INVESTIGATION OF MECHANICAL PROPERTIES OF ZEOLITE-Y AND ZSM-5 CATALYSTS SYNTHESIZED FROM LOCALLY AVAILABLE CLAYS

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By

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DECLARATION

I declare that the work in this Thesis entitled "investigation of mechanical properties of zeolite-Y and ZSM-5 catalysts synthesized from locally available clays was carried out by me in the Department of Mechanical Engineering, Ahmadu Bello University, Zaria, under the supervision of Dr. M. Dauda, Dr. D. S. Yawas and Prof. A. S. Ahmed (PTDF chair Professor), Department of Mechanical Engineering, Ahmadu Bello University, Zaria.

The information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of this thesis was previously presented for another degree or diploma at this or any other Institution.

Adeiza Ojo YUSUF	-------------------	--------------
Name of student	Signature	Date

CERTIFICATION

The thesis entitled Investigation of Mechanical Properties of Zeolite-Y and ZSM-5 catalysts synthesized from locally available clays by ADEIZA OJO YUSUF meets the regulations governing the award of the degree of Master in Mechanical Engineering of the Ahmadu Bello University, and is approved for its contribution to knowledge and literary presentation.

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Chairman Supervisory Committee Date -------------------------------------

Dr. D. S. Yawas (Signature) -----------------------------------
Member Supervisory Committee Date -------------------------------------

Prof. A. S. Ahmed (Signature) -----------------------------------
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ABSTRACT

This research work was centred on mechanical properties of zeolite-Y and ZSM-5 catalysts synthesized from locally available clays such as Kaolin, Alumina, Gambe and Pindiga respectively. Pressure absorption capacities of the synthesised zeolite catalysts and that of the supportive clays were carried out with the aid of a bourdon gauge machine. Plastimeter machine alongside with a micro tensometer machine were equally used to determine the young’s modulus of the synthesized zeolite catalysts and flow characteristics of the clays. XRD analysis was carried out using Cu K α-radiation to find the textural properties. Results of the analysis indicated that synthesised ZSM-5 catalyst was denser, could absorbed more pressure as compared with synthesized Zeolite-Y catalyst. Though, in terms of flowability, synthesised Zeolite-Y is more effective than synthesise ZSM-5 catalyst. The young’s modulus of the synthesised catalysts was found to lie between 3.5 N/mm^2 – 4.0 N/mm^2. Among the clays analysed, alumina clay had proved a more superior support material for the synthesis of the zeolite catalysts in terms of flowability, porosity, and pore size. XRD analysis indicated that at point 2-theta (point where the highest peak occurred), the peak level of the synthesized zeolite catalysts and that of the referenced zeolite are, at the same level which indicated similarity in their performance ability.

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CHAPTER ONE

INTRODUCTION

1.1 Background to The Study

Zeolites are micro-porous alumina-silicate minerals used for numerous commercial and domestic applications. These include applications in petroleum and petrochemical industries as catalysts, adsorbents and ion exchangers, nuclear industries for nuclear reprocessing, heating and refrigeration, detergents, construction as material additives, medicine, agriculture as a soil treatment, gemstones, as ion-exchange beds in domestic and commercial water purification and softening. The term Zeolite was originally coined in 1756 by Swedish mineralogist Axel Fredrik Cronstedt, who observed that upon rapidly heating the material stilbite, it produced large amounts of steam from water that has been adsorbed by the material. Base on this, he called the material zeolite, from the Greekzep, meaning “to boil” And lithos meaning “stone” (Cejka et al, 2007).

Today, Zeolite-Y is used commercially as catalyst in petroleum refinery because of its high concentration of active acid sites, its high thermal stability and high size selectivity. Zeolite-Y is a synthetic analog to the mineral faujasite and crystallizes with cubic symmetry. It has crystal sizes in the approximate range of 0.2-0.5µm and pore diameter of 7.4À. It thermally decomposes at 793^{o}C (Htay et al, 2008).

The content of the zeolites themselves in zeolites-containing catalysts is not greater than 3-15%. In the preparation of catalyst, zeolites modified with metals (chromium, rhenium, platinum, palladium, etc.) are introduced into a matrix of inorganic oxides such as SiO_2, Al_2O_3, Clays, etc. (Erikh et al, 1988).

In view of this, it is important to ascertain mechanical properties of these zeolite catalysts because, they are subjected to shear and compression forces when applied as fluidized catalytic cracking catalyst (FCC). Shear and compression stresses can lead not only to fracture of the crystals, but also to collapse of the micro-porous volume, loss of crystalline structure and a subsequent loss of catalytic properties (Zhongmin et al., 2002).

Moreover, energy is the vital basis of the development of human society, and is associated with several aspects of the social activities and daily life. With increasing world population and rising living standards, the demand for energy is steadily increasing in the world (energy information administration , 2007). Energy being an important resource, its cheapness and a stable supply is necessary to safeguard the economy and social development.

Developing countries face the double pressure of economic growth and environmental protection as they enter the 21st century. Petroleum became more and more important to the world's economy, so important that today, without a steady flow of oil, most human activities on this planet would grind to a halt. The fuels that are derived from petroleum, supply more than half of the world's total output of energy, such as gasoline, kerosene, and diesel oil provide fuel for automobiles, tractors, trucks, aircraft, and ships (energy information administration, 2007).

At the end of 2006, the world was consuming 84.8 million barrels of oil per day. Global petroleum demand is expected to rise by 1.5 million barrels per day in 2007, an increase of 0.7 million barrels per day above the 2006 growth (energy information administration, 2007).

Majority of machines and equipment made at present are designed to run using liquid fuel. For these reasons, it is important to extract much useful products from crude oil (energy information administration, 2007). Zeolites have porous structures that can accommodate wide varieties of cations, such as Na^+, K^+, Ca^{2+}, Mg^{2+}. These positive ions are loosely held and can readily be exchanged for others in a contact solution. Some common mineral zeolites are analcime, chatazite, dingotilolite, heulandites, natrolite, phillipsite and stilbite. An example of mineral formula is $Na_2Al_2Si_3O_{10}.2H_20$, for natrolite (Weitkamp, 1999).

Zeolite occurred naturally or it could be synthesized. Natural zeolites are formed where volcanic rocks and ash layers react with alkaline ground water or it could be crystallized in post-depositional environments over periods ranging from thousands to millions of years in shallow marine basin (Weitkamp, 1999).

Zeolite possess proton (H^+) ion that make it active when it come in contact with carbon, it will donate that proton ions to carbon and accept electron from it and thereby weaking and breaking the long chain bond of the carbon. In doing that, the zeolite need to possess pore sizes that is not too large neither too small so that any particles that comes in will neither escape or unable to pass through.

Currently, the world's annual production of natural zeolite is about 3 million tonnes. The major producers in 2010 were china (2 million tonnes, South Korea (210,000tonnes), Japan (150,000tonnes), Jordan (140,000tonnes), Turkey (100,000tonnes) Slovakia (85,000tonnes) and United States (59,000tonnes) (international zeolite administration, 2012).

Synthetic zeolites are used as an additive in the production process of woven's mix asphalt concrete. The development of the application started in Germany in the 1990s. It helps by decreasing the temperature level during manufacture and lying of asphalt concrete, resulting in lower consumption of fossil fuels, thus releasing less carbon dioxides, aerosols, and vapors to the atmosphere. Zeolite and related crystalline molecular sieves are widely used as catalyst in the industry since they possess catalytically active sites as well as uniform sized and shaped micro-pores that allow for their use as shape selective catalysts in oil refining, petro-chemistry and organic synthesis.

However, due to the pore size constraints, the unique catalytic properties of zeolites are limited to reactant molecules, having kinetics diameters below 10À (Maesen et al., 2004).

Zeolites have been successful when used as catalyst for cracking process because of their crystalline nature, high surface area, adsorption capacity, and uniform size distribution which enable shape selectivity (Weitkamp, 2000).

1.2 Statement of the Problem

Zeolite catalysts are subjected to wear, high temperature and compression stresses while in operations and thereby lose their catalytic properties with time. Hence it becomes necessary to investigate the mechanical properties of any zeolite catalyst especially those synthesised from locally available materials in order to estimate their possible period of life span before failure.

1.3 The Present Reseacrh Work

The present research examines the ability of the synthesized zeolite catalysts to resist compressive stresses and the extents to which these compressive forces can act on them before deformation occur. The work also examines the morphology of supporting materials used in the synthesis of the zeolite catalysts. The stress and strain analysis of the developed zeolites would be carried out to determined the young's modulus and compare with that of the reference zeolite. Wearing mode of each sample would be determined as a measure of its porosity.

1.4.0 Aim and Objectives

1.4.1 Aim

The aim of this research work is to investigate the mechanical properties of Zeolite-Y and ZSM-5 catalyst synthesised from locally available clays in order to establish its mechanical stability.

1.4.2 Specific Objectives

The specific objectives of the research work are:

i. To examine the morphology of the support materials used for the synthesis of the zeolite catalysts.

ii. To investigate mechanical properties (wear, compression pressure and young's modulus) of the synthesized catalysts.

iii. To determine the resistance of synthesized zeolite catalysts to crushing in a packed-bed reactor.

1.5 Significance of Study

Catalysts used for cracking operations are subjected to high compression stress, rubbing action between the particles and high temperature thereby losing their crystalline structure and micro-porous volume earlier than expected. Hence it becomes necessary to ascertain that any catalysts synthesized especially from local raw materials be characterized mechanically in order to ascertain its mechanical properties that will enable it function satisfactorily before losing its micro-porous volume and its crystalline structure when compared with the commercial zeolite catalysts in use.

1.6 Justification

Most works done on characterization of zeolite catalysts synthesized from local raw materials had not given much attention to their mechanical properties which is a major factor that determines the stability of the catalyst. In this work, necessary mechanical properties (Young modulus, Wear, Plasticity and porosity) had been examined.

1.7 Scope of Study

The scope of this work is to investigate mechanical properties of synthesized Zeolite-Y catalyst, ZSM-5 catalyst and support materials used in synthesis by determining their compression pressure absorption, examine the morphology of the support materials and to determine the Young's Modulus.

1.8 Limitation

Listed below are some of the challenges encountered during the course of the experiment:

i. The presence of substantial void fractions within the crystals allows for unusual mechanical and thermal properties.
ii. In availability of sensitive equipment that can be used for testing at the smallest scale.
iii. Difficulty of preparation of zeolite crystal that is large enough for measurement in standard mechanical testing machine

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction

ZSM-5 means Zeolite Sacony-Mobil number 5 and was discovered in 1971 by Argauer and Landolt. It is intrinsically hydrophobic and organophillic. The particle size is in micron range and pore size of 0.58 nm. It is a high silica zeolite with Si/Al >5 (Yunusa, 2012)

Zeolite Y was discovered in 1842 by Damour. It has a Si/A approximately 2.5, Synthetic counter- part to natural faujasite and extensively used as a component of FCC process in the petroleum industry.

