

# **RECYCLING OF NICKEL OXIDE FROM PALM OIL MILL FUEL ASH (POMFA)**

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## **ABSTRACT**

Nickel oxide has been used in lots of industries especially in petrochemical industries, mainly as catalyst in chemical processes due to its unique combination of properties. Nickel oxide also known as heterogeneous catalyst or noble metal catalyst, usually used in hydrogenation reaction. Since nickel oxide demand is extraordinary, production plant for nickel oxide also increase significantly. The downsides of developing a plant is the massive cost required and environmental effect due to disposal of palm oil mill fuel ash (POMFA) from palm oil plant. To solve this issue, other alternative method identified, which is recycling of nickel oxide from palm oil mill fuel ash (POMFA). In Malaysia itself, there are many palm oil plants where the waste from the plant can be used to recover nickel oxide. The objective of this research is to study experimentally the recycling method of nickel oxide from palm oil mill fuel ash and the parameter involve in this experiment (pH value, type of leaching agent and concentration of leaching agent). The recycling of nickel oxide from palm oil mill fuel ash (POMFA) consists of six-step processes which are POMFA characterization, nickel oxide extraction, separation, purification, nickel oxide analysis and lastly yield determination. Once POMFA undergoes those six stages of processes, nickel will be recycled and nickel yield can be determined.

## **ABSTRAK**

Nickel oxide atau nikel oxida banyak digunakan di dalam industri khususnya industri petrokimia. Ianya selalu digunakan sebagai catalyst atau pemangkin didalam proses kimia disebabkan oleh keunikan sifat dan komposisi yang diperoleh. Nickel oxide dikategorikan sebagai pemangkin yang bersifat pelbagai atau heterogeneous, selalunya digunakan dalam proses penghidrogenan. Memandangkan permintaan nickel oxide semakin bertambah, sudah tentu kilang pengeluaran bagi nickel oxide semakin bertambah. Kos yang besar untuk membina tapak dan kilang pengeluaran nickel oxide serta pencemaran yang dihasilkan menyebabkan pembangunan kilang dan tapak nickel oxida tidak begitu membuahkan hasil. Untuk menyelesaikan masalah ini, alternatif atau cara lain dipraktikkan, iaitu mengguna semula nickel oxide daripada bahan terbuang kilang sawit (POMFA). Di Malaysia sendiri, terdapat banyak kilang sawit yang menghasilkan bahan terbuang terbuang seperti POMFA. Objektif kajian ini ialah untuk mengitar semula nickel oxida dari bahan terbuang POMFA dan untuk mengetahui implikasi menggunakan pembolehubah yang berbeza (kepekatan larutan asid, nilai pH dan jenis asid yang digunakan). Proses mengguna semula bahan terbuang daripada kilang sawit ini mengandungi enam peringkat. Setelah bahan terbuang menjalani keenam-enam peringkat, maka nickel oxida dapat diperoleh.

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## LIST OF ABBREVIATION

AAS	Atomic Absorption Spectrometer
FESEM	Field Emission Scanning Electron Microscope
ICP	Inductive Coupled Plasma
POMFA	Palm Oil Mill Fuel Ash
µm	Micro Meter
wt.%	Weight Percentage



# 1 INTRODUCTION

## *1.1 Background*

Chemical processes in industries which involve gases reaction often require catalyst in order to speed up the reaction. Catalyst is substances that speed up reaction by providing an alternative pathway for the breaking and making of bonds. It also means that by adding a catalyst in a chemical reaction, less activation energy is required(Nur, n.d.). Titanium oxide is an example of catalyst. There are two types of catalyst, homogenous and heterogeneous. If the catalyst is in the same phase as the reactants, it is called as homogeneous catalyst. A heterogeneous catalyst on the other hand is in a different phase to the reactants and products(Barbaro & Liguori, 2010). Nickel oxide is an example of heterogeneous catalyst. Nickel oxide is used in manufacturing of synthesis gases such as carbon monoxide and hydrogen (CIEC, 2013).

