

**A NEW NON REGENERATIVE GAS ABSORBANT CATALYST FOR FCC UNIT**

**EZANI BIN ZAHARI**

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**Faculty of Chemical & Natural Resources Engineering  
Universiti Malaysia Pahang**

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## ABSTRACT

The FCC process operates in a dynamic heat balance with hot regenerated catalyst supplying the net heat demand required by the reaction system. Finely sized solid catalyst continuously circulates in a closed loop between the reaction system and the catalyst regeneration system. The feed and catalyst are intimately contacted in the riser reactor, in the proper ratio and with the proper residence time and temperature to achieve the desired level of conversion. The catalyst passes through a highly efficient, patented, spent catalyst stripper where any hydrocarbon product vapors entrained with the catalyst are removed and recovered. The process of cracking hydrocarbon provide human a fuel but the side effect is very dangerous to the environment because of releasing flue gases at regenerator. In this study, the non regenerate catalyst will be used in order to replace the current catalyst that need to be regenerate. This experiment was carried out in the reactor that has high test pressure so that it will stand still at high temperature. The catalyst was mixed with kerosene and burned at 190°C. The result obtained were test for it viscosity, density and funtional group. The catalyst funtional group is similar to the zeolite Y which have been used in modern cracking process. The funtional group for product contain of several type of disturbance. The density obtained was 0.7461 g/cm<sup>3</sup> and the viscosity obtained was 2.4 cP. The result showed that catalyst that had been used can crack down the hydrocarbon.

## ABSTRAK

Proses FCC beroperasi dalam keseimbangan panas dinamik dengan mangkin dipulih panas membekalkan keperluan panas yang cukup yang diperlukan oleh sistem reaksi. Mangkin padat bersaiz halus terus menerus beredar dalam satu lingkaran tertutup antara sistem reaksi dan sistem regenerasi mangkin. Mangkin kemudian memasuki stripper dan dibuang semua hidrokarbon yang menghalang permukaan reaksi pada mangkin. Sistem regenerasi mengembalikan aktiviti katalitik dari mangkin menghabiskan coke-sarat dengan pembakaran dengan udara. Hal ini juga menyediakan reaksi panas dan pengewapan bahan mentah dengan kembali panas, mangkin baru kembali ke sistem proses. Pemecahan hidrokarbon memberikan manusia bahan bakar tetapi kesan samping yang sangat berbahaya bagi persekitaran kerana mengeluarkan gas rumah hijau dari regenerator. Dalam kajian ini, mangkin yang tidak perlu diregenerasi akan digunakan untuk menggantikan mangkin saat ini yang perlu regenerasi. Penelitian ini dilakukan dalam reaktor yang memiliki ujian tekanan tinggi yang tahan pada suhu yang tinggi. Mangkin dicampur dengan minyak tanah dan dibakar di 190°C. Keputusan yang diperolehi ujian untuk itu viskositas, ketumpatan dan kumpulan funtional. Kepadatan diperolehi 0,7461 g/cm<sup>3</sup> dan viskositas diperolehi 2,4 cP. Keputusan kajian menunjukkan bahawa mangkin yang telah digunakan boleh menindak hidrokarbon.

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## LIST OF SYMBOLS

$^{\circ}\text{C}$	-	Degree Celsius
$^{\circ}\text{F}$	-	Degree Fahrenheit
$\text{cm}^3$	-	Cubic Centimeter
cP	-	centi Pases
$\text{CO}_2$	-	Carbon Dioxide
CO	-	Carbon monoxide
g	-	gram
$\text{N}_2$	-	Nitrogen
$\text{NO}_x$	-	Nitrogen oxide

## CHAPTER 1

### INTRODUCTION

#### 1.0 BACKGROUND OF THE STUDY

In all modern refineries, the fluid catalytic cracking unit (FCCU) remains the major means of gasoline production. In a FCCU heavy oil feedstocks are converted (cracked) into high-value liquids such as gasoline and heating oil in the presence of a fluid cracking catalyst (FCC) operating at temperatures in the 480–550 °C range. Following the cracking reaction, the spent catalyst is first exposed to steam (480–540 °C) to remove occluded hydrocarbons, and then it is sent to the regenerator where coke deposits are removed by heating in air at temperatures in the 600–700 °C range. The regenerated FCC is then ready to be recycled to the FCCU reaction zone (the riser). Easy accessibility of active sites in the internal porosity of fluid cracking catalysts is essential to the efficient and selective cracking of gas oils to gasoline and other valuable products. [1]