Zeolites are micro-porous crystalline aluminosilicate minerals. The framework of these materials consists of Si^{4+}ions tetrahedrally connected to four oxygen atoms. These tetrahedral are linked to each other by sharing their four oxygen atoms with neighboring tetrahedral, thus forming a framework of interconnected tetrahedral, as shown in Figure 1.

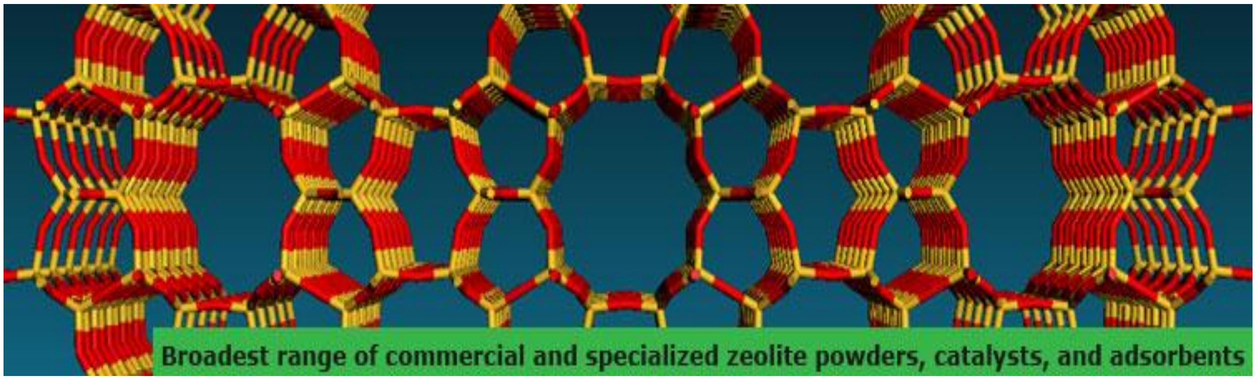


Figure 2.1: Tetrahedral framework of the zeolite.

(Sources: www. Goggle.com: 16/04/2012, 9:40am)

The regular ways in which the tetrahedral are linked within a certain framework give rise to pores in the structure. Because the tetrahedral can be linked in different ways, there are many different zeolite structures. Since the pores are smaller than 2nm in diameter, they are classified as micro-pores according to International Union of Pure and Applied Chemistry (IUPAC) nomenclature.

The micro-pores of zeolites have diameters of the same order of size as the molecules to be converted. Therefore, they can act as molecular sieves, which are often advantageous to induce shape-selectivity in catalysis (Baerlocher et al., 2001).

Scientists are interested in developing methods that can give reliable values of elementary mechanical properties which have been unavailable. Over the last decade, several groups have developed methods and instrument to measure the mechanical properties of increasingly smaller solid bodies.

The mechanical properties of zeolites are important for fundamental scientific reasons. The presence of a substantial void fraction within the crystals allows for unusual mechanical and thermal properties (Raul et al., 2002).

2.2 Properties of Zeolite

Zeolites have a porous structure that can accommodate a wide variety of cations, such as Na^+, K^+, Ca^{2+}, $Mg^{2+}$and others (www.elsevier.com)

These positive ions are rather loosely held and can readily be exchanged for others in contact solution.

Zeolite has the ability to selectively sort molecules based primarily on a size exclusion process, due to a regular pore structure of molecular dimensions of the channels as shown in figure 2.2 and figure 2.3

Figure 2.2: Regular Molecular Pore Structure of Zeolite

(Sources: www.elsevier.com/locate/micromeso: 17/04/2012, 8:25pm)

Figure 2.3: Enlarge crystal form of Zeolite

(Sources: www.elsevier.com/locate/micromeso: 17/04/2012, 8:40pm)

Natural zeolites are formed where volcanic rocks and ash layers react with alkaline ground water. Also, zeolites crystallize in post – depositional environments over period ranging from thousands to millions of years in shallow marine basins. Though, natural zeolites are rarely pure and are contaminated to varying degrees by other minerals, metals, quartz or other zeolites.

For this reason, naturally occurring zeolites are excluded from many important commercial applications where uniformity and purity are essential (Satterfield et al., 1991).

2.3 Catalysis

A catalyst is a substance that increases the rate of a reaction without being consumed itself. For heterogeneous material, catalyst is in a different phase as the reactants and products. Usually, the catalyst is a porous solid while the reactants and products are in the liquid or gas phase (van Sante et al., 1999)

. A catalytic reaction can be divided roughly into the following steps:

- diffusion into the porous catalyst particle
- adsorption on the catalyst

- the actual reaction
- desorption from the catalyst and
- Diffusion of products from the catalyst particle.

From these five steps, two main factors that influence the reaction are:

i. Interaction with the active site of the catalyst (of both reactant and product) and
ii. Diffusion (to and from the active site).

For a good catalyst there is a delicate balance between the interaction between the reactant and the active site and the interaction between the formed products and the active site.

The interaction of the reactants with the active site is needed for the breaking of bonds. If the interaction of the products with the active site is too large, the products will not be able to desorb from the active site, thus causing a blockage of the active site.

Optimal interactions with reactants and products will give a lowering of the overall activation energy of the reaction and thus an increase of the reaction rate. If the reaction at the active site is faster, the diffusion of the reactants and products has to keep pace in order to maximizing the final yield of products per unit of time. If the reaction rate is much higher than the diffusion, the process will become diffusion limited and the final rate and selectivity of the reaction will be determined by the rate of diffusion (van Sante et al., 1999)

In particular, if diffusion limitation prevails, intermediate products may accumulate in the catalyst pores thus promoting secondary reactions.

Therefore, not only the nature of the active site but also the architecture of the pores strongly affects the performance of a catalyst (Van Sante et al., 1999).

2.4 Catalytic Cracking

Catalytic cracking is the process of breaking down the larger, heavier and complex hydrocarbon molecules into simpler and lighter molecules to produce higher value products by using heat, pressure and a catalyst (Van Sante et al., 1999).

The essence is to increase the octane rating of the oils or motor fuels.

There are two types of catalytic cracking and these are:

i. Fixed–bed processes in which the catalyst is in the form of small lumps or pellets in layers or beds.
ii. Fluid catalytic cracking processes which are characterized by the use of finely powdered catalyst through the processing unit (van Santen et al, 1999).

2.5 Uses of Zeolite

2.5.1 Commercial and Domestic; Zeolites are widely used as ion-exchange beds in domestic and commercial water purification, softening, and other applications. In chemistry, zeollites are used to separate molecules (only molecules of certain sizes and shapes can pass through), and as traps for molecules so they can be analyzed.

Zeolites have the potential of providing precise and specific separation of gases including the removal of H_2O, CO_2 and SO_2 from low-grade natural gas streams.

2.5.2 Petrochemical industry

Synthetic zeolites are widely used as catalysts in the petrochemical industry, for instance in fluid catalytic cracking and hydrocracking. Zeolites confine molecules in small spaces, which causes changes in their structure and reactivity.

Catalytic cracking uses reactor and a regenerator. Feed is injected onto hot fluidized catalyst where large gasoline molecules and olefins were mixed. The vapour-phase products are separated from the catalyst and distilled into various products. The catalyst is circulated to a regenerator where air is used to burn coke off the surface of the catalyst that was formed as a by-product in the cracking process. The hot regenerated catalyst is then circulated back to the reactor to complete its cycle (Wang et al., 2006).

2.5.3 Nuclear industry

Zeolites have uses in advanced reprocessing methods, where their micro-porous ability to capture some ions while allowing others to pass freely allowing many fission products to be efficiently removed from nuclear waste and permanently trapped.

Once they are loaded with trapped fission products, the zeolite-waste combination can be hot pressed into an extremely durable ceramic form, closing the pores and trapping the waste in a solid stone blocks (Sargentis et al., 2009).

2.5.4 Heating and Refrigeration

Zeolite can be used as solar thermal collectors and for adsorption. In this application, their high heat of adsorption and ability to hydrate and dehydrate while maintaining structural stability is exploited. This hygroscopic property coupled with an inherent exothermic (heat-producing) reaction in transforming from a dehydrated to a hydrated form make natural zeolites useful in harvesting waste heat and solar heat energy.

2.5.5 Construction

Synthetic zeolite is also being used as an additive in the production process of warm mix asphalt concrete. The development of this application started in Germany in the 1990s. It helps by decreasing the temperature level during manufacture and laying of asphalt concrete, resulting in lowering consumption of fossil fuels, thus releasing less carbondioxide, aerosols, and vapors. In addition, the use of synthetic zeolite in hot mixed asphalt leads to easier compaction and, to a certain degree, allows cold weather paving and longer hauls.

When added to Portland cement as a pozzolan, it can reduce chloride permeability and improve work ability.

Zeolites are also used in many others field such as:

i. Production of detergent.
ii. Gemstones.
iii. medical
iv. agriculture
v. domestic pet care, and
vi. Cat litter.

Moreover, it reduces weight and helps moderate water content while allowing for slower drying which improves break strength.

2.6 Review of Past Works

Evan et al (2005) carried out an investigation on the formation of a synthetic zeolite by reacting silica glass and aqueous solution of amines and sodium hydroxide for the preparation of large crystals of siliceous zeolites in the presence of n-butylamine, at 160^{oc}, and a needle shaped crystals of zeolite ZSM–22 were formed. By adjusting the amount of n-butylamine used in crystallization, crystals of up to 400μm in length may be formed after 5weeks of reaction. Calcinations in air at 800^{oc} produces crystals free of temperate and scanning electron microscopy shows the samples to be free of cracks, with no evidence of loss of integrity of the specimens.

Mike et al (2006) carried out a preliminary investigation into mechanical properties of clay reinforced with natural fibres by using Laser light scattering method and found that, clay can be used as binder compound in walls built of cob or rammed earth.

Huat et al (2005) milled flax to fractions with different length and mixed with clay, sand and water. Dried the clay mix cube and carried out a compression test and measured the compressive strength with a testing system machine (INSTRON 8516). They found out that, reinforcement interacts with the soil to produce a composite material in which the roots are fibers of relatively high tensile strength and in adhesion inherent in a matrix of lower shear strength soil.