Since nickel oxide used in industrial process is increasing, production plant for nickel oxide also increase. Currently nickel oxide is produced from non-renewable sources such as ore. To obtain the ore, we need to develop a mining area and production plant. But nickel oxide production plant is really expensive. In order to reduce money lost as well as environmental issue, a method to obtain nickel oxide without building a production plant is introduced. The method is called recycle of nickel oxide from palm oil mill fuel ash. This method use six-stage processes to recycle nickel oxide from palm oil mill fuel ash (POMFA).

## *1.2 Motivation, problem statement and brief review*

Nickel oxide or nickel is widely used in variety of field. Currently, nickel is produced by 2 types of ore which is laterites and sulphides(Hatch, n.d.). Based on (Dalvi et al., 2004), production of nickel from laterite ores has occurred for over 100 years beginning with processing of garnieritic ores from New Caledonia. However, until now the world nickel supply has been predominantly from sulphide sources. Plant development not only requires huge cost, but it also produces waste which pollute our environment. Based on International Finance Group report (Description, 2007), nickel plant produce three types of waste which are air emissions, liquid effluents and solid

wastes or sludge. In order to treat the waste from the processing plant, we need a lot of money.

Currently, we identified that in order to obtain nickel oxide, lots of money need to be invested. Based on report from (Goro Nickel Project, 2014), the total cost for production nickel plant is \$3.2 billion. This is because currently, nickel oxide is obtained from Lateritic nickel ore deposits, which are non-renewable resources.

One of the options to solve this problem is to recycle nickel oxide from waste product. There are many type of waste such as fly ashes, Ni-Cd batteries, plating and deposition industries waste, electronic waste, spent catalyst, button cell batteries and medical waste. There are three ways to recover metal specifically nickel oxide which are pyrometallurgy, hydrometallurgy and lastly bio-hydrometallurgy(Jadhav & Hocheng, 2012).

POMFA is a short form for palm oil mill fuel ash. It is from waste by-product of oil mills arising from the burning of palm oil shell and palm oil bunches which are used to power oil mill plants. The POMFA which is disposed-off by the Malaysian palm oil mills into landfills could be causing environmental pollution and there is a need to study the possibility of recycling this waste product (Chemical,Biological and Environmental Engineering, 2010). Considering the amount of POMFA arising from palm oil mills in Malaysia, Thailand, Indonesia and other palm oil producing nations and the desire to address environmental problem posed by this waste, there is a need to examine further on the application of POMFA at higher volume particularly in petrochemical process(Embrandiri, Ibrahim, & Singh, 2013).

According research done by Sandra Vitolo (Vitolo, 2001), vanadium and nickel can be recovered from previously burned heavy oil fly ash. Basically, this research shows that waste from palm oil plant can be recycle to obtain desired product which is nickel. By recycling waste from palm oil plant, we can reduce the cost required to build nickel production plant as well as maintain our environment from waste produced by palm oil plant. There are six stages in nickel oxide recycling process. The six stages are POMFA characterization, nickel oxide extraction, separation process, washing and purification process, nickel oxide analysis and lastly, yield determination. These are the six stages used in order to recycle nickel oxide from palm oil mill fuel ash.

### ***1.3 Objectives***

The following are the objectives of this research:

- To study experimentally the recycling method of nickel oxide from palm oil mill fuel ash.
- To study the parameter involve in this experiment (pH value of leaching agent, type of leaching agent, concentration of leaching agent.)

### ***1.4 Scope of this research***

The following are the scope of this research:

- i) Characterization of POMFA
- ii) Recycling of nickel oxide from POMFA
- iii) Separation nickel oxide by using acid leaching, filtration and precipitation.
- iv) Purification of nickel oxide by washing process.
- v) Analysis of nickel oxide.
- vi) Yield determination of nickel oxide.