In the FCC unit, hydrocarbon feed contacts the catalyst. The hydrocarbons crack and deposit carbonaceous material (coke) on the catalyst. Cracked hydrocarbon products are separated from the coked catalyst. The spent catalyst is then stripped of volatiles by steam and regenerated. In the catalyst regenerator, coke is burned from the catalyst with an oxygen-containing gas.[2]

Thus, every time the catalyst contact with coke, the catalyst will have a weight loses due to the burn of coke at the catalyst regenerator. This will produce a economical loses and every time the process of cracking hydrocarbon start, the catalyst need to be added in the cracking unit and this will lead to non economical process. The contact of coke with the catalyst also will reduce the catalyst activity due to contact surface of the catalyst and the hydrocarbon will become limited.[5]

An approximately 2000 t/year of  $\text{NO}_x$  is released from a typical refinery and that FCC regenerator flue gases contribute approximately 50% of the total  $\text{NO}_x$  emissions. The concentrations of  $\text{NO}_x$  emissions from regenerator flue gases vary between 50 and 500 ppm depending on the feed, the operating conditions, and the amount of CO combustion promoter (Pt-based additive) used. [3]

In the FCC process, nitrogen-containing species in the feedstock are cracked in the riser reactor to lighter molecules, while approximately 40% of the nitrogen is deposited in the coke on the spent catalyst. The aromatic feedstock composition and, in particular, the basic nitrogen-containing molecules enhance the coke yield. During oxidative regeneration, approximately 90% of the coke-bound nitrogen is converted to molecular nitrogen ( $\text{N}_2$ ), and the rest is released in the form of  $\text{NO}_x$ . The main source of nitrogen leading to  $\text{NO}_x$  formation is the FCC feedstock (“fuel  $\text{NO}_x$ ”), whereas only minor amounts (<10 ppm) are formed by  $\text{N}_2$  oxidation (“thermal  $\text{NO}_x$ ”) and the reaction between radicals (“prompt  $\text{NO}_x$ ”) in the regenerator. [3]

In the regenerator reactor, the process of regenerating catalyst is very hot. In order to cool the flue gas, the hot regenerator is injected with oxygen and then the cooled flue gas is contacted with a solid particulate material which has the capability of associating with and binding sulfur oxides in the flue gas to form a stable solid material which is separated from the gases. It also generates carbon dioxide, carbon monoxide and water as combustion products and releases large amounts of heat. This produce another hazardous gases that is not environmental friendly. [4]

From those statement, the catalyst regenerator will produce environmental pollution which is hazardous to human being. Thus, the study of a new non regenerative catalyst is needed in order to reduce the pollution.

### **1.1 PROBLEM STATEMENT**

When the reaction occurred between the catalyst and crude oil in the cracking reactor, the coke will surrounding the catalyst. This lead the catalyst to have a lower activity and result the lower yield. In order to save the cost of catalyst, the spent catalyst is need to be regenerate and the method to is fill the spent catalyst into the reactor. In the reactor, the spent catalyst will be burn at high temperature in order to eliminate coke that surrounding the catalyst. The catalyst composition is weaker during the burning of coke in the regenerator and causes the industries the economical loses.

The burning of coke also produce the environmental hazard because it releases some hazardous gases such as  $\text{NO}_x$ ,  $\text{SO}_x$ ,  $\text{CO}_2$  and some other flue gas. Due to government legislation for releasing of the flue gas, the industries need to find a way to overcome the problem.

### **1.2 RESEARCH OBJECTIVES**

1. To apply the non regenerative catalyst for fluid catalyst cracking unit.
2. To determine the performance of the non regenerative catalyst.