Martens *et al* (1989) reported results on thermal cracking of asphaltenic residue from synthetic crude obtained by coal liquefaction. A second-order reaction was suggested for asphaltene cracking at temperature 425, 435 and450^{0}C. The amount of asphaltene and yields of products, oil, gas and coke were presented as a function of residence time.

Wang and Anthony (2003) studied the thermal cracking of asphaltenes by re-examining data obtained by Martens *et al* (1989). They derived the concentration and conversion of residence time relation for the yields involving the secondary cracking of oil by direct integration of the rate equations. They reported that thermal cracking of asphaltenes occurs is an important heavy-oil upgrading processes such as coking and via breaking. Their analyses confirmed that at lower temperatures the three-lump model which considered parallel reactions of oil, gas and coke formation described the cracking behavior whereas at higher temperature the secondary cracking of oil may be considered. This development has the potential to be useful in describing thermal-cracking processes for heavy oils.

Shank and Wuff (1949) measured radial pressures on the die and estimated coefficient of friction. Their results were at complete variance with those of Duwez and Zwell (1949) who studied the pressure distribution in compacting copper powder by means of small piston dynamometer and resistance sensitivity strain gauges. It was established that die varies almost linearly from the bottom to the compact, a fact to support the influence of friction in the compaction of metal powders.

Lambros et al (2002) carried out a micromechanical compressive response on siliceous ZSM – (silicate) using in – house built micromechanical tester on a prepared single – crystals (300μm).

They found that, no differences in Young’s modulus of synthesized crystal and calcined crystals were detected and little mechanical anisotropy was observed.

Alireza et al (2011) investigated the effect of modified clay on the mechanical efficiency of epoxy resin by using X–ray diffraction and microscopic transient electron method and discovered that modified clay distribution in polymer area is intercalated kind. Examination of the results of mechanical tests show that the existence of modified clay in epoxy area increase pressure yield strength, tension module and Nano composite fracture toughness in relate of pure epoxy. By microscopic examinations, it is recognized too that the action of toughness growth is due to crack deflection, formation of new surfaces and fracture of clay piles.

Procio et al (1993) carried out wear and mechanical test on metal matrix composite and it was found that about 90% of wear reduction occurred in composite with 30% SiC compared with pressed matrix.

Zhongmin et al (2003) carried out an investigation on the mechanical testing of smallest scale material by the uses of atomic probe microscope along side with techniques of indentation. They found out an estimate of semi–quantitative mechanical properties of the surface of small particles, such as silica, sol-gel silica and organic-temperate mesoporous silica.

Michael et al (2006) carried out a mechanical testing on Direct Metal Laser-Sintering (DMLS) parts, and reported that Young’s Modulus E (G pa) estimated from the slope of the stress/strain curve in the elastic region is a measure of the stiffness of a material and concluded that the higher the modulus of elasticity, the more resistant the material behaves towards an external stress.

2.7 Catalyst Support

Platinum and platinum containing bimetallic catalysts supported on alumina are widely used for naphtha reforming and for heavy linear alkanes ($C_{10} - C_{15}$) dehydrogenation in the petrochemical industries.

However, recently this kind of catalysts dehydrogenation of hydrocarbons are an important commercial process because of the great and expanding demand for dehydrogenated hydrocarbons for use in the manufacture of various chemical products.

The difference between dehydrogenating and reforming catalysts relies mainly on the acidity of the support (such as isomerization cracking and polymerization) must be inhibited in order to increase the yield to olefins in the dehydrogenation processes (Pieck et al., 2005).

In the case of dehydrogenation of long chain alkanes, a multimetallic alumina supported platinum catalyst is implemented, which contains In and Sn promoters as platinum modifiers. Promoters improve the activity, selectivity and stability of platinum- alumina catalyst (Gokak et al, 1996).

Alumina is the most widely used support material for the dehydrogenation catalysts because

of its superior capability to maintain a high degree of platinum dispersion which is essential for achieving high dehydrogenation activity (Akia et al., 2011).

The addition of alkaline and alkaline – earth ions (for example, Li) to gamma Al_2O_3 selectivity poisons the isomerization active sites, without affecting the dehydrogenation capacity of the catalytic system (Akia et al., 2011).

A properly designed catalyst should posses the essential attributes of activity, stability, selectivity and regenerability. The most popular in application of the various supports is gamma-alumina due to its high surface area (15 – 300m^2/g), high thermal stability, outstanding mechanical properties and nature of interaction with the Zeolite active phase (Salahudeen, 2012).

2.8 Supported Catalyst

The feature of supported catalysts is especially important with regard to precious – metal catalysts, because it allows more effective utilization of the metal than can be achieved

Bulk- metal systems. The case of base- metal catalysts, the use of the support is often primarily aimed at improving the catalyst stability. This can be achieved by suitable interaction between the active material and the support.

For instant, unsupported copper oxide is a very active oxidation catalyst but suffers from thermal instability at high temperatures. However, when copper oxide is supported on a high-surface-area alumina, its thermal stability is improved.

A wide range of techniques has been employed for incorporation of a catalytically active species onto supported materials such as Alumina and Gamma-Alumina (Pieck et al., 2005).

Alumina possesses the ability to maintain high degree of dispersion of hydrogen ions which is essential for achieving high dehydrogenation activity.

2.9 Uses of Kaolin Clay

Kaolin is used in ceramics, medicine, coated paper, as a food additive in toothpaste, as a light diffusing material in white incandescent light bulbs, and in cosmetics (Htay,2008). It is generally the main component in porcelain. It is also used in paint to extend titanium dioxide (TiO_2) and modify gloss levels; in rubber for semi-reinforcing properties; and in adhesives to modify rheology. Kaolin was used in the production of common smoking pipes in Europe and Asia. The largest use is in the production of paper, including ensuring the gloss on some grades of paper. Commercial grades of kaolin are supplied and transported as dry powder, semi-dry noodle or as liquid slurry.

Kaolinite can contain very small traces of uranium and thorium, and is therefore useful in radiological dating. While a single magazine made using kaolin does not contain enough radioactive material to be detected by a security-oriented monitor, this could result in the possibility of a truckload of glossy paper occasionally tripping an overly-sensitive radiation monitor. Kaolinite has also seen some use in organic farming, as a spray applied to crops to deter insect damage, and in the case of apples, to prevent sun scald. Kaolin is extensively used as a paint or white wash in traditional stone masonry homes in Nepal. The most common method is to paint the upper part with white kaolin clay and middle with red clay. The red clay may extend to the bottom or the bottom may be painted black (Bawa, 2012).

The XRD pattern of kaolin and metal kaolin is shown in figure 2.4 and SEM image of the kaolin clay is shown in figure 2.5. The SEM image had shown that natural kaolin contains impurities such as silica sol, iron, and slag which need removing before it could be used as supportive material in the synthesis of zeolite catalysts.

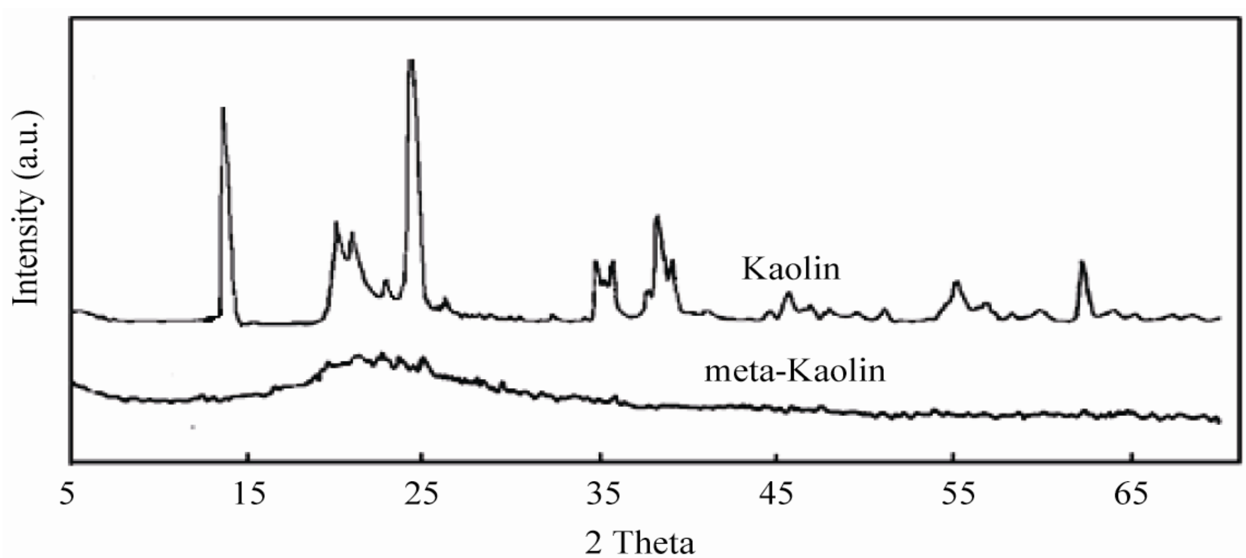


Figure 2.4: XRD pattern of kaolin and meta-kaolin

(Source: Bawa, 2012)

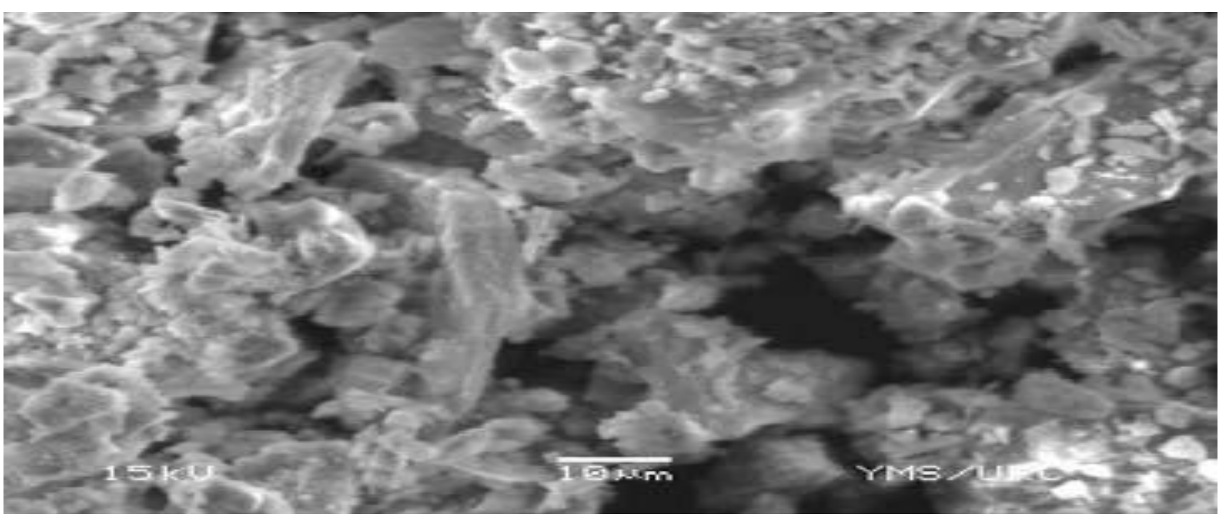


Figure 2.5: SEM Image of Kaolin clay

(Source: Bawa, 2012)

2.10 Compaction and Mechanical Properties

Articles on the modelling of tablet compaction had shown experimentally that inputs related to the constitutive model of the powder and the powder/tooling friction was determined. The continuum-based analysis of tableting makes use of an elasto-plastic model, which incorporates the elements of yield, plastic flow potential, and hardening, to describe the mechanical behavior of microcrystalline cellulose over the range of densities experienced during tableting. Specifically, a modified Drucker-Prager/cap plasticity model, which includes material parameters such as cohesion, internal friction, and hydrostatic yield pressure that evolve with the internal state variable relative density, was applied. Linear elasticity is assumed with the elastic parameters, Young's modulus, and Poisson's ratio dependent on the relative density. The calibration techniques were developed based on a series of simple mechanical tests including diametrical compression, simple compression, and die compaction using an instrumented die. The friction behaviour is measured using an instrumented die and the experimental data are analyzed using the method of differential slices (Wiley-Liss et al, 2004).