### ***1.5 Main contribution of this work***

The following are the contributions

### ***1.6 Organisation of this thesis***

The structure of the reminder of the thesis is outlined as follow:

Chapter 2 provides a description of the method that involved in metal separation process. This chapter also provides a brief discussion on current method to obtain nickel oxide, nickel oxide properties and application in industry. Analysis of worldwide nickel demand and production also provided. Properties of POMFA also included in this chapter. A brief review on type of catalyst is also presented. A summary of previous experimental work done which is similar to this research is also presented in this chapter.

Chapter 3 gives a review of the experimental method used in this research. POMFA characterization including sample analysis and sample sizing is presented. Nickel

oxide extraction procedure and separation process is discussed in detail. Washing and purification procedure is also presented. Lastly, nickel oxide analysis and yield determination method can be obtained in this chapter.

Chapter 4 is devoted to the preliminary result done by the researcher before continuing the research to the next level. In this chapter, the researcher had focused on the availability of nickel oxide in the sample. Sample characterization according to size is also conducted. The illustrated figures along the preliminary works were also provided in this chapter.

Chapter 7 draws together a summary of the thesis and outlines the future work which might be derived from the model developed in this work.

## **2 LITERATURE REVIEW**

### ***2.1 Overview***

This paper presents the experimental studies for recycling of nickel oxide from palm oil mill fuel ash (POMFA) and parameters involved which effect the yield of nickel oxide. The metal separation method used in this research is hydrometallurgy method and the solvent used for leaching or separation process is acidic solvent.

### ***2.1 Type of metal separation process***

#### ***2.1.1 Pyro-metallurgy***

It is a process used to recover valuable elements from industrial wastes. Pyro-metallurgical processes have proved to be more efficient for the extraction of metals, such as Ti, Zr, Nb, Ta and Mo. Pyro-metallurgy employs the thermal treatment to bring about physical and chemical transformations in the materials to enable recovery of valuable metals. The methods used for metal recovery are smelting, roasting, converting and refining. This process is fast because the physical form of the scrap is not as important as that required in chemical treatments. However, most methods involving thermal processing are quite expensive due to high-energy requirement. Furthermore, this thermal process usually produces polluting emissions and causes the loss of metals from the scrap during combustion (Jadhav & Hocheng, 2012)

#### ***2.1.2 Hydrometallurgy***

It is a process in which chemical reactions are carried out in aqueous or organic solutions for the recovery of metals. Typically three general steps are carried out during hydrometallurgical recovery of metals, which is leaching, solution concentration and purification, and metal recovery. Various reagents have been used in chemical leaching. These include any type of acid and alkaline. In the solution concentration and purification step, the solutions are subjected to separation procedures such as solvent extraction, precipitation, cementation, ion exchange, filtration and distillation to isolate and concentrate the metals of interest. Metal recovery is the final step in a hydro-metallurgical process. The electrolysis, gaseous reduction, and precipitation are the metal recovery processes (Jadhav & Hocheng, 2012).

### 2.1.3 Bioleaching

Bioleaching has been applied successfully and commercially in biohydrometallurgy for extracting copper and precious metals from low-grade ores and tailings for many years. In biotechnological processes, solubilization of metals is based on the interactions between metals and microorganisms. This technique allows metal recycling by processes similar to that in the natural biogeochemical cycles, and is therefore environmentally friendly, with low cost and low energy requirement. The microbial leaching process, using aerobic sulphur- and iron-oxidizing microorganisms, has been shown to be capable of eluting the heavy metals associated with solids. Heterotrophic bacteria and fungi have been used to mobilize metals (*Bacillus* sp., *Aspergillus niger* and *Penicillium simplicissimum*, *Saccharomyces cerevisiae*, *Yarrowia lipolytica*). For this study, we are using hydrometallurgy recovery method because it is more convenient compare to other methods.