### 1.3 SCOPE OF RESEARCH

In order to achieve the objectives of the study, the scopes of research were listed down as below:

1. The study of non regenerative catalyst that contained zeolite and have a large amount in Malaysia.
2. Used non regenerative catalyst can be used for the other purpose such as construction.
3. The studied catalyst must have low price value.

## CHAPTER 2

### LITERATURE RIVIEW

Fluid catalytic cracking (FCC) is a key process in modern refineries. Worldwide approximately 300 FCC units are operated, converting vacuum gas oil and high boiling residues into lighter fuel products and petrochemical feedstocks. Because of the central function of the FCC process, a range of technological improvements have been implemented to increase its economical benefits [4]. In addition to investments concerning the process design, new catalysts and additives have been developed to fulfill the economic demands of the market and to obtain the desired products. [7]. However, refiners are bound to invest also in eco-efficient technologies for the production of fuels and petrochemicals with significantly reduced emissions of environmental pollutants.

This is imposed by various national and international regulations addressing emissions from a range of refinery processes and especially FCC regenerators, such as NO, SO<sub>x</sub>, CO, and CO<sub>2</sub> emissions from regenerator flue gases. Approximately 2000 tons of NO<sub>x</sub>/year are released from a typical refinery, among which the FCC units contribute approximately 50%. The concentrations of the NO<sub>x</sub> emissions from regenerator flue gases vary between 50 and 500 ppm depending on the nature of the

feed, the operating conditions of the FCC unit, and the amount of CO promoter added. [8]