CHAPTER THREE

MATERIALS AND METHODS

3.1 Materials

All materials used in this research were obtained from Petroleum Trust Development Fund (PTDF) Chair Laboratory, Department of Chemical Engineering, Ahmadu Bello University, Zaria.

Listed below are materials used in this research work:

a. Synthesized Zeolite-Y

b. Synthesized ZSM-5

c. Commercial Zeolites

d. Alumina

e. Kaolin clay

f. Gambe clay

g. Pindiga clay

h. Compounded Pure chemical ZSM-5

i. ZSM-5 compounded Gamma alumina

j. Compounded commercial Zeolite-Y

3.2 Equipment

The following equipment were used in this research work:

- Bourdon Gauge Machine; this machine was used to measured the absorption pressure of clays, synthesized catalysts and the referenced catalysts used in this research work.
- Plastimeter B70; the machine was used to measure the flow characteristics of the various clays analyzed.
- Micro-Electronic Tensometer; this machine was used to determined stress/stain values of the synthesized zeolite-Y and ZSM-5 catalyst.
- X– Ray powder diffractometer; this equipment was used to reveal the intensity count and peaks of the synthesized zeolite-Y and ZSM-5 catalyst analyzed in order to indicate the level of their crystalline structure.

3.3 Methods and Experimental Procedures

3.3.1 Uniaxial Compression Test

A micro deformation tester was use to carried out uniaxial compression tests in which a mechanical force is exerted on the crystal by an actuator and measured by a load cell. The actuator is programmed to control the rate of deformation which is measured by a linear voltage differential translator (LVDT) in form of relative displacement according to the method of Wang et al (2003). In the absence of this micro deformation tester, Bourdon Gauge was improvised to determine the compression pressure at the point of deformation of the crystal in the form of pressure displacement. A maximum load of 30N was kept constant while the loading was done repeatedly with an increase of 5N and the displacement was measured in terms of pressure in mmH_2O. Material with high compression values possess high density, less porous, can withstand greater external pressure and can perform relatively longer catalytically before losing its crystalline structure, which is an indication of possessing higher mechanical strength. Though, in terms of flow ability, it may not serve effectively. The compression pressure of each of the sample of Zeolite-Y, ZSM-5, Alumina and Kaolin (from Kankara clay) was determined by the use of the burdon gauge. The pressure is measured in terms of mmH_20 which serve as the compression strain in respect to water movement. The water did not dissolve the samples; it only served as a transmitting medium. Various standard loads were applied on each of the samples. 0.5g of each sample was measured and filled into the cylinder of the gauge, while load was applied and reading was taking respectively. The result obtained is as shown in Table 4.1.

Plate 3.1: Bourdon Gauge Machine

3.3.2 Flow Characteristics

The plastimeter B70 is a miniature extruder coupled to a compression cage, which provides a method of measuring flow characteristics of plastics, rubber, bitumen, clay etc. It has a scale that indicates the stress applied and the strains in the material before fracture (Atkinson et al, 1973)

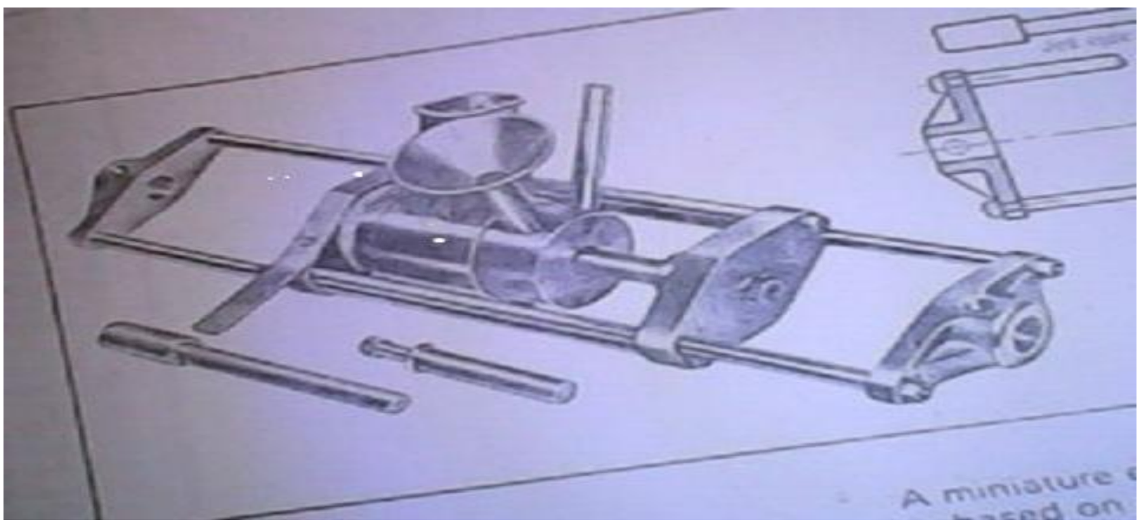


Plate 3.2: Plastimeter B70 machine

Source: Atkinson, E.B and Nancarrow, H.A. (1973)

Two samples were prepared as follows:

Zeolite-Y, Alumina and Kaolin were measured (2g each), mixed and label sample A.

ZSM-5, Alumina and Kaolin are measured (2g each), mixed and label sample B.

Each of the samples was fed into the cylinder of the plastimeter through the charging funnel.

Force was applied through the plunger via the ram and pushes the powder material into the compression chamber. At every 5N of force applied, the strain values were recorded as shown in Table 4.3.

3.3.3 The Stress - Strain Analysis

The stress/strain analysis was carried out at Defence Industries Corporation of Nigeria (DICON) Kaduna, by the use of a microelectronic tensometer machine. A mould was prepared for the preparation of samples of synthesized zeolite catalysts (zeolite-Y and ZSM-5) and the support materials (Gambe clay, Pindiga clay, Alumina and Kaolin clay) in accordance to the machine specification (ASTM D3039-89). Specimens were placed in between the machine load cells and appropriate load cells were selected. Also, appropriate fixtures were loaded for the compression test for push off and push in load tests. After loading specimens, it was ensured that the display on the micro controller flows at 0.0 in order to avoid error due to parallax. The machine was switched ON and readings were taking immediately and recorded as shown in table 4. 2.

3.3.4 X-Ray Diffractometer Analysis

X-ray diffraction (XRD) patterns for gamma alumina base ZSM-5, activated alumina based ZSM-5 and rice haust ash base zeolite-Y were obtained using Empyreal, Analytical diffractometer employing Cu Kα radiation ($\lambda = 0.154$nm) at the National Geoscience Research Laboratories (NGRL) Kaduna. The chart obtained for various X-ray diffraction analyses carried out are presented in figure 4.12 – figure 4.17.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Results

Results of various experiments carried out in this research are presented and discussed in this chapter.

Data obtained for various experiments carried out are presented in tables 4.1-4.9 and figure 4.1 - 4.17

4.1.1 Compressive pressure absorption of Kaolin clay, Zeolite-Y, ZSM-5 and Alumina clay

The result of the compressive pressure absorption test carried out on Kaolin clay, Zeolite-Y, ZSM-5 and Alumina clay is presented in Table 4.1, and plotted in figure 4.1 and figure 4.2.

Table 4.1: compressive pressure of (Kaolin, Zeolite-Y, ZSM-5 and Alumina) in respect to the applied loads:

Load (N)	Kaolin (mmH_2O)	Zeolite-Y (mmH_2O)	ZSM-5 (mmH_2O)	Alumina (mmH_2O)
5.00	2.00	1.00	2.00	1.50
10.00	4.50	1.50	4.10	3.50
15.00	6.80	2.20	6.50	6.20
20.00	8.90	3.50	8.50	8.20
25.00	9.00	4.50	9.80	10.20
30.00	11.00	7.00	10.50	11.00

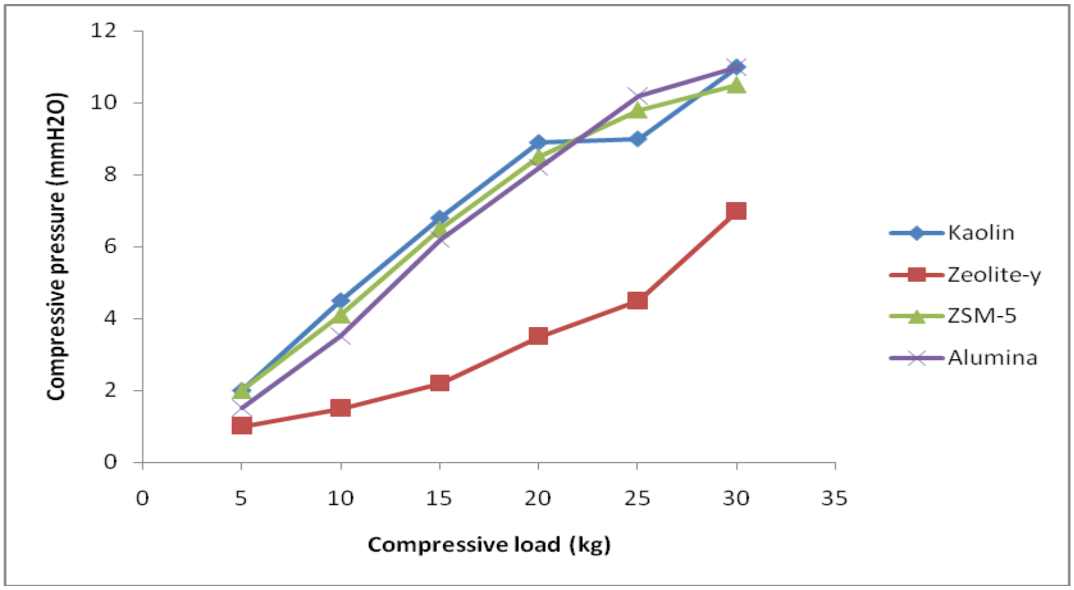


Figure 4.1: Variation of compressive pressure against compressive Load (Kg).