## 2.2 Nickel oxide

### 2.2.1 Properties of nickel oxide

Nickel is a hard silver white metal, which occurs as cubic crystals. It is malleable, ductile and has superior strength and corrosion resistance. The metal is a fair conductor of heat and electricity and exhibits magnetic properties below 345°C. Five isotopes of nickel are known.

In its metallic form nickel is chemically unreactive. It is insoluble in cold and hot water and ammonia and is unaffected by concentrated nitric acid and alkalis. It is however soluble in dilute nitric acid and sparingly soluble in dilute hydrochloric and sulphuric acids. Nickel oxide is a powdery green solid that becomes yellow on heating. Table below is the summary of nickel oxide properties.

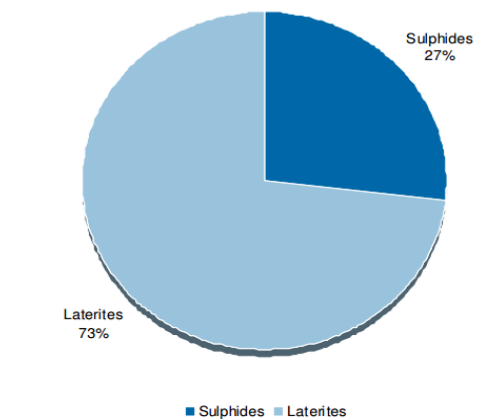
Formula	Colour	Oxidation State	MP
NiO	green powder	Ni <sup>2+</sup>	1955

**Table 1: Summary of nickel oxide properties**  
Source: (Chemistry, 2006)

### 2.2.2 *Current method to obtain nickel oxide*

Primary nickel is produced from two very different ores, laterites and sulphides. Laterites ores are normally found in tropical climates where weathering, with time, extracts and deposits the ore in layers at varying depths below the surface. Lateritic ores are excavated using large earth-moving equipment and are screened to remove boulders. Sulphides ores on the other hand often found in conjunction with copper-bearing ores, are mined from underground (Description, 2007).

Based on the research conducted by (Hatch, n.d.), 73% nickel produce by laterite ore while the remaining 27% produced by sulphides ore. Pie chart for the data is shown below.



**Figure 1: Nickel Resources by Ore Type**  
Source: (Hatch, n.d.)

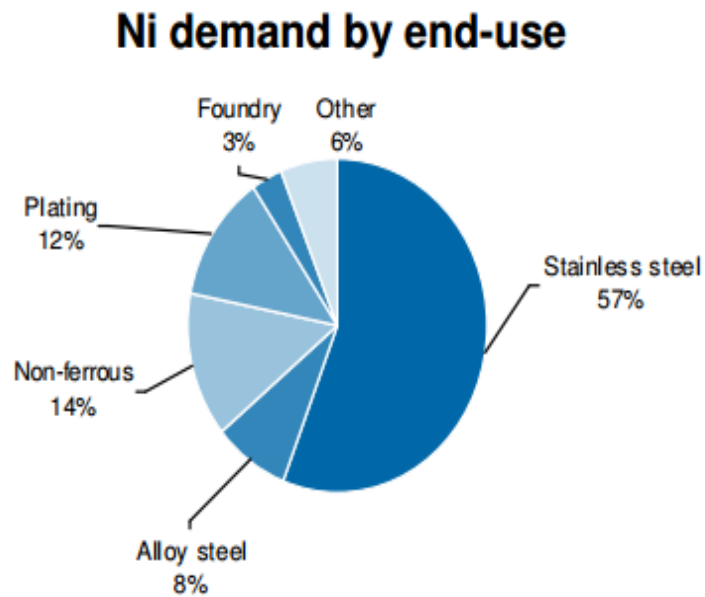
There are two types of processes laterites and sulphides ore processing. Both of the processes used dried concentrates. Various processes are used to refine nickel matte. Examples of the processes are fluid bed roasting and chlorine-hydrogen reduction. These processes are able to produce high-grade nickel oxide (more than 95% nickel).

