

**Ministry of Higher Education and  
Scientific Research  
University of Babylon  
Collage of Materials Engineering  
Department of Ceramic Engineering and Building Materials**



**Numerical study to additives sawdust rice,  
husk and effects on the refractory clay  
properties**

**By:**

**Wissam Mohsen Hamid**

**Supervised:**

**Prof. Dr. Mohammed Al- dujaili**

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بِسْمِ اللّٰهِ الرَّحْمٰنِ الرَّحِیْمِ

( وَقُلْ اَعْمَلُوا فِیْ سَبِیْلِ اللّٰهِ عَمَلِكُمْ وَرِسُوْلَهُ وَالْمُؤْمِنِیْنَ وَاسْتَرْدُوْا

اِلَىْ عَالَمِ الْغَیْبِ وَالشَّهَادَةِ فِیْ نَبْئِكُمْ بِمَا كُنْتُمْ تَعْمَلُوْنَ )

صَدَقَ اللّٰهُ الْعَلِیُّ الْعَظِیْمُ

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## الاهداء

الى من اوصى الله بهما برا واحسانا

(والدي العزيزين)

الى من اشد بهم ازري

(اخوتي الاعزاء)

الى من بجانبني دوما (الاصدقاء والاحبه)

الى من لا يبخلون بالعلم والكلمه الطيبه

## شكر وتقدير

" وَمَنْ يَشْكُرْ فَإِنَّمَا يَشْكُرُ لِنَفْسِهِ وَمَنْ كَفَرَ فَإِنَّ اللَّهَ غَنِيٌّ حَمِيدٌ (12) "

احمد الله حمدا وشكرا وتعظيما الذي هداني ويسر لي امري ومنحني العزم والصبر وحبب لي البحث العلمي المتواضع وما توفيقى الا بالله الذي علم بالقلم علم الانسان ما لم يعلم.

فمن هنا اغتتم هذه الفرصة كي أتقدم بجزيل الشكر والامتنان والعرفان الى كل من أفاض على حبه فأثمر ذلك وساهم في غرس بذور هذا العمل المتواضع حتى ضهر الى النور وأخص بالشكر والدي ووالدتي الذي هم نبع الحب والحنان والعطاء .

كما لا يسعني الا أن اتقدم بجزيل الشكر والامتنان والعرفان لمشرفي الاستاذ الدكتور محمد الدجيلي والذي اعطاني من وقته وفكره وتوجيهاته الكثير وزودني من الافكار والمعلومات والتوجيهات لاعداد هذا البحث ، كما اتقدم بالشكر والتقدير لكافه اساتذه وكوادر قسم السيراميك .

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# Chapter One: Introduction



# Chapter One: Introduction

## Introduction

Most metallurgical processes are heat generating systems. Such systems require materials that can withstand not only the high temperature generated but equally must be able to withstand both physical and chemical action of molten metal, slag and gases without deteriorating. Engineering materials that possess these attributes are referred to as refractory materials. The base material for refractory production is clay. Clays are naturally occurring sediments produced by chemical actions resulting from weathering of rocks [1]. An earthy fine-grained material, which develops plasticity when mixed with water, clay has silica ( $\text{SiO}_2$ ), alumina ( $\text{Al}_2\text{O}_3$ ) and water as primary constituents. Other constituents are iron, alkaline, and alkaline earth metals [2]. Nigeria has appreciable distribution of metal and process industries where high temperatures are generated and as such have considerable needs for Refractories. Present economic realities dictate the need for internal sourcing of raw materials to be used in the production of engineering materials. Refractories, being a class of materials largely used in metallurgical plants and cement industries, are presently largely sourced by importation (about 38,000 to 120,000 tons annually) with enormous financial implications to the country [3].

At present the generation of solid waste is enormously high. Hence large amount of environment impacts occur. Most of the solid waste has appreciable properties. Therefore waste materials can be used for various purposes as building materials. The major quantities of wastes generated from agricultural sources are rice husk, sugarcane bagasse, jute fiber, coconut husk, cotton stalk, etc. [4]. Sri Lankans have been engaged in agricultural tasks since ancient time. Sri Lankan farmers produce large amount of rice, as a result, large amount of rice husk is disposed as waste. Rice husk ash (RHA) is obtained from burning of Rice husk. The husk is a by-product of the rice-milling industry. By weight, 10 % of the rice grain is rice husk. On burning the rice husk about 20 % becomes RHA. [5]. In Sri Lanka, rice husk is often used as a fuel. For example, in Embilipitiya area the handmade brick producers used rice husk to fire clay bricks and the resulting RHA is open dumped. The use of rice husk on energy production is a good practice. However, the wasting of RHA, which has high percentage of silica, is not appreciable. In some areas of Sri Lanka, the Rice husk is open burning. This practice causes a lot of environmental issues. [6] concluded that the risk husk ash burnt in a controlled