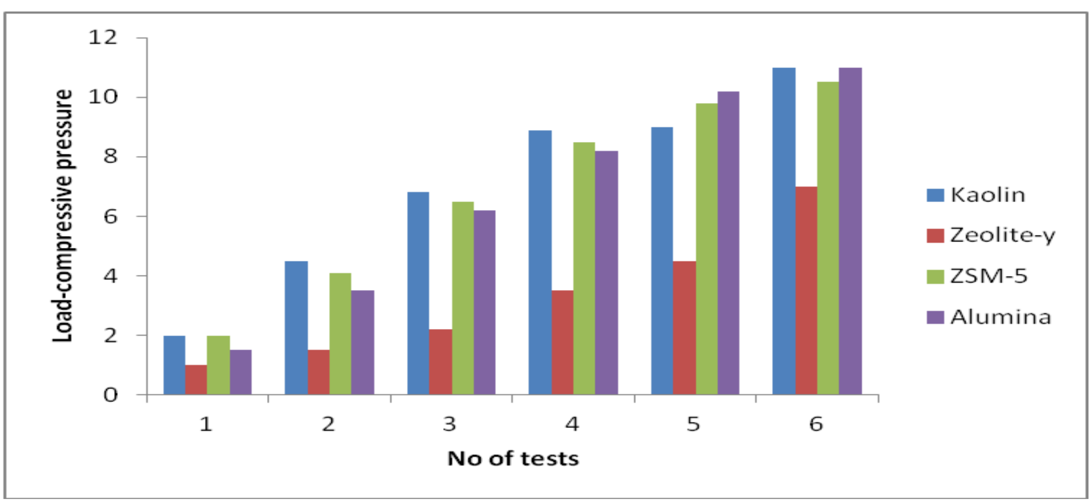


Figure 4.2: compressibility of kaolin clay, zeolite-y, ZSM-5 and alumina clay against load.

4.1.2 Stress / Strain Analysis on Gambe clay, Pindiga clay and Kaoli clay

The results of the stress / strain experiment carried out on three clays are presented in table 4.2, figure 4.3 and figure 4.4.

Table 4.2: Load (Kg) on gambe, pindiga and kaolin clays against strain

Gambe clay		Pindiga clay		Kaolin clay	
Load (Kg)	Strain	Load (Kg)	Strain	Load (Kg)	Strain
24.00	0.00	28.00	0.00	32.00	0.00
21.00	0.60	22.00	1.50	25.00	1.40
16.00	1.75	15.00	3.40	15.00	3.80
13.00	3.60	11.00	5.40	6.00	6.00
12.00	3.80	10.50	6.50	5.00	9.00

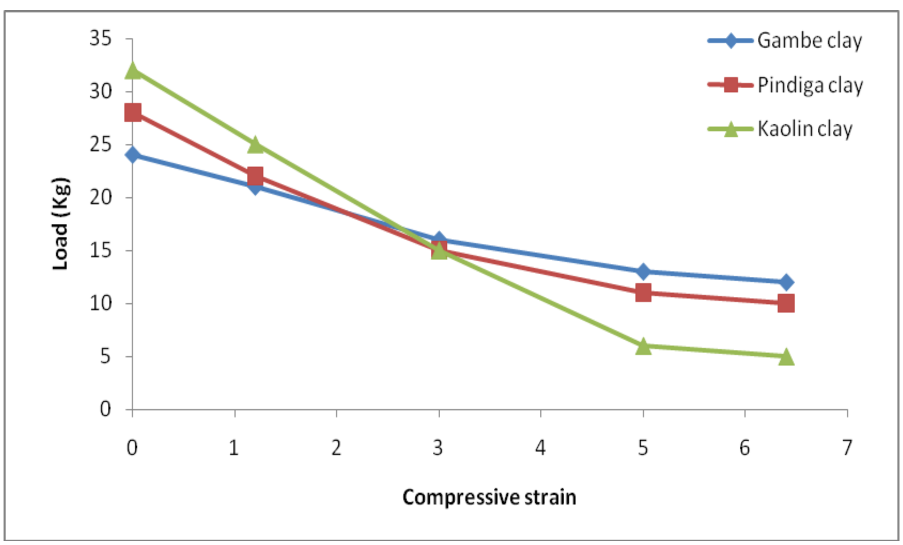


Figure 4.3: load on gambe, pindiga and kaolin clays against compressive strain

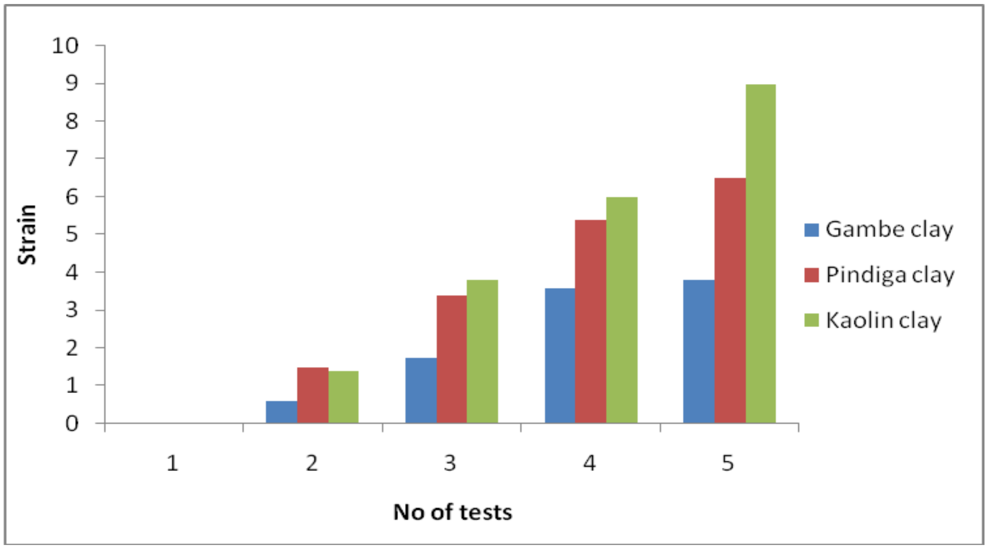


Figure 4.4: load against strain behaviour of gambe, pindiga and kaolin clays.

4.1.3 Stress / Strain Analysis on Sample A and Sample B

The results of the stress / strain test carried out on sample A (contains Zeolite-Y, Alumina and Kaolin clay) and sample B (ZSM-5, Alumina and Kaolin clay) is presented in table 4.3 and figure 4.5.

Table 4.3: stress against strain on sample A and B

Stress (N)	Sample A Strain (%)	Sample B Strain (%)
0.00	0.00	0.00
5.00	7.00	6.00
10.00	15.00	8.00
15.00	20.12	10.00
22.00	22.00	12.20
25.00	25.00	14.00
30.00	27.50	16.00
35-00	30.00	18.10
40.00	32.00	20.00
45.00	36.00	22.00
50.00	40.40	24.00
55.00	50.00	26.50
60.00	56.00	27.50

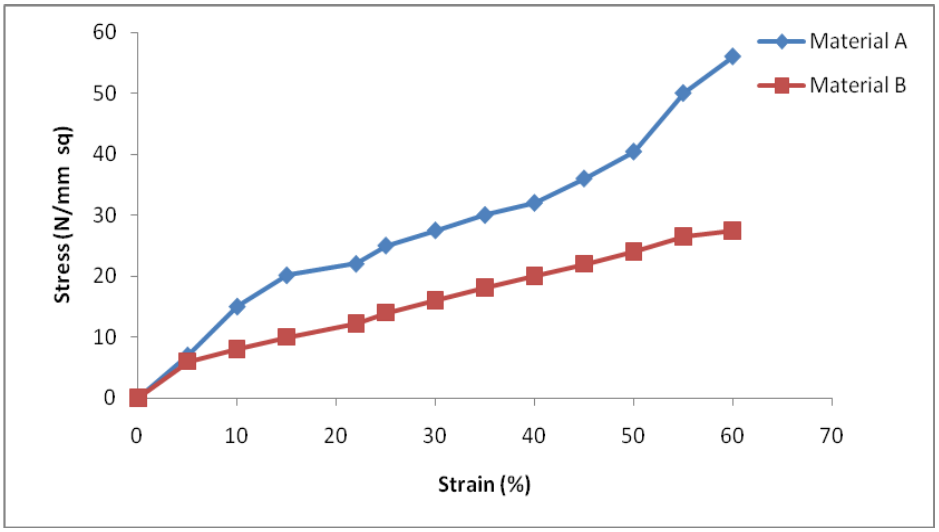


Figure 4.5: stress against strain for samples A and B

4.1.4 Compression test on Zeolite-Y_n, Gambe clay and Pindiga clay

The results of compression pressure absorption test carried out on Zeolite-Y_n, Gambe clay and Pindiga clay is presented in table 4.4 and figure 4.6

Table 4.4: compressive pressure (mmH_2O) of clays and zeolite-Y_n against Load (N)

Load (N)	Zeolit-Y_n (mmH_2O)	Gambe clay (mmH_2O)	Pindiga clay (mmH_2O)
5	1.80	1.90	2.10
10	4.10	4.10	4.10
15	6.00	5.50	6.00
20	8.00	7.50	7.50
25	10.00	9.90	9.80
30	12.10	11.50	11.10