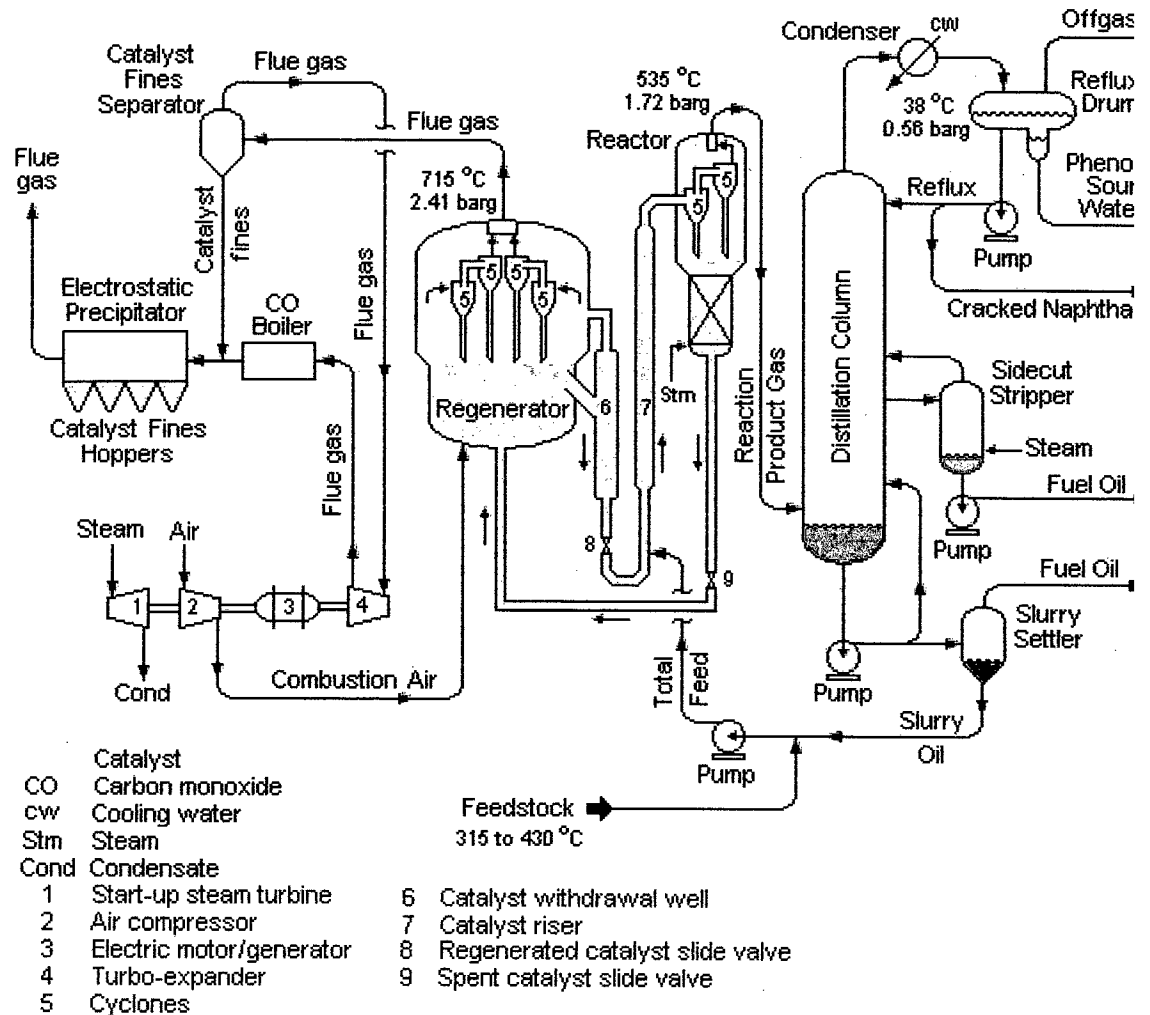


FIGURE 2.1: FLUID CRACKING CATALYTIC DIAGRAM

## 2.1 Coke Formation

In the fluid catalytic cracking (FCC) process, the reversible deactivation of FCC catalysts has been mainly associated with the carbonaceous deposits or coke formed on the catalyst surface. [10]. Coke yield is an important measurement of FCC catalyst performance and one of the key criteria in catalyst selection and catalyst performance optimization. A detailed understanding of coke composition and the impact of catalyst properties, feed composition, and feed–catalyst interactions on coke formation is critical to the development of FCC catalysts and commercial FCC operation. In addition, since a majority of the coke is combusted in the FCC regenerator and released as flue gases, a better understanding of coke composition, particularly the heteroatomic coke components, may have significant implications for FCC environmental technologies, (SO<sub>x</sub>, NO<sub>x</sub>, and CO controls).

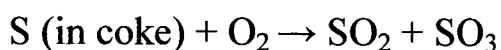
It has been generally recognized that coke is formed in an FCC process via reactions of feedstock molecules on acid sites of the FCC catalyst. Both feedstock composition and catalyst formulation have significant impact on coke yield. Hydrocarbon coking on catalyst have been extensively studied, where coking rate and selectivity were found to be closely related to the zeolite types and pore structures. Coke can be formed from a wide range of hydrocarbons, including small molecules such as propene, via a series of polymerization and dehydrogenation reactions. Polynuclear aromatics (PNA) and heteroatomic molecules in feedstocks are the most important precursors of FCC coke. Nitrogen molecules, particularly basic nitrogen compounds (pyridinic compounds), have the greatest impact on coke yield. [13]



## 2.2 Sulfur oxide formation

In the last few years great attention has been focused to control pollutant emissions. Among the major contributors to these emissions are energy power plants, which contribute with 65% of sulfur oxides, and petroleum refinery processes, more specifically the fluid catalytic cracking (FCC) process, with 7%. Sulfur oxides, a mixture of  $\text{SO}_2 + \text{SO}_3$ , commonly referred as  $\text{SO}_x$ , are one of the most dangerous atmospheric pollutants since they contribute directly to acid rain formation and the destruction of the ozone layer [19]. In the FCC process the  $\text{SO}_x$  production and removal mechanisms are different from those of energy power plants; for this purpose the characteristics of the sorbent should be different. After cracking reactions, the catalyst is deactivated and the coke deposited on it needs to be burned off to regenerate the catalyst activity; thus, sulfur compounds present in coke are oxidized to produce  $\text{SO}_x$  emissions in the regeneration zone. [12]