### 2.2.3 *Application of nickel oxide*

Based on (Bradley, 2011), nickel oxide has wide application such as electrochemical material, battery, electronic application, gas sensors and mainly as catalyst. The catalyst usually used for various reactions, such as reduction of NO<sub>x</sub>, desulphurisation of hydrocarbons, partial and complete oxidation reactions of various

organic substances and many more. According to (Wang, 2004) and (Kumar et al., 2008) nickel also used as nickel based catalyst for certain chemical process.

According to (Dalvi et al., 2004), most of the nickel is used as stainless steel industry which is 57%. Nickel also used in non-ferrous metal industry which is 14%, plating industry which is 12%, alloy steel industry which is 8%, other industry such as petrochemical which is 6% and lastly, foundry industry which is 3%. The pie chart for the data is shown below.



**Figure 2: Nickel Demand by End-use Worldwide**  
Source: (Dalvi et al., 2004),



### 2.2.4 Sources and demand of nickel oxide

Since nickel oxide or nickel is used in multiple field worldwide, the demand will increase from time to time. Data below show nickel production from 1950 up to 2003.

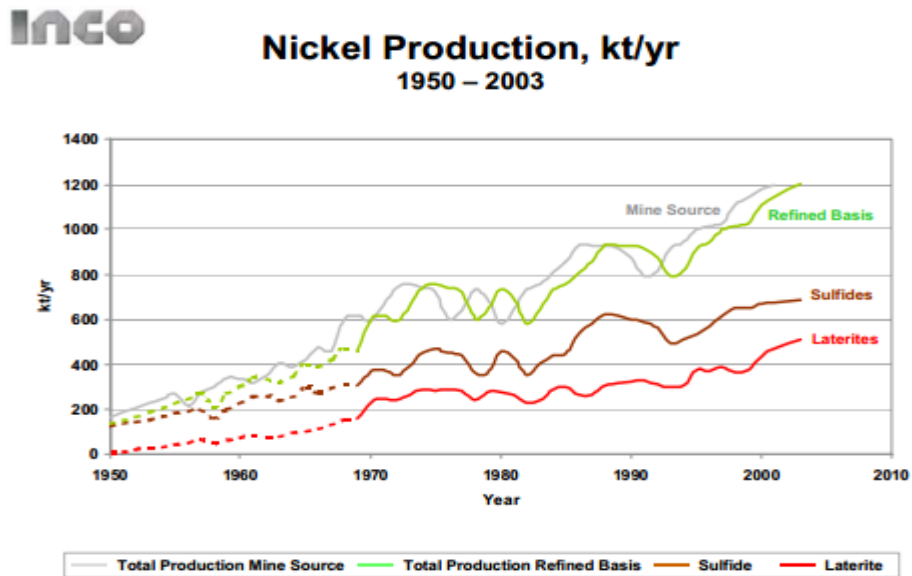


Figure 3: Nickel production base on demand from 1950 to 2003  
Source: (Dalvi et al., 2004),

In order to fulfil worldwide demand, some countries have taken initiative to develop mining and processing plant for nickel. Not all countries can simply develop mining area since it depends on the geographical state of the country itself. Data below show world nickel laterite resources.

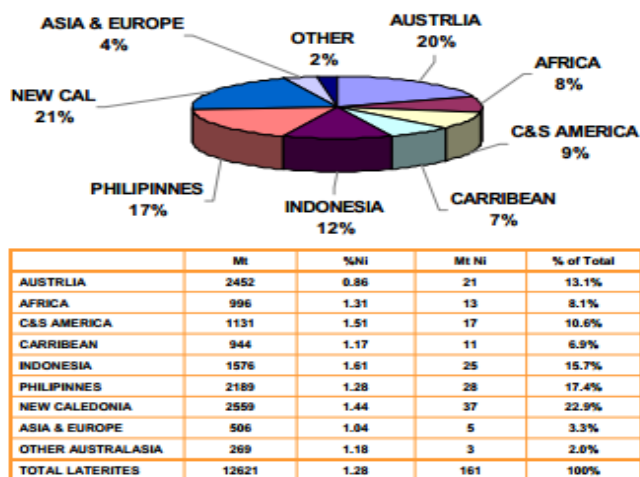


Figure 4: World Nickel Laterite Resources

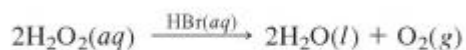
Source: (Dalvi et al., 2004)