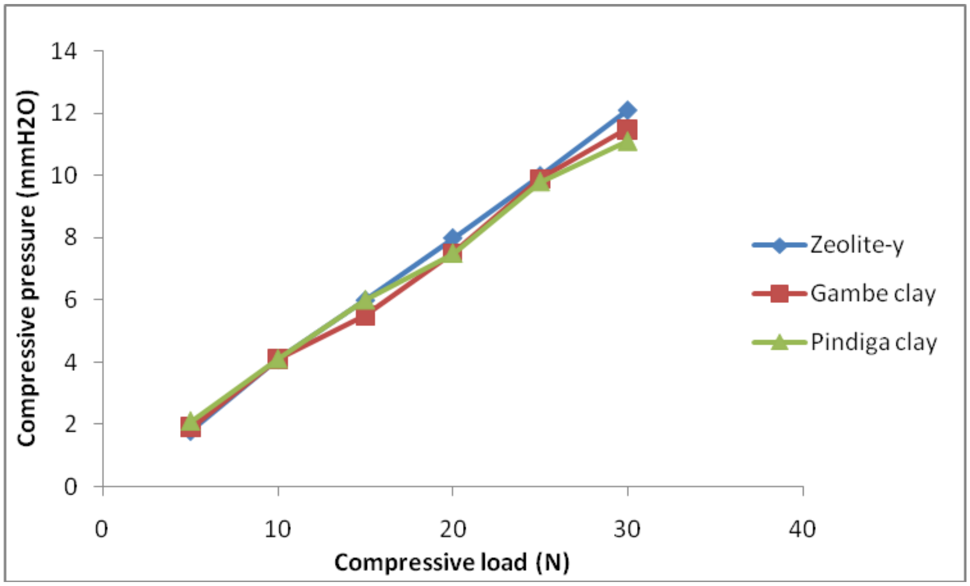


Figure 4.6: compressive pressure (mmH_2O) against compressive load (N) for two clays and Zeolite-Y_n

4.1.5 Analytical Comparison between CPD commercial Zeolite-Y, CPD pure chemical ZSM-5 and CPD ZSM-5 ϒ alumina.

The results of stress absorption capacity comparison between CPD commercial Zeolite-Y, CPD pure chemical ZSM-5 and CPD ZSM-5 ϒ alumina is presented in table 4.5 and figure 4.7.

Table 4.5: comparison between CPD commercial Zeolite-Y, CPD pure chemical ZSM-5 and CPD ZSM-5 ϒ alumina

Load (N)	Compounded commercial Zeolite-Y (mmH_2O)	Pure chemical compounded ZSM-5 (mmH_2O)	Compounded ZSM-5 γ alumina (mmH_2O)
5	1.50	2.20	2.00
10	2.50	3.50	4.20
15	4.90	6.10	6.60
20	7.00	7.80	8.50
25	9.00	10.50	10.50
30	11.20	11.80	12.50

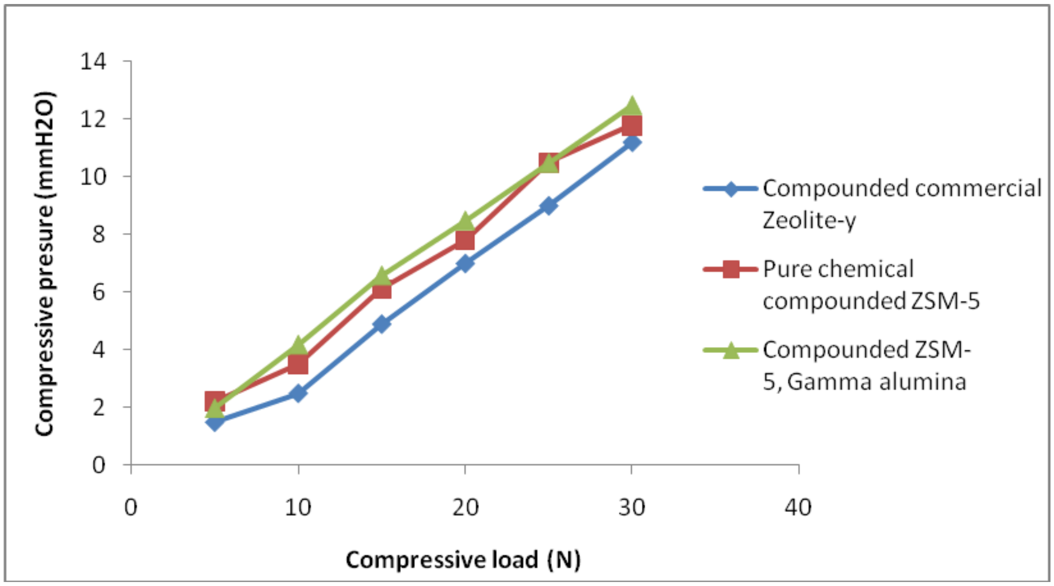


Figure 4.7: graph of compressive pressure against compressive load for compounded commercial zeolite-y, pure chemical compounded ZSM-5 and compounded ZSM-5 γ alumina.

4.1.6 Comparison of compressive pressure against Load for Zeolite-Y_n, Zeolite-Y_s and Compounded commercial Zeolite-Y

The results of the compressive pressure against load test of the three Zeolites are presented in table 4.6 and figure 4.8.

Table 4.6: comparison of compressive pressure (mmH_2O) against Load (N) of Zeolite-Y_n, Zeolite-Y_s and Compounded commercial Zeolite-Y

Load (N)	Zeolite-Y_n (mmH_2O)	Zeolite-Y_s (mmH_2O)	CPD-Commercial Zeolite-Y (mmH_2O)
5	1.80	1.00	1.50
10	4.10	1.50	2.50
15	6.00	2.20	4.90
20	8.00	3.50	7.00
25	10.00	4.50	9.00
30	12.10	7.00	11.20

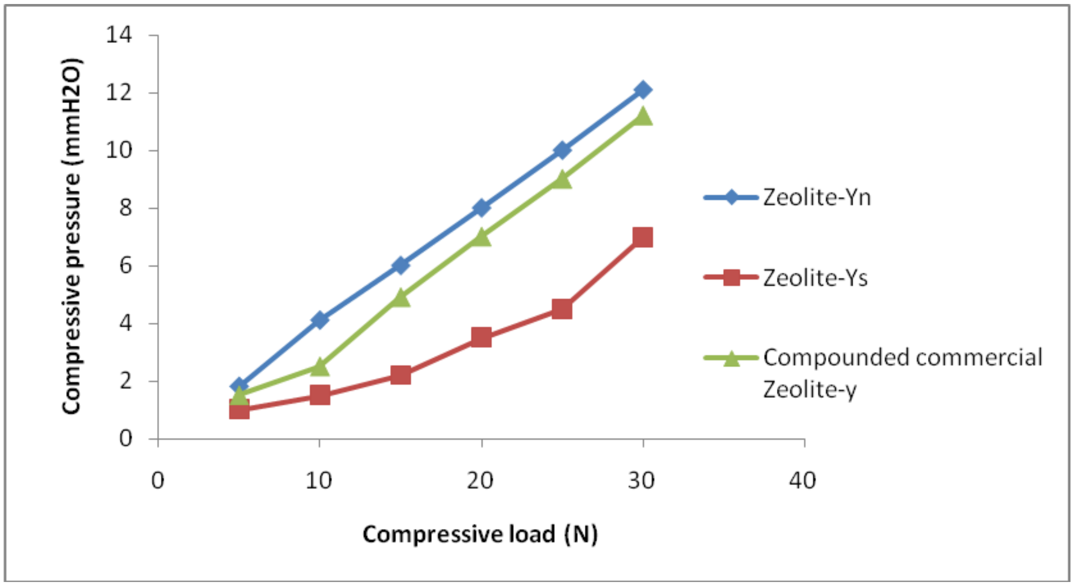


Figure 4.8: compressive pressure against compressive loads of the three zeolite-y compared

4.1.7 Pressure absorption comparison between Gambe clay, Pindiga clay, Kaolin and Alumina clay

The results obtained on the pressure absorption capacity test carried out are presented in table 4.7 and figure 4.9.

Table 4.7: comparison between Gambe clay, Pindiga clay, Kaolin and Alumina in terms of pressure absorption

Load (N)	Gambe clay (mmH_2O)	Pindiga clay (mmH_2O)	Kaolin (mmH_2O)	Alumina (mmH_2O)
5	1.90	2.10	2.00	1.50
10	4.10	4.10	4.50	3.50
15	5.50	6.00	6.80	6.20
20	7.50	7.50	8.90	8.20
25	9.90	9.80	9.00	10.20
30	11.50	11.10	11.00	11.00

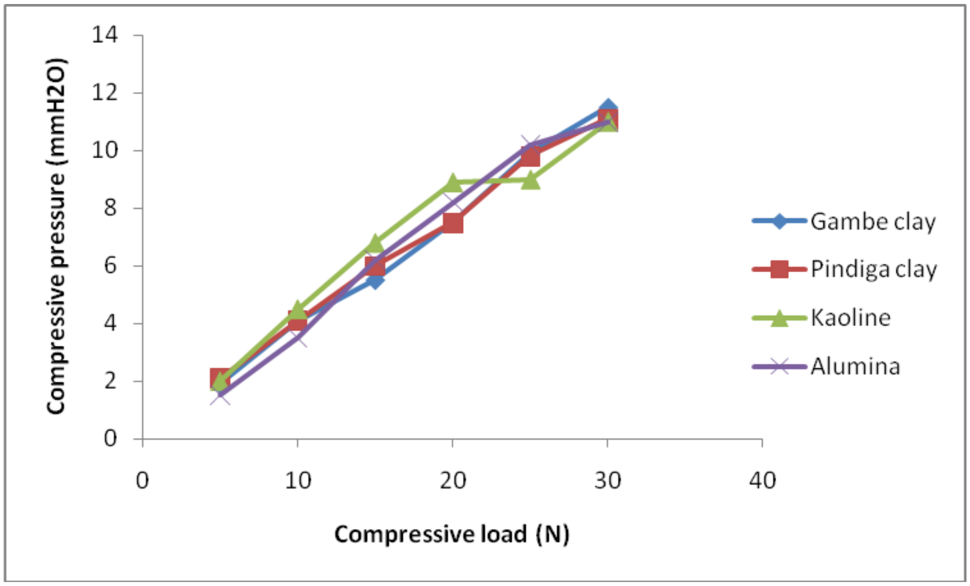


Figure 4.9: compressive pressure against compressive load for the four clays

4.1.8 Pressure absorption capacity of Synthesized ZSM-5, pure chemical ZSM-5 and Compounded ZSM-5 γ alumina

The results of the comparison test carried out on Synthesized ZSM-5, pure chemical ZSM-5 and Compounded ZSM-5 γ alumina are presented in table 4.8 and figure 4.10.