Sulfur oxide emissions ( $\text{SOX} = \text{SO}_2 + \text{SO}_3$ ) from fluid catalytic cracking units (FCCU) are increasingly becoming the target of EPA and local regulations [17]. The removal of such pollutants from FCC units has been the subject of a considerable amount of attention over the past few years. The amount of  $\text{SOX}$  emitted from a FCC unit regenerator is a function of the quantity of sulfur in the feed, coke yield, and conversion. Generally, 45-55% of feed sulfur is converted to  $\text{H}_2\text{S}$  in the FCC reactor, 35-45% remains in the liquid products, and about 5-10% is deposited on the catalyst in the coke. It is this sulfur in the coke which is oxidized to  $\text{SO}_2$  (90%) and  $\text{SO}_3$  (10%) in the FCC regenerator.



FCC regenerators operate in total and/or partial combustion, depending on the operating conditions. In the total combustion regime an oxygen excess will

promote the  $\text{SO}_2 \rightarrow \text{SO}_3$  oxidation, while in partial combustion the oxygen deficit could result in an incomplete  $\text{SO}_2 \rightarrow \text{SO}_3$  oxidation [21]

### 2.3 Formation of Nitrogen

Emission of  $\text{NO}_x$  (i.e.,  $\text{NO}$ ,  $\text{NO}_2$ , and  $\text{N}_2\text{O}$ ) from the fluid catalytic cracking regenerator is increasingly controlled by various state and local regulations. The FCC regenerator poses a very challenging environment for controlling  $\text{NO}_x$ . Other than  $\text{NO}$ , the high-temperature flue gas contains  $\text{O}_2$ ,  $\text{CO}$ ,  $\text{CO}_2$ ,  $\text{SO}_2$ ,  $\text{SO}_3$ ,  $\text{H}_2\text{O}$ , and possibly other nitrogen oxygen species. Any  $\text{NO}_x$  control technology has to be designed in a way that it neither interferes with the catalytic cracking reaction in the riser nor substantially increases the emissions of other pollutants, e.g.,  $\text{CO}$  or  $\text{SO}_2$ .  $\text{NO}_x$  levels in the FCC regenerator flue gas are typically in the range of 100–500 ppm.  $\text{NO}$  is the primary component of  $\text{NO}_x$  from the FCC regenerator.

$\text{NO}_2$  is formed only after being released to the air, while  $\text{N}_2\text{O}$  exists typically at very low levels. In addition to the level of feed nitrogen, it is also known that operating conditions and hardware design of the regenerator can significantly affect  $\text{NO}_x$  emission. For example, higher excess oxygen in the flue gas is known to correlate with higher  $\text{NO}_x$  emission, which has led to the notion that  $\text{NO}_x$  is formed from the oxidation of molecular nitrogen from the air, or the so-called thermal  $\text{NO}_x$ . Until very recently, a few systematic studies existed on the formation and control of nitrogen oxide in the FCC regenerator.[23]

### 2.4 Nitrogen Compounds in Feeds.

The nitrogen content in most FCC feeds is quite low, typically between 0.005 to about 0.5 wt %. Nitrogen is found at ppm levels in light and middle distillates, but increase significantly around 620 K (650 °F). The nitrogen compounds are typically distinguished by their basicity. Much work has been reported to determine the basicity of each particular group of nitrogen compounds [24]. According to their

molecular structure, most of the nitrogen compounds fall into the following four groups with decreasing basicity: amines, pyridine derivatives, pyrrole derivatives, and amides.

Typically, about one-third of the nitrogen is considered basic nitrogen according to titration analysis with perchloric acid. Most of the amine and pyridine types of nitrogen compounds are considered to be basic and are expected to be converted to coke. Literature also showed that these two types of nitrogen constitutes about one-third of the total nitrogen [32]. Most of the basic nitrogen is expected to be adsorbed on the acidic sites of the catalysts and be converted to coke during the cracking process, as we have shown in the nitrogen balance experiments. The percentage of nitrogen being converted to coke approximates the percentage of basic nitrogen in total nitrogen. Some of the nonbasic nitrogen may also contribute to coke. The fractions of nitrogen compounds left in the liquid products are expected to be the neutral or acidic types of nitrogen, e.g., pyrrole derivatives [27].