Based on figure 4, we can see that the biggest nickel laterite resource are from New Caledonia which is 21%, followed by Australia which is 20%, 17% for Philippines, 12% for Indonesia, 9% for C&S America, 8% for Africa, 7% for Caribbean, 4% for Asia and Europe and lastly, 2% for other Australasia

## 2.3 Nickel as catalyst

### 2.3.1 Catalyst

A catalyst is a substance that increases the reaction rate without being consumed in the overall reaction process (D.Gammon, 2011/2009). Since is not consumed in the reaction, it does not appear in the balance chemical equation. For example, a solution of pure hydrogen peroxide ,  $H_2O_2$ , is stable. But when Hydrobromic acid,  $HBr(aq)$ , is added,  $H_2O_2$  decomposes rapidly into  $H_2O$  and  $O_2$ .



Here,  $HBr$  act a s a catalyst to speed up decomposition process. A suitable catalyst can enhance the rate of a thermodynamically feasible reaction but cannot change the position of the thermodynamic equilibrium. Most catalysts are solids or liquids, but they may also be gases. However, catalysts do not have infinite life. Products of side reactions or changes in the catalyst structure lead to catalyst deactivation. In practice spent catalysts must be reactivated or replaced (DEUTSCHMANN, 2009).

Catalysis plays a vital role in the chemical industry by contributing to both its economical success and environmental sustainability. More than 75% of all industrial chemical transformations employ catalysts in areas as diverse as polymers, pharmaceuticals, agrochemicals, and petrochemicals. In fact, 90% of newly developed processes involve the use of catalysts . Heterogeneous catalysis is widely used in industrial applications because of the facile separation, which often results in lower operating costs. On the other hand, homogeneous catalysis has limited industrial applications due to the difficult and costly catalyst separation and recovery. Table 1 compares homogeneous and heterogeneous catalysis in terms of catalytic effectiveness, catalyst properties, and catalyst separation. Homogeneous catalysts offers improved selectivity, increased activity, and avoid mass transfer limitations, which may permit lower temperatures (Ali Z. Fadhel 1, 2010). Table 2 shows comparison between homogeneous and heterogeneous catalyst.

	<b>Homogeneous</b>	<b>Heterogeneous</b>
<b>Active Centers</b>	All atoms	Only surface atoms
<b>Selectivity</b>	High	Low
<b>Mass Transfer Limitations</b>	Very rare	Can be severe
<b>Structure/Mechanism</b>	Defined	Undefined
<b>Catalyst Separation</b>	Tedious/Expensive (extraction or distillation)	Easy
<b>Applicability</b>	Limited	Wide
<b>Cost of Catalyst Losses</b>	High	Low

**Table 2: Comparison Between Homogeneous and Heterogeneous Catalyst**

### **2.3.2 Homogeneous catalyst**

If the catalyst and reactants or their solution form a common physical phase, then the reaction is called homogeneously catalysed. Metal salts of organic acids, organometallic complexes, and carbonyls of Co, Fe, and Rh are typical homogeneous catalysts. Examples of homogeneously catalysed reactions are oxidation of toluene to benzoic acid in the presence of Co and Mn .This reaction is catalysed by carbonyls of Co or Rh (DEUTSCHMANN, 2009). Catalysts are classified as homogeneous if they are present in the same phase as the reagents. This normally means that catalysts are present as solutes in a liquid reaction mixture.