Table 4.8: comparison between Synthesized ZSM-5, pure chemical ZSM-5 and Compounded ZSM-5 γ Alumina:

Load (N)	Synthesized ZSM-5 (mmH_2O)	Pure chemical ZSM-5 (mmH_2O)	Compounded ZSM-5 γ alumina (mmH_2O)
5	2.00	2.20	2.00
10	4.10	3.50	4.20
15	6.50	6.10	6.60
20	8.50	7.80	8.50
25	9.80	10.50	10.50
30	10.00	11.80	12.50

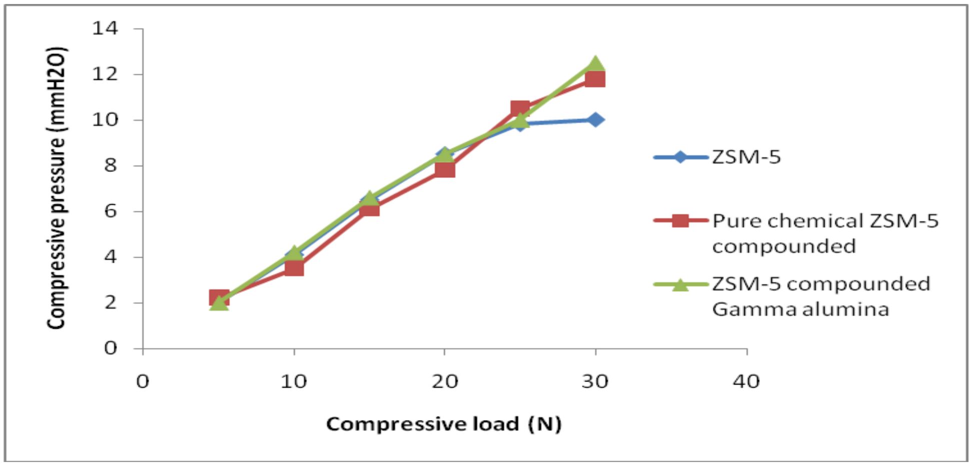


Figure 4.10: compressive pressure against compressive load for different ZSM-5 catalysts

4.1.9 Stress / Strain Analysis on Zeolite-Y_n

The results of the stress / strain test carried out on Zeolite-Y_n are presented in table 4.9 and figure 4.11.

Table 4.9: stress against strain of Zeolite-Y_n

Stress (N)	Strain (%)
0	0
5	6
8	9
10	14
15	17
20	21
22	22
25	24
30	30
32	31
38	36
40	39
45	43
48	47
52	49
56	54
60	58

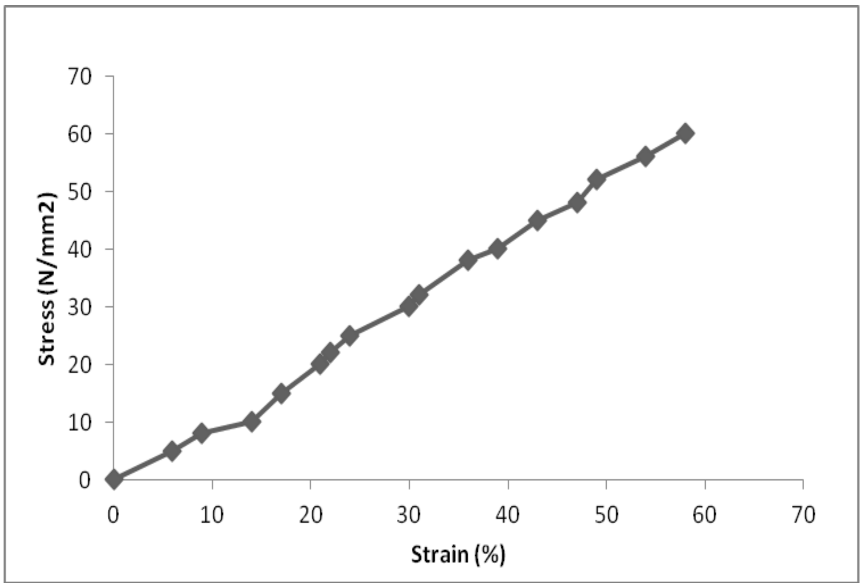
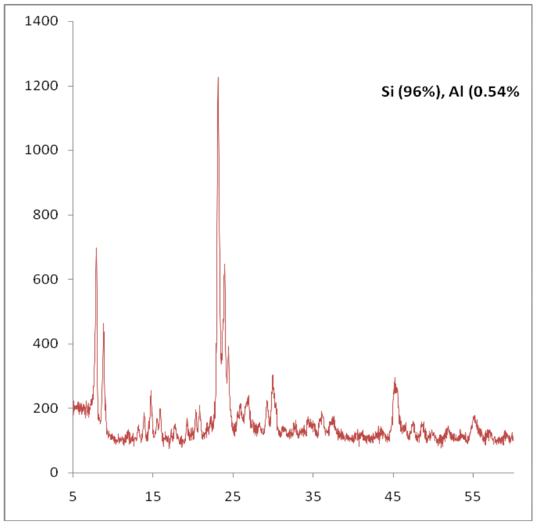


Figure 4.11: graph of stress against strain for Zeolite-Y_n

4.1.10: X-ray diffractometer (XRD) analyses

The pattern of the X-ray diffractometer analyses carried out on gamma alumina based ZSM-5, activated alumina based ZSM-5 and rice haust ash based zeolite-Y are presented in figure 4.12, figure 4.14 and figure 4.16.



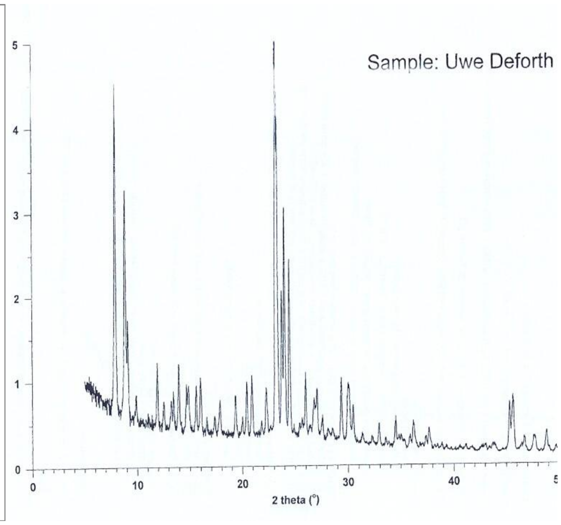
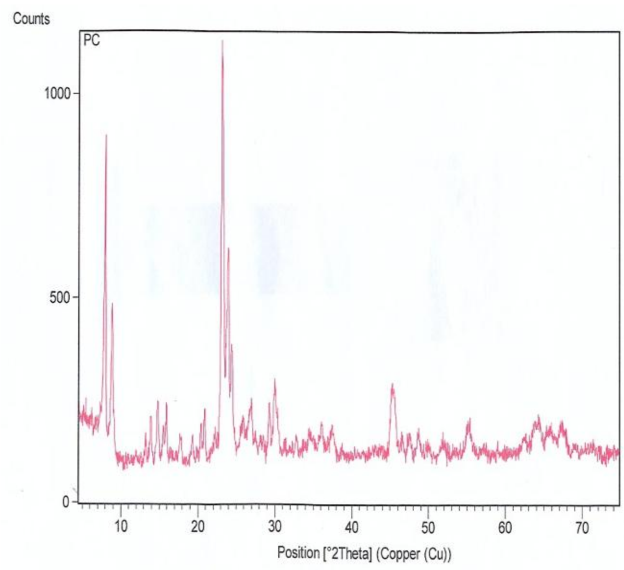


Figure 4.12: XRD pattern for gamma Alumina based ZSM-5 zeolite

Figure 4.13: XRD pattern for reference ZSM-5 zeolite

(Source: Robson et al, 2001)



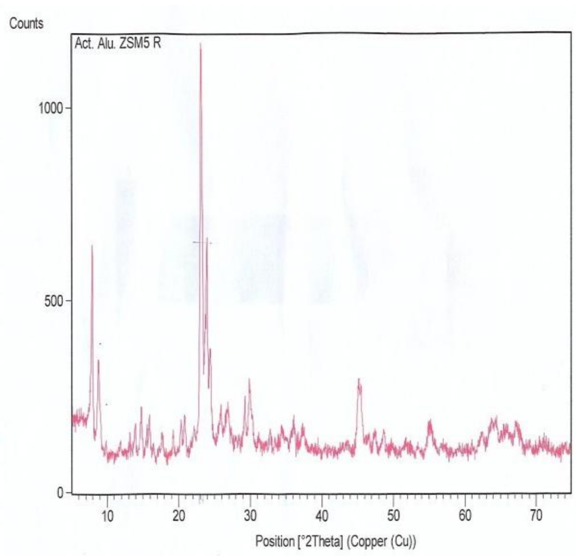
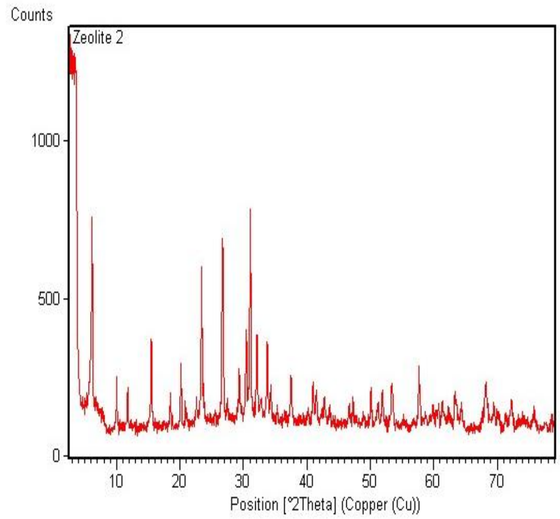


Figure 4.14: XRD pattern for activated alumina based ZSM-5 (repeated run 1)

Figure 4.15: XRD pattern for activated alumina based ZSM-5 (repeated run 2)



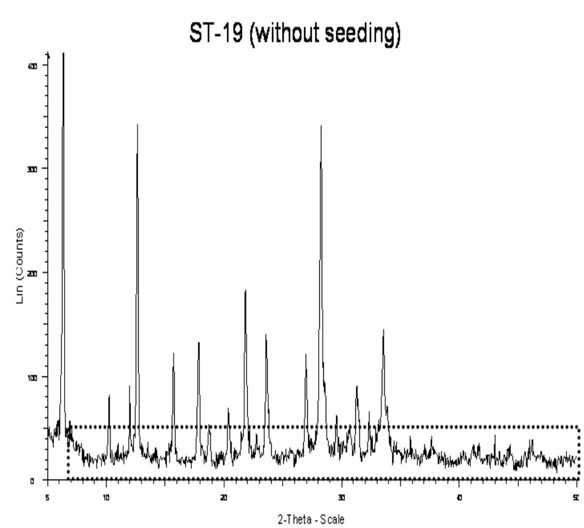


Figure 4.16: XRD pattern for RHA based Zeolite-Y without seeding

Figure 4.17: XRD pattern for referenced Zeolite-Y

(Source: Robson et al, 2001)

4.2 Discussion of Results

4.2.1: Discussion on compressive pressure absorption of kaolin clay, zeolite-Y, ZSM-5 and alumina clay

Referring to figure 4.1 and 4.2, it was observed that Zeolite-Y possess more uniform pore sizes, smaller pore sizes compare with ZSM-5, kaolin and alumina clay that were investigated under same condition. Also, it could be observed that zeolite-Y is denser and can offer highest resistance to external forces than other samples compared because; at maximum load (30N) it has least pressure deflection of 7mmH_2O. Though least effective in terms of flowability. Figure 4.1 shows clearly that, ZSM-5 can easily loss its micro-porous volume than Zeolite-Y because, ZSM-5 has larger pore sizes compare with that of Zeolite-Y and that is while it shows higher pressure displacement (10.50 mmH_2O) than Zeolite-Y at maximum load of 30N. In addition, synthesized ZSM-5 catalyst will deform faster than Zeolite-Y in respect of its higher porosity than Zeolite-Y catalyst.