## 2.5 Nitrogen in Coke.

Judging from the nature of the nitrogen compounds in the feed, a significant fraction of the nitrogen probably exists in aromatic rings. If one assumes the coke molecule contains about 15–20 aromatic rings, each molecule has to contain at least two nitrogen atoms at a nitrogen in a coke level of 5 wt % [27]. One of the byproducts from the reaction between carbon and NO is  $C_2N_2$ . It has been speculated that the polymerization of  $C_2N_2$  could in turn form a high melting point  $(CN)_x$  polymer [28].

## 2.6 Formation of NO<sub>x</sub>

The exact chemistry on how the coke-bound nitrogen is being converted to NO<sub>x</sub>/N<sub>2</sub> is not sufficiently understood. However, we can draw an analogy between the regeneration process (coke combustion) with fluidized bed coal combustion. There exists extensive literature on the nitrogen chemistry during coal combustion [29,30]. Systematic work was also reported for the formation of nitrogen compounds during the regeneration of spent hydroprocessing catalysts [31].

Nitrogen in the coke has to go through some intermediates before being converted to NO or molecular nitrogen. For coal-bound nitrogen, HCN, and NH<sub>3</sub> are considered the intermediates for the formation of NO<sub>x</sub>. Regeneration of spent hydroprocessing catalysts also follows the same mechanisms. It is expected that nitrogen in FCC coke would probably go through a similar route. Regeneration of hydroprocessing catalysts indicated that the selectivity to HCN/NH<sub>3</sub>/N<sub>2</sub> from fuel-bound nitrogen is strongly affected by the type of nitrogen in the coke, or originally in the feedstocks [33]. Pyrrolic-N yields higher HCN than pyridinic nitrogen does. Model compounds studies for solid fuel combustion also showed that phenolic OH groups were found to increase the conversion of HCN to NH<sub>3</sub> [34]. Although there is very little evidence of amine groups in coal, the amine groups in FCC feed may be easily adsorbed on the catalyst and converted to coke.

The nitrogen in amine groups are converted to ammonium intermediates before being further converted. Some of the amine groups may also be cracked and released as ammonia in the riser. The subsequent destruction of HCN or NH<sub>3</sub> is more affected by the process or operating conditions. In a typical full combustion regenerator condition, most of the HCN and NH<sub>3</sub> should be oxidized to NO/N<sub>2</sub>O as long as enough oxygen is available, especially with the presence of catalysts/metals. Part of the N<sub>2</sub>O and NO can then go through reduction or decomposition to nitrogen.

In the coal combustion process, there is evidence that N<sub>2</sub>O is formed mainly from cyano species, whereas NH<sub>3</sub>-based compounds tend to react toward NO.

Laboratory experiments have shown [32] that HCN is more readily oxidized than ammonia, though both are very reactive and are readily oxidized to  $N_2/NO_x$ . In partial combustion conditions, it is not difficult to imagine the presence of a significant amount of  $NH_3/HCN$ , which subsequently are converted to  $NO/N_2$  in the downstream CO boiler. Thus,  $NO_x$  reduction in partial combustion requires different approaches from full combustion.

## 2.7 Catalysts

Modern FCC catalysts are fine powders with a bulk density of 0.80 to 0.96 g/cc and having a particle size distribution ranging from 10 to 150  $\mu m$  and an average particle size of 60 to 100  $\mu m$ . [37] The design and operation of an FCC unit is largely dependent upon the chemical and physical properties of the catalyst. The desirable properties of an FCC catalyst are:

- Good stability to high temperature and to steam
- High activity
- Large pore sizes
- Good resistance to attrition
- Low coke production

A modern FCC catalyst has four major components: crystalline zeolite, matrix, binder and filler. Zeolite is the primary active component and can range from about 15 to 50 weight percent of the catalyst. The zeolite used in FCC catalysts is referred to as faujasite or as Type Y and is composed of silica and alumina tetrahedra with each tetrahedron having either an aluminum or a silicon atom at the center and four oxygen atoms at the corners.