Homogeneous catalysis by organometallic complexes is finding wide application in both bulk and fine chemicals and is the method of choice in e.g. carbonylation and hydroformylation. Furthermore, the incorporation of transition metal ions and complexes into molecular sieves extends their catalytic scope to redox reactions and a variety of other transition metal-catalysed processes (Nur, 2006).

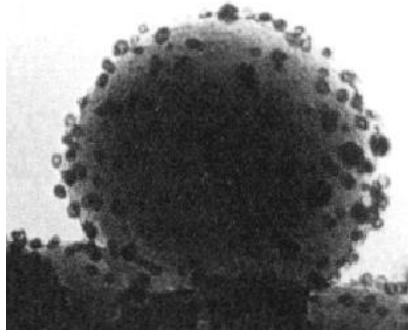
One of the simplest examples is found in atmospheric chemistry. Ozone in the atmosphere decomposes, among other routes, via a reaction with chlorine atoms.

Ozone can decompose spontaneously, and also under the influence of light, but a Cl atom accelerates the reaction tremendously. As it leaves the reaction cycle unaltered, the Cl atom is a catalyst. Because both reactant and catalyst are both in the same phase, namely the gas phase, the reaction cycle is an example of homogeneous catalysis (J.A. Moulijn, 1993).

### ***2.3.3 Heterogeneous catalyst***

Heterogeneous catalysis involves systems in which catalyst and reactants form separate physical phases. Typical heterogeneous catalysts are inorganic solids such as metals, oxides, sulphides, and metal salts, but they may also be organic materials such as organic hydro-peroxides, ion exchangers, and enzymes. Examples of heterogeneously catalysed reactions are ammonia synthesis from the elements over promoted iron catalysts in the gas phase and hydrogenation of edible oils on Ni – kieselguhr catalysts in the liquid phase, which are examples of inorganic and organic catalysis, respectively. (DEUTSCHMANN, Heterogeneous Catalysis, 2009)

In heterogeneous catalysis, solids catalysed reactions of molecules in gas or solution. As solids ,unless they are porous, are commonly impenetrable, catalytic reactions occur at the surface. To use the often expensive materials (e.g. platinum) in an economical way, catalysts are usually nanometer-sized particles, supported on an inert and have porous structure. Heterogeneous catalysts are the workhorses of the chemical and petrochemical industry



**Figure 5: Catalyst as nanomaterial**

As we can see in Figure 5, catalyst is nanomaterial and catalysis is nanotechnology. If we define nanotechnology as the branch of materials science aiming to control material properties on the nanometer scale, then catalysis represents a field where nanomaterial have been applied commercially for about a century. Many synthetic techniques are available to produce small particles for heterogeneous catalysts, and to keep them sufficiently stable so that they can withstand the often hostile conditions of an industrial reactor. Modern catalysis is pre-eminently nanotechnology. (Adapted from A.K. Datye and N.J. Long, *Ultramicroscopy* 25 (1988) 203).

#### ***2.4 Palm oil mill fuel ash (POMFA)***

One such industrial by-product is palm oil mill fuel ash (POMFA), which is produced by burning palm oil shell and husk as fuel in a palm oil mill boiler in order to produce steam to generate electricity for the palm oil extraction process (Igwe & Onyegbado, 2007) and (Bamaga, Hussin, & Ismail, 2013).

Usually this kind of waste will go to landfill for disposal but in this study, we are using this kind of waste to recycle valuable product such as nickel oxide. For this experiment, POMFA were collected from CH Biotech Sdn.Bhd.