It could be observed also that, Kaolin clay and Alumina clay emerge at maximum load of 30N where fracture occurred and this is an indication that, both of them possess same ability to serve as good support material for the synthesis of zeolite catalysts. Though in terms of porosity, kaolin clay is more porous when compared with alumina clay but, at a higher load or after a long period of use, alumina losses its strength faster than kaolin. It was observed from figure 4.2 that, at various load applied, Zeolite-Y is least compressed and offer the highest resistance to applied force when compared with other samples. At maximum load of 30N, alumina clay and kaolin clay were displaced in a similar pattern which is an indication that both of them possess similar pore sizes.

4.2.2: Discussion on stress / strain analysis on gambe clay, pindiga clay and kaolin clay

Referring to figure 4.3 and 4.4, it was noticed that at maximum load (32Kg), Zero strain value was recorded which was an indication that powder material does not yield before fracture. Continuous reduction in the amount of force applied gradually display strain effect on the clays analysed as shown in Figure 4.3.

It was shown clearly that, as the applied load increases, the compressive strain decreases, the particle size decreases, and loss of crystalline structure became faster and finally the material collapse at a maximum load of 32 Kg. Moreover, comparing the three clays analysed, it was shown that, Kaolin is more porous and offer least resistance to applied forces, while Gambe clay possess more strength and smaller pore size.

Figure 4.4 shows clearly that, for each of the sample analysed no strain was recorded for the first experiment carried out. Strain effect started manifesting at different forces applied for the various samples analysed. This had shown that Gambe clay has least pore sizes compared with Pindiga clay and Kaolin clay. Hence, kaolin is higher in terms of flowability than Pindiga and Gambe clay.

4.2.3: Discussion on stress / strain analysis on sample A and sample B

Referring to table 4.3, it was observed that, at the same strain value, sample A can withstand higher stress than sample B, though sample B is used in a fixed bed reactor while sample A is used in a fluidized bed reactor which is expected to possess higher resistance to external applied force than that of sample B. From Figure 4.5, the young's modulus was determined as follows:

From stress-strain relationship,

$\sigma = E.\varepsilon$

Where:

σ Is the stress applied

E Is the Young modulus

ε Is the strain

For sample A,

$E = \frac{\sigma}{\varepsilon} = \frac{22-15}{22-20} = \frac{7}{2}$ = 3.5N/mm^2

For sample B

$E = \frac{\sigma}{\varepsilon} = \frac{23-15}{12-10} = \frac{8}{2}$ = 4.0N/mm^2

The value gotten for the young's modulus of both samples represents part behaviour of the materials. In conclusion the higher the value of Young's modulus, E the higher the resistance of the material to deformation. Hence material B offers more resistance to deformation when compared with material A. Comparing the results obtained with that of the standard value of young's modulus for commercial zeolites (4.0N/mm^2 – 2.5N/mm^2), (Elsevier, 2002), the values obtained for the synthesized zeolites fall within, therefore synthesized zeolite catalysts can replace the commercial ones in use.

4.2.4: Discussion on compression test on zeolite-Y_n, Gambe clay and Pindiga clay

The results of the experiment obtained as presented in table 4.4 and plotted in Figure 4.6 shows similarity in trend pattern of the three samples analysed. It was observed that, there is no significant difference in stress absorption of the three samples analysed and any of the two clays will definitely serve as good supportive material in the synthesis of zeolite-Y catalyst for fluidised catalytic cracking application.

4.2.5: Discussion on Analytical Comparison between CPD commercial Zeolite-Y, CPD pure chemical ZSM-5 and CPD ZSM-5 ϒ alumina.

Referring to Table 4.5, it was cleared that compounded commercial zeolite-Y is of the highest density, less porous, but retains its crystalline structure and micro porous volume longer than the other zeolites examined under the same condition. This was due to its ability to withstand more pressure displacement compared to others at the same stress values. This was further represented in Figure 4.7.

4.2.6: Discussion on Comparison of compressive pressure against Load for Zeolite-Y_n, Zeolite-Y_s and Compounded commercial Zeolite-Y

Referring to table 4.6, it was noticed that at the same compressive load of (30N), zeolite-Y_s is denser, less porous compare to other Zeolites investigated under the same condition, but it fracture at a low pressure (8 mmH_2O).

While compounded commercial zeolite-Y and zeolite-Y_n are similar in terms of porosity and pressure absorption as presented in figure 4.8.

4.2.7: Discussion on Pressure absorption comparison between Gambe clay, Pindiga clay, Kaolin and Alumina clay

Referring to table 4.7, the results of the experiment carried out shows that Kaolin obtained from Kankara clay should be recommended preferable support material in the synthesis of zeolite catalysts compared with other clays. Though, other clays are good as well only that at a long run, they might lose their strength faster compared with Kaolin clay. This is because, according to the data obtained (9–12mmH_2O) Kaolin is least compressed. From figure 4.9, it was observed that at a high compressive pressure, Kaolin clay was least displaced which indicated its tendency to absorb stress longer than other clays.

4.2.8: Discussion on Pressure absorption capacity of Synthesized ZSM-5, pure chemical ZSM-5 and Compounded ZSM-5 γ alumina

Referring to table 4.8, the three ZSM-5 catalysts samples analysed shows same trend pattern (figure 4.10), but at a compressive pressure of 10mmH_2O and above, synthesized ZSM-5 might deform earlier because of its density. It is obvious that pure chemical ZSM-5 and ZSM-5 compounded Gamma alumina behave in a similar way. Though, the compounded ZSM-5 tends to possess more strength than the pure chemical ZSM-5 because, ZSM-5 compounded gathered additional strength from other support materials (Alumina and Gamma-Alumina) used in compounding it.

4.2.9: Discussion on Stress / Strain Analysis on Zeolite-Y_n

Referring to table 4.9, data obtained from stress / strain test carried out on Zeolite-Y_n gave an expected graph as represented in figure 4.11. This proved that to an extent, Zeolite-Y_n possess the ability to perform catalytically when used in fluidized catalytic cracking operation for a long period before losing its catalytic activities.

4.2.10: Discussion on X-ray diffractometer (XRD) analyses

X-ray diffractometer (XRD) analyses were carried out for Gamma alumina based ZSM-5 and compared with that of the reference pattern as shown in figure 4.12 and 4.13. While figure 4.14 and 4.15 shows the XRD pattern for activated alumina based ZSM-5 (repeated run 1) and that of the activated alumina based ZSM-5 (repeated run 2). In addition, figure 4.16 and 4.17 shows the XRD pattern for the RHA based zeolite-Y without seeding and the referenced Zeolite-Y. Comparing the XRD pattern for gamma alumina based ZSM-5 with that of the referenced pattern; there is similarity in their peak level. For instance at point 2-theta, where the highest peak occurred (at point 7.9, 10.1, 23.0,) it could be observed that, the peaks are almost at the same level. Hence, the result of the XRD pattern shows that, there is conformity of both samples which is an indication of possessing similar crystalline structure.

Comparing the XRD pattern for activated alumina based ZSM-5 repeated Run1 (figure 4.14) and repeated Run2 (figure 4.15) it could be observed that, their peaks level are similar and there is no difference at the point (2-theta) where the peaks occur. This is an indication that the activated alumina is well prepared.

Comparing the XRD pattern for RHA based zeolite-Y without seeding and that for reference zeolite-Y, there is similarity in their pattern. Though, little differences at the point where the highest peak occurred which might be attributed to the impurity of the non seeding RHA material.

CHAPTER FIVE

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

From the results of the analysis obtained on the work done, the following conclusions are drawn:

- Wearing rate of the synthesized zeolite catalysts depends on their pore sizes. Hence comparing the pore sizes of the synthesized zeolite catalysts with that of the referenced zeolite catalyst shows conformity of pore sizes which is an indication that, synthesized zeolite catalysts will offer similar resistance to wear when used as fluidized catalytic cracking catalyst like that of the referenced catalyst in used.
- The result of the powder compression test carried out on synthesized zeolite catalysts and support materials shows that, the larger the pore sizes the more porous the material and low pressure absorption.
- The Young's moduli of elasticity of synthesized catalysts were found to be in agreement with earlier investigation.
- Developed zeolite catalysts and support materials are resistant to crushing in packed-bed reactors because of their appreciable value of Young's modulus which indicate high level of crystalline structure which is a measure of high mechanical strength.

5.2 Recommendations for Further Research Work

- Design and construction of a mini-cyclone, where samples of synthesized zeolites can be subjected to flow in order to determine the particle size before and after the flow as a measure of its wear.
- Synthesis of large particle zeolites in order to enhance a proper mechanical test to be carried out.
- Studying the economic implications of the synthesize zeolite catalysts.
- Transmission electron microscope (TEM) should be used to determine the morphology of support materials.
- Thermo Gravimetric Analysis (TGA) of synthesized zeolite catalysts and support materials should be carrying out in order to determine their thermal characteristics.

5.3 Contribution to Knowledge

This work has established the following;

- The use of alumina clay as supportive material in the synthesis of zeolite catalyst has been established very good compared with gambe clay and pindiga clay.
- The possibility of using bourdon gauge equipment to investigate the pressure absorption ability of local available clays, synthesized zeolite-Y and ZSM-5 catalysts had been confirmed satisfactory.
- That the pore size of zeolite catalysts contributed to their mode of wear and flowability in fluidized catalytic cracking operations.

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