It is a molecular sieve with a distinctive lattice structure that allows only a certain size range of hydrocarbon molecules to enter the lattice. In general, the zeolite does not allow molecules larger than 8 to 10 nm to enter the lattice [35]. The

faujasite price per specimen size of 30x25x15 mm is €7.00. The catalytic sites in the zeolite are strong acids (equivalent to 90% sulfuric acid) and provide most of the catalytic activity. The acidic sites are provided by the alumina tetrahedra. The aluminum atom at the center of each alumina tetrahedra is at a +3 oxidation state surrounded by four oxygen atoms at the corners which are shared by the neighboring tetrahedra. Thus, the net charge of the alumina tetrahedra is -1 which is balanced by a sodium ion during the production of the catalyst.

The sodium ion is later replaced by an ammonium ion which is vaporized when the catalyst is subsequently dried, resulting in the formation of Lewis and Brønsted acidic sites. In some FCC catalysts, the Brønsted sites may be later replaced by rare earth metals such as cerium and lanthanum to provide alternative activity and stability levels. [35]

The matrix component of an FCC catalyst contains amorphous alumina which also provides catalytic activity sites and in larger pores that allows entry for larger molecules than does the zeolite. That enables the cracking of higher-boiling, larger feedstock molecules than are cracked by the zeolite. The binder and filler components provide the physical strength and integrity of the catalyst. The binder is usually silica sol and the filler is usually a clay. Nickel, vanadium, iron, copper and other metal contaminants, present in FCC feedstocks in the parts per million range, all have detrimental effects on the catalyst activity and performance. [37]

### **2.7.1 Natural Zeolite**

Conventional open pit mining techniques are used to mine natural zeolites. The overburden is removed to allow access to the ore. The ore may be blasted or stripped for processing by using front-end loaders or tractors equipped with ripper blades. In processing, the ore is crushed, dried, and milled. The milled ore may be air-classified based on particle size and shipped in bags or bulk. The crushed product may be screened to remove fine material when a granular product is required, and

some pelletized products are produced from fine material. Producers also may modify the properties of the zeolite or blend their zeolite products with other materials before sale to enhance their performance.

### **2.7.2 Natural Zeolite Consumption**

Approximately 55,800 t of natural zeolite was sold in 2006 in the United States compared with an estimated 58,000 t in 2005. Domestic uses for natural zeolite were, in decreasing order by tonnage, animal feed, pet litter, water purification, odor control, horticultural applications (soil conditioners and growth media), oil absorbent, fungicide or pesticide carrier, gas absorbent, wastewater cleanup, desiccant, and aquaculture. Animal feed, pet litter, and water purification applications accounted for nearly 70% of the domestic sales tonnage. Sales in all except two end-use categories increased.

The largest increases in tonnage sales were for animal feed applications, and the largest declines in tonnage sales were for fungicide and pesticide carrier, pet litter, and water purification applications. The conference Zeolite '06, held in Socorro, NM, highlighted new uses being investigated for natural zeolites.

The discussions ranged from modeling of the crystalline structure of zeolite minerals to application-oriented work. The conference talks covered a range of topics including deposit formation, theoretical modeling of zeolite structures during adsorption of exchange cations, thermodynamics of ion exchange, radiation and thermal effects on the zeolite structure, environmental applications of zeolites, use of natural zeolites in agriculture, and innovative industrial systems that use zeolites. Surfactant-modified zeolites were shown to be a possible solution to the removal of microbial contamination in groundwater, making it potable.

A system for removing pollutants from storm water in densely populated areas was discussed. The zeolite-base system removed polycyclic aromatic hydrocarbons, mineral oils, and heavy metals. Another sector of interest considering

the current issues of petroleum resources was the potential for natural zeolites to pretreat oil sands in Canada.

Currently, the bitumen is separated from the sand by flotation. Because it is so viscous, it must be thinned with toluene for shipment by pipeline to the refineries. A Canadian research group found that chabazite can crack the long-chain organics in the bitumen, thereby reducing the viscosity of the bitumen. Such a system could reduce or eliminate the need for toluene to thin the bitumen and reduce costs [22].