0.322
2	preparing catalyst	0.855

The results it showed that the density of the catalyst which was prepared is higher than the density of alumina and this refer to that the alumina had been loaded on the Nickel catalyst.

3.2 Ratio of loading Nickel Catalyst on Alumina

In the beginning and before the impregnation the amount of Nickel in the solution was calculated as a part per million using atomic absorption spectroscopy. After that, and when the impregnation had been finished the calculated of the Nickel amount in the remaining solvent was repeated again to get the following results in the table (3-2):

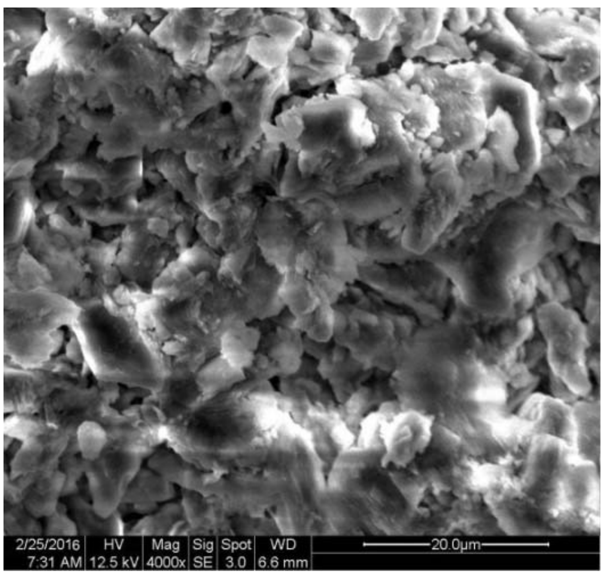
Atomic absorption results for the solvents before and after the impregnation process

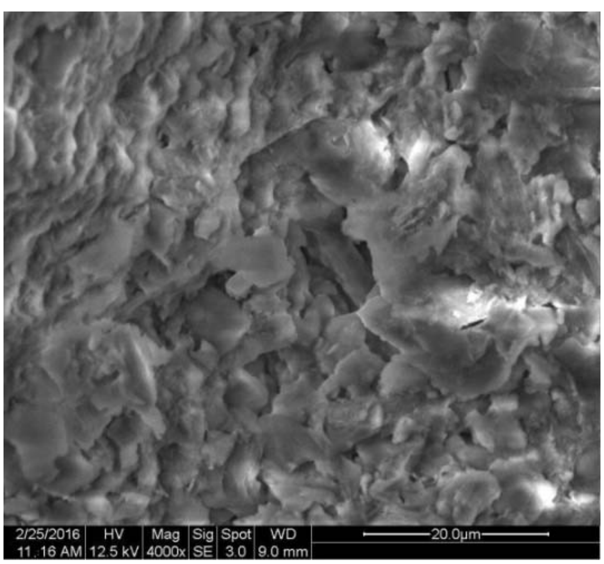
NO.	Wt. of Alumina	Wt. of nickel sulphate	Solvent	Wt. of Solvent Required	ppm of Ni before impregnation by A.A	Time of impregnation	ppm of Ni after impregnation by A.A	Time in oven	Time of calcinations 400 C˚
1 Stage	10 g	4.5 g	Acetone	50 g	23000 ppm	2.0 hours	490 ppm	3 hours	2 hours
2 Stage	10 g	4.5 g	Acetone	50 g	22600 ppm	2.0 hours	530 ppm	2 hours	2 hours
1Stage	20 g	9.0 g	Acetone	100 g	23900 ppm	2.0 hours	505 ppm	3 hours	2 hours
2Stage	20 g	9.0 g	Acetone	100 g	23980 ppm	2.0 hours	545 ppm	2 hours	2 hours

The results showed that the amount of remaining nickel in the solvent after the impregnation had reduced comparing to the used amount of nickel before the impregnation process. This refers to deposition of the nickel on the surface of alumina. In addition, the preparing catalysts were characterized by the XRD and SEM.

3.3. Scanning electron microscopy (SEM) and X-ray Diffraction (XRD)

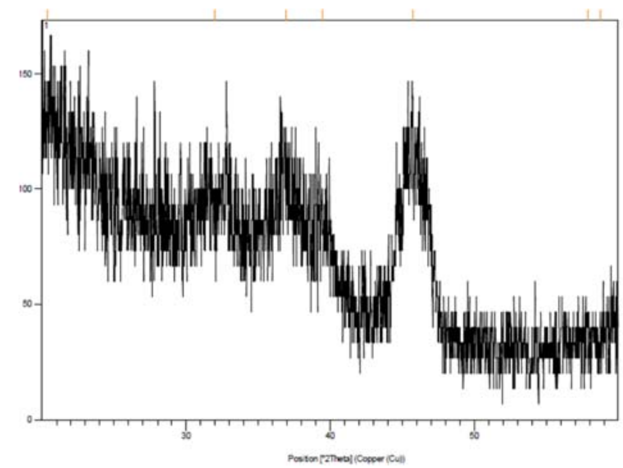
The scanning electron microscopy analysis was performed in this study, and the image of (SEM) was found that preparing Nickel Catalyst supported on Alumina was given similar spectra comparison to the commercial catalyst.

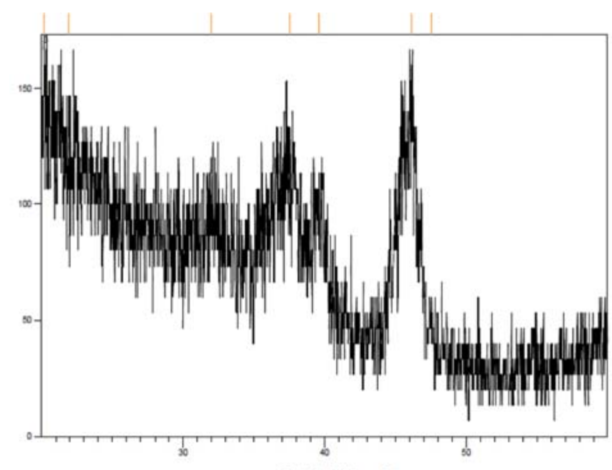




Preparing Nickel-alumina catalyst Commercial Nickel-alumina catalyst

XRD figures below showed pattern of impregnation synthesized Nickel-alumina catalyst of the sample calcined at 400 C˚ for 2 h and commercial catalyst. The spectrum of the preparing catalyst showed broad and low peaks between (30 - 34) and between (36-39), while it showed a sharp and high peak between 44-47. Same peaks could be seen clearly in the XRD figure for the commercial catalyst.





Evaluation of catalyst: in this process the results showed that the more than 98% of p-nitrophenol had been converted to the p-aminophenol and this refer to the effectiveness and selectivity of the catalytic catalyst.

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