manner with high temperature have high percentage of SiO<sub>2</sub>. According to the investigation of the [7], ideal temperature for producing RHA with high pozzalonic activities is control burning at 600 °C for 2-3 hrs. In addition, they found that in the brick kiln, where the rice husks were burnt, the temperature varies from 600°C to 850°C. Chemical composition of RHA that are available in different countries has been investigated in previous studies [5, 8,9] have investigated the properties of the Sri Lankan RHA, which was collected from brick kiln. [5, 8] have investigated the properties of RHA obtained from control burning process. Chemical composition of RHA reported in above mentioned studies are compared in Table 1. It can be observed that the RHA collected from brick burning process contains high amount of SiO<sub>2</sub>; similar to SiO<sub>2</sub> in RHA obtained from control burning process. The fired clay bricks are very popular among public due to its low cost and thermal performance. The demand for the graded clay bricks is comparatively high. As mention in the Sri Lankan standards for handmade fired clay bricks the average compressive strength of bricks should be more than 2.8 N/mm<sup>2</sup> and the water absorption should be less than 28%. At present, bricks produced in most of the areas, was not able to reach these standards: the compressive strength and water absorption properties have a considerable deviation. However, if the brick has low compressive strength properties it directly influences on propagation of cracks. The wall should be capable of withstand humid weather conditions; if the walls have less water resistance, the water will penetrate into the building. When constructing a wall, burnt brick should be immersed in water to absorb water, otherwise the water, which is in the mortar will be absorbed by the blocks and then the mortar will not be properly strengthen. Hence the water absorption should be balanced. Major properties (i.e., compressive strength and water absorption) of the bricks should be satisfied in order to use bricks for constructing buildings.

Chapter two:  
The theoretical part and literature  
review

# The theoretical part and literature review

## Introduction

In this chapter of the research, we will study the properties of clay and clay-based minerals, ion exchange capacity, cation exchange capacity, adsorption and permeability properties of clay. We will also study in this chapter how to classify clay by structure and layers, and we will also learn about the importance of clay and clay-based minerals. This chapter includes the effects of sawdust and rice husk additives on the properties of clay and the uses of both rice husk and sawdust.

## 2.1. Clay and clay-based minerals properties

### 2.1.1 Clay minerals as electron acceptors and/or donors in organic reactions

The electron-accepting and the electron-donating sites of clay can be explained by the fact the electron acceptor sites are aluminum at crystal edges and transition metals in the lower valency state. The catalysed polymerizations involve the conversion of the organic molecule to a reactive intermediate; hence, the clay mineral accepts an electron from the vinyl monomer and a radical cation is formed, where the organic compound gains an electron and forms a radical anion. The inhibition of polymerization processes involves the conversion of reactive organic intermediate, such as free radicals, which has been formed by heat or radical initiators, to non-reactive entities. An example of a thermal polymerization is illustrated by the loss of an electron from the free radical which gives a carbonium ion. In predicting the electron-accepting or electron-donating behaviour, colour reactions on clay minerals are useful for the reason that it similarly proceeds with mechanisms of polymerization reactions. For example, a blue reaction of benzidine: here there is one electron transfer from the organic molecule to the electron-accepting sites in the mineral (aluminum edges, transition metals in the higher valency state). In order to understand the many abilities of electron exchange of clay minerals, masking the crystal edge with

polyphosphate destroys the electron-accepting properties of the crystal edges. This method is used to assess the control of the reactivity of the mineral and distinguish the crystal edge from the transition metal sites as electron-acceptor sites in the clay minerals. [10]

#### 2.1.1.1. Ion exchange and cation exchange capacity

When erosion, transport, and deposition take place, clay minerals react to change in the environment. Ion exchange, reconstruction of degraded mineral, and formation of one type clay-based mineral from another or simpler substance appear as a result of those processes. Exchange reactions are dominated by physicochemical laws and depend upon the clay mineral, the nature, and ion population of the exchange sites and on the concentration and the composition of the solution in which the clay mineral is found. However, the increase in salinity when in a marine environment results in a decrease in the total exchange capacities of clay mineral when clay enters the sea. So the crystal chemistry of clay mineral is considered when exchange of cations takes place. Interlayer water cations and layer charge appear to be particularly important in the understanding of selective adsorption and fixation in the process of cation and ion exchange [11].