## **2.5 Past Research**

Recycle of nickel from heavy fuel fly ash is important from the viewpoint of reclamation of nickel which are considered as one of the most hazardous metal species creating serious hydrometallurgical problem in terms of environmental conservation. Recovery of nickel from fly ash is also of economical concern. The fly ash deriving from heavy fuel oil combustion contains appreciable levels nickel (up to 7 wt.%) in highly leachable forms that makes their recovery interesting. Nickel is an important metal applied in many fields, such as energy materials and functional materials, chemicals and catalysts, cathodes, batteries and it has several metallurgical benefits such as high melting point, ferromagnetic properties and ease of electroplating. T-sai et al. reported a two-stage leaching method, applicable to recovery of nickel, vanadium and ammonium sulphate from fly ash. Amer (Amer, 2002) worked on the hydrometallurgical processing that involved acid leaching under oxygen pressure of ground ash, followed by electrolytic separation of nickel from sulphate solution and nickel was then neutralized and precipitated by adjusting pH value of the solution. Abdel-latif (Xiao et al., 2010) reported a developed process flow sheet used as a basis to identify the test work required for the major processing units. The flow sheet consisted of drying, de-carburization and desulfurization stages, followed by a smelting stage at the end. In another study, extraction of nickel from heavy fuel oil fly ash was performed using a burning stage to reduce the carbonaceous fraction, followed by acid leaching and oxidative precipitation of vanadium oxide. The advantage of this approach over the direct leaching of the raw fly ash is the reduced volume of a more concentrated residue as a feed entering to the nickel recycling process. According to Tsygankova et al. (Mahdavian et al., 2006) there is no single approach to directly recover vanadium from various ashes and each ash needs its own leaching procedure depending on its chemical and phase compositions. Navarro et al. (Al-ghouti et al., 2011) used leaching processes (acidic and alkaline treatments), followed by a second step of metal recovery from leachates involving either solvent extraction or selective precipitation.

In one such study, acidic solution was discovered to be compelling and economic than the alkaline solution for nickel and other related metals from the waste at the same time. The leach solution created using dilute sulphuric acid as leaching reagent was utilized for the separation of the dissolved metal ions by dissolvable extraction and precipitation procedures (Barik et al., 2014). Nickel leaching has yielded an extensive variety of

results. Shifting conditions have been connected in order to acquire reasonable recovery of nickel from distinctive sources. (Tavakoli et al., 2014).

In this work, a straightforward four-stage process for the recycle of nickel from palm oil plant fuel ashes was considered. The recycle procedure comprised of preliminary burning to reduce the carbonaceous fraction, acid leaching, oxidation and precipitation of nickel oxide. The utilization of extraction solvents was stayed away from and the purity of the precipitate was accomplished through a particular washing by using distilled water of the precipitate in order to control the concentration of the impurities.

## **3 MATERIALS AND METHODS**

### ***3.1 Overview***

This paper presents methodology that will be implemented during the lab work process.

### ***3.2 Material and equipment***

#### ***3.2.1 Palm oil mill fuel ash***

Palm oil mill fuel ash or POMFA is used as raw material in this research. Palm oil mill fuel ash (POMFA), is a waste by-product from palm oil mills which continuously increasing in amount. The POMFA which is disposed off by the Malaysian palm oil mills into landfills could be causing environmental pollution and there is a need to study the possibility of recycling this waste product. In this research, nickel oxide is to be recycled from palm oil mill fuel ash. POMFA were collected from palm oil plant in Perak and Pahang, Malaysia.

According to (Arabia, 2013), fuel ash or fly ash is a powdery residue generated by plants that use oil as the source of fuel. Fuel oil fly ash contains relatively high heavy metal content such as V and Ni. Furthermore, the residual carbon level in the fly ash is relatively high. Typical fuel oils contain Fe, Ni, V, and Zn. The composition inside fly ash is mainly vanadium 27 %, sulphur 12%, nickel 6%, magnesium 3%, cadmium 1%, Fe 1% and C 37% .

#### ***3.2.2 Other chemicals and reagents***

Other reagents were purchased by University Malaysia Pahang laboratory such as sodium carbonate, nitric acid, sulphuric acid, hydrofluoric acid, hydrochloric acid and sodium chlorate. All of these chemicals were analytical grade reagents. Hydrochloric and sulphuric acid is used as an acidic leaching agent. Sodium chlorate is used as oxidants and sodium carbonate is used to maintain ideal ph.