#### 2.1.1.2. Swelling behaviour

Clay mineral swelling is dependent on clay mineral type, the electrolyte concentration, and the nature of the cations in the solution. The swelling mechanism can be divided into mechanical and physicochemical processes. Through burial diagenesis, expandable layers are removed in the clay mineral structure such that inter and intralayer swelling of expandable clay mineral types can be expected to be at a minimum in older rocks than in younger rocks. Mechanical swelling occurs in response to elastic and time-dependent stress unloading, which can be brought by man in digging excavations or by nature in tectonic uplift and erosion because the clay is free to expand in the vertical direction but not in the horizontal direction. On the other side, physicochemical swelling is governed by intrinsic effective stress which commands the size disparity between larger, interaggregate voids and smaller intra-aggregate voids within clay mineral domains and between clay minerals themselves, all those forces which exist in a clay–electrolyte system subject to unloading after water had entered the system in response to mechanical cause [12].

### 2.1.1.3. Adsorptive and low permeability properties

As absorptive material, there are three ways clay minerals and clay-based minerals can exert non-covalent adsorptive power on various molecules from liquid to gaseous states. Firstly, physical adsorption: there is non-ionic adsorption onto the surfaces of finely divided material (large surface areas of clay minerals are comprised in small volumes), secondly ion exchange adsorption through electrostatic interaction and exchange, and finally, the inclusion of small molecules in pore or cavities, and partial or complete exclusion of larger molecules by those cavities through the zeolitic adsorption action. [13]. Permeability properties of clay minerals can be explained by the type and distribution of the clay minerals within the pore system. Generally, in rocks which are predominantly argillaceous, permeability is low. The mineralogy of different types of rocks semi-permeable in nature gives a noticeably different set of chemical parameters, whereas low to medium permeability can be assimilated to a closed system where rocks and fluid are effectively part of the same physicochemical unit.

## 2.2. Clay mineral classified by its structure and layer type

The interaction between clay minerals depends on their structure. This structure controls the behaviour of clay minerals' double layer which is the primary generator of repulsive pressure in the double layer model. The forces controlling the repulsive pressure are governed by physicochemical swelling in clay minerals since the attractive forces by comparison are small within the range of the external forces involved in the clay structure. Cations are attracted to external surfaces of clay minerals which are negatively charged and can also be drawn to internal surfaces of expandable minerals, so that clay mineral properties and structure can be changed. The sequence of replacement in clay minerals sites in nature of some principal cations is the same as their abundance

( $\text{Ca}^{2+} > \text{Mg}^{2+} > \text{K}^{+} > \text{Na}^{+}$ ).

### 2.2.1. Clay minerals classified by layer

Clay minerals can be described very simply by the stacking of two kinds of layers: 1:1 layers and 2:1 layers. They are layered by silicate in which each layer in the structure in reality consists of two sublayers. The sublayer consists of octahedral coordinates and structural water in the form of hydroxyl groups. Anionic clays also known as layered double hydroxides (LDHs) show a great example of the influence of clay layers on their cation–anion exchange capabilities. The intercalated structure and isomorphous replacement of trivalent cations for a fraction of divalent cations leads to positively charged host layers, where oxygen atoms coordinate each metal cation, forming an octahedron. Octahedra are composed of two-dimensional sheets formed by a wide range of interlayer anions, which then can constitute various kinds of anionic clay materials. The computation model of anionic clay minerals by [14] in their layered double hydroxides (LDHs) formula corroborated with previous work that the value of the stoichiometry coefficient ( $X$ ), the identities of the interlayer anion ( $An^-$ ), and the intra-layer cation when they vary enable to produce a wide range of specific tailor-made materials. To understand the electronic structure inside the LDHs is important for the stability formula of clay-based minerals. The electronic structure of LDH materials is often firstly focused on properties related with the whole bulk of the LDHs crystal in its entire extension in the periodicity coupled with *ab initio* plane-wave density functional theory or linear combination of atomic orbitals methods and secondly to predict the geometry of layer structure and the structural and chemical properties are investigated through semi-empirical molecular orbital method concluded in their work that the distortion angle of an octahedral coordinated hexahydrated cation plays a significant role in the formation of anionic clay layers. Also, the structural properties of the hexahydrated cations such as metal–oxygen bond length, O–M–O bond angle distortion, binding energy, and valence electronic configuration ligand field are in great agreement with the construction of anionic clay layers. Therefore, metal cations with similar ion size to  $Mg^{2+}$  are able to form the canonical hexahydrated structure with the distortion angle  $\theta$  smaller than  $1^\circ$  which are easily integrated into LDH layers on the basis of DFT theoretical calculation results. Adding ion size rule to this precept can give more insight into the application of layered double hydroxides [15]. (Figure 1).

### 2.2.2. Clay minerals classified by the structure

### 2.2.2.1. Adsorption

Weathering and precipitation at the mineral–water interface are of interest in mineral structure separation processes such as flotation, sedimentation, adsorption, scavenging of trace element, and transport of nuclear or other materials in groundwater [16]. Chemical reactivity of the mineral–water interface is influenced by properties which can be electrically charged at the surface of the mineral leading to the formation of an electrical double layer, less mobility of ions and water molecules. However, effects of the perturbed layer of water and the electrical double layer on chemical reactions at the interface play an important role. Understanding mechanism reactions of sorption is valuable and their kinetic interpretation explains the rate of attachment between an ion and the surface mineral. So the arrangement of group sites on mineral surface may influence the adsorption; however, under certain conditions, the formation of a monolayer of adsorbing ions may be less favourable than the formation of multi-layers or precipitated material; such a process plays a critical role in accelerating the rate of redox reactions, polymerization, hydrolysis, and other transformations taking place in the surface structure of clay minerals.

### 2.2.2.2. Layer charge

Either electrically neutral or negatively charged structure of clay mineral may occur as a result of the tetrahedral and octahedral sheets' junction in clay. The electrical neutral charge exists if the octahedral sheet contains trivalent cations in two octahedral sites, with a vacancy in the third octahedron, or with the divalent cation occupying all the octahedral site, secondly in the lower charge cation where all the octahedral sites  $Al^{3+}$  and  $Mg^{2+}$  are substituted, and thirdly when there is presence of vacancies. This aspect of the layer charge is the most important feature of 2:1 clay minerals because it influences occupancy of the interlayer space by exchangeable cations (Figure 2).

### 2.2.2.3. Polytypism



This feature of clay minerals structure is mentioned in several diverse structural modifications in which layers of identical structure and composition are stacked in different ways. The normal periodicity to layers varies with stacking sequence between polytypes according to the number of layers involved.

#### 2.2.2.4. Mixed layers structures

Mixed layer structures or inter-stratified layers can be built by two or more than two different components. This clay minerals can have ordered or regular-mixed layer structures if different layers alternate along the direction in a periodic pattern and disordered or irregular mixed layers structures if the stacking along the direction of the type of layer is random [17]. The interfaces between clay particles are capable of adsorbing water or organic molecules so that the adjacent layers are perceived as interstratified with non-expanding silicates layers, thus having important geochemical consequences. In addition, illitization is affected by the abundance of water in the system. This conversion phenomenon can actually involve inter-stratified clays like illite-smectite [18]. This conversion may involve smectite dissolution and thin Illite precipitation or growth. When major amounts of smectite are not no more present, either elementary or possibly thicker, Illite particles become dominant and will yield interstratified illite-smectite and as the thickness of the Illite particles increases, the diagenesis increases. This interstratification also has implications to clay chemistry and their stability in the environment to some extent [19]. Indeed, the versatile structures of clay minerals are key factors responsible for clay minerals' behaviours and use for a wide range of applications both as colloid stabilizers, catalysts, catalytic and chemicals supports, coagulants, sorbents, coating, and perhaps in many more areas.

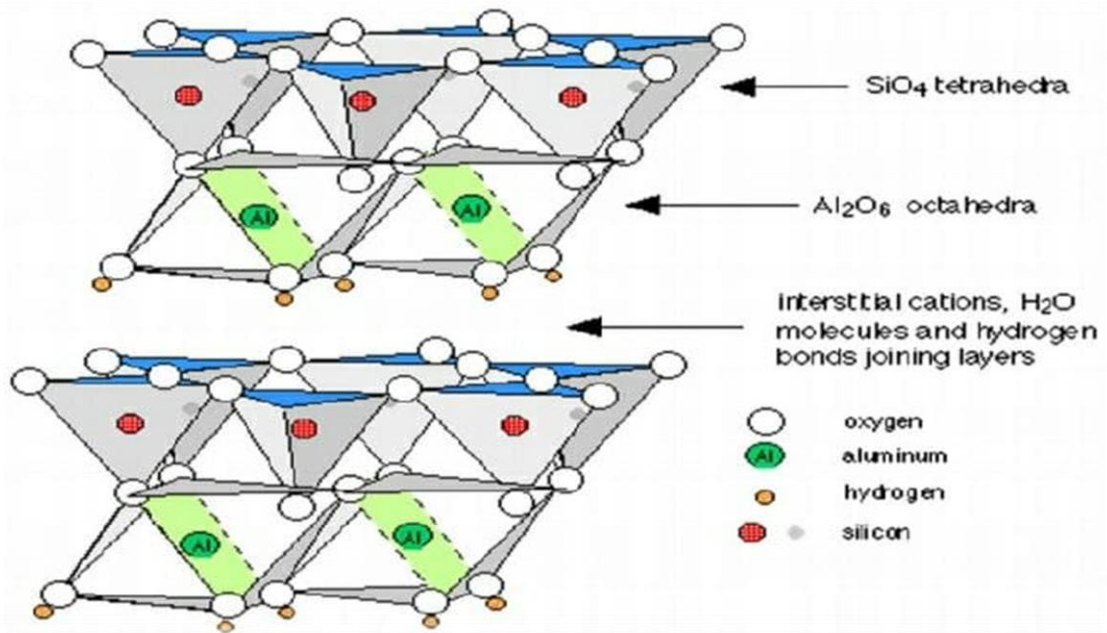
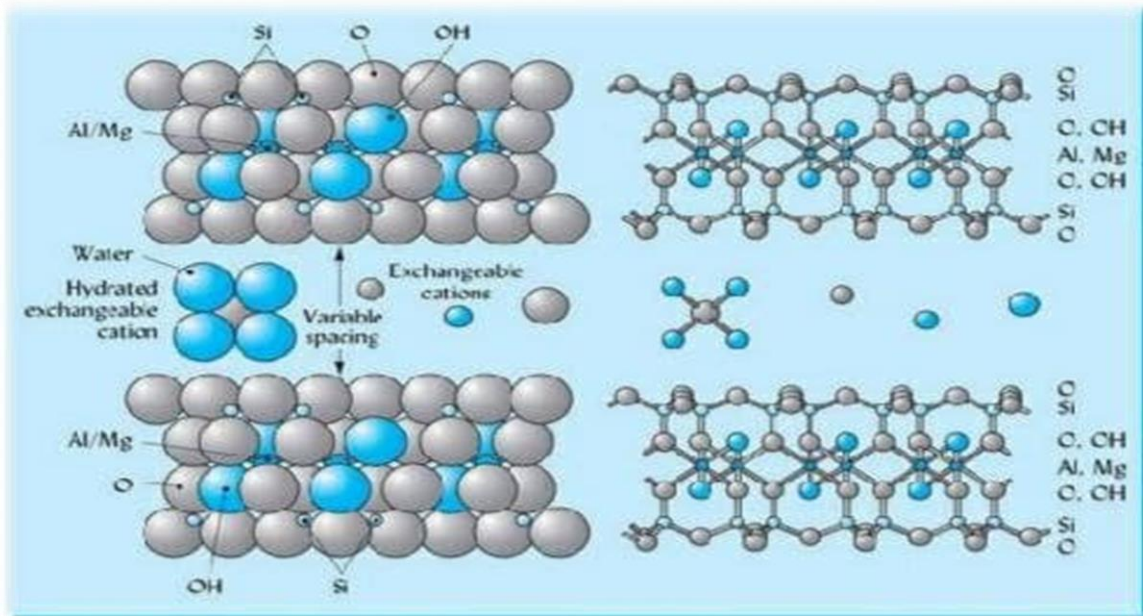


Figure (1). Structure of clay showing two layers of the stacked sheets of kaolinite.



Figure(2). Exchangeable cation in clay minerals

### 2.3. Importance of clays and clay-based minerals in the environmental aspect of their characteristics.

Clay mineral particles' aggregation and dispersion take place under changing conditions in natural systems. Thus, the structure of clay particles is inherently influenced by the mineral matrix and the different fractions bound to it since properties of clays and clay-based minerals play an important role in the clay mineral assemblage, nature, structure, and ionic strength related to the interrelation between the surface of clay and the minerals' surface bound for example to organic matter compounds determines the organo-clay interaction and influence of the sorption capacity at the solid aqueous interface [20].

Many mechanisms of biological protection in environment containing clay minerals and clay-based mineral particles operating from the smallest to the largest scale depend on the chemical properties and the dimensional arrangement of layers in the mineral. As a result, diverse mechanisms of protection can be attributed to clay minerals in some matrix-like soil for example. Due to firstly the physical nature of the mineral fraction, especially the presence of surfaces capable of adsorbing organic materials, and secondly the architecture of layers in the clay surface, there are multiple applications of clay minerals because of their versatile arrangements [21]. Surface reactive phases of clay minerals and clay-based minerals also play an important role in the regulation of contaminant fate and transport in surface and subsurface of the environment because these surfaces are the primary controllers of sorption processes in soils, thus acting as important regulators of contaminant transport. The modification of surface charge of clay minerals by organic constituents is responsible for dispersion/flocculation mechanisms of clay assemblage in the surface as well as the transport of mineral colloidal phases through soil.

This surface charge is manifested by a significant retention of anions which assure that complex chemical properties have a number of important implications for solute and contaminant transport, [22]. Mutations and transformations of clays and clay based minerals respond to their chemical and thermal environments, their properties and species change at each step from the origin, weathering, through their transportation, sedimentation, burial diagenesis, and metamorphism. Another transformation through the bounding mechanism

between the organic cation and the charged clay layers is essentially electrostatic. Through ion exchange

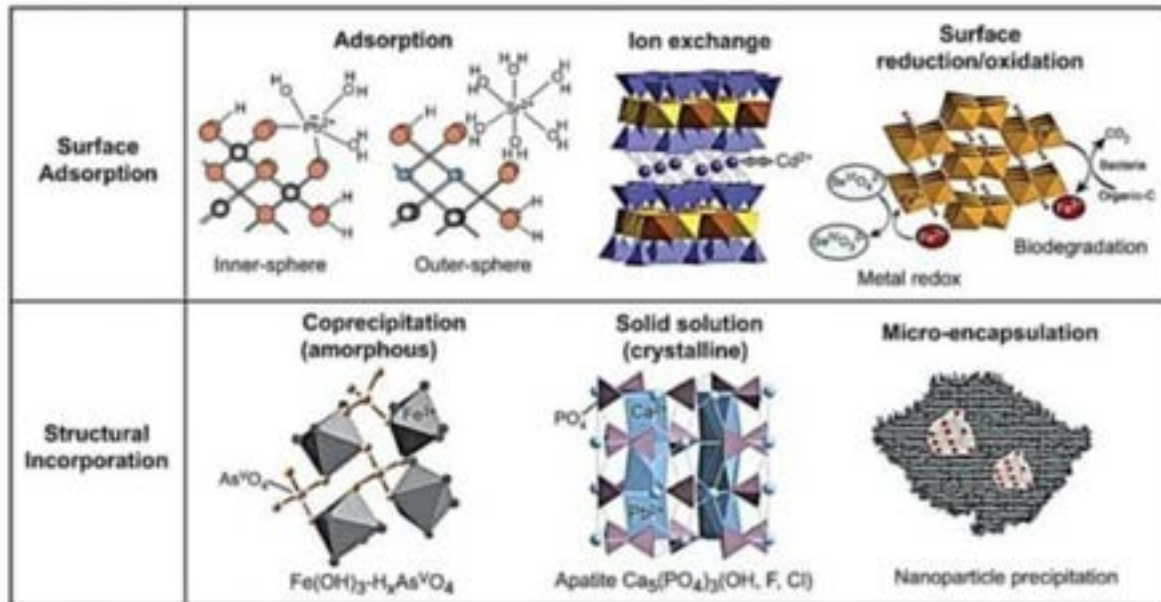


Figure 3. Examples of structural transformation of clay to nanoparticle clays

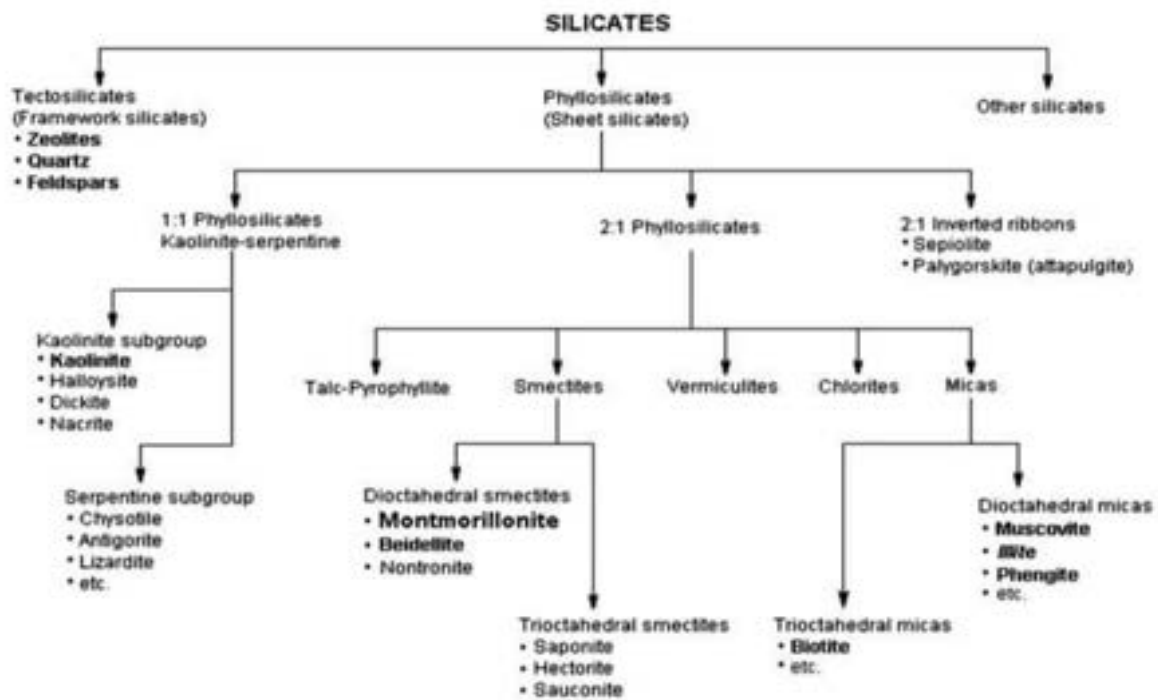


Figure 4. Classification of silicates by Bailey [23]

Reactions, the inorganic exchangeable cation of layered silicates belonging to clay minerals can be replaced by organic cations Figure (3). The ions used for this purpose result in the formation of organophilic clay minerals which may greatly adsorb a wide variety of organic compounds. These materials are known as organoclays. Their properties and applications strongly depend on the nano and micro-structural arrangement of hybrid materials and from the mechanisms involved in the clay–organic interactions. Hence, diverse applications can be derived from the characteristics of organoclay minerals. For example in the preparation of polymer-clay nano composites, organophilic polymers and clays acting as nanofillers are also used to develop inorganic heterostructures and inorganic polymer clay nano-composites that generates porous silica acting as pillared materials. Another use of organoclay is the preparation of sepiolite or palygorskite microfibrillar clays with quaternary ammonium salts producing organophilic solids which can be used as paint thickeners and for other industrial uses and similar applications.

These transformations make organo-clay prepared formulations suitable for environmental applications since they are generally based on the (i) substrate: due to its unique mineralogical structure, it offers several binding sites to different types of molecules, (ii) the modifier: the organic molecule bound to the clay mineral allows the modification of the substrate surface to increase the affinity of the hybrid nanomaterial obtained, and (iii) the molecule of interest: the organoclay is prepared either to remove contaminants by adsorption, to avoid the leaching or decomposition, or to enhance the activity of an organic molecule. So organophilic clay minerals as mentioned above like clay modifiers have the capability to efficiently sorb organic compounds and remove them from water or effluents, reaching high capacity of adsorption at high pollutant concentrations allowing the use or reuse of water that before treatment would be considered unusable ,[ 24] (Figure 4)

## 2.4 Effects of Sawdust and Rice Husk Additives on Properties of Local Refractory Clay.

### 2.4.1. Material Preparation

The clay sample was dug from the deposit site using a hand-held hoe. The sample was sun-dried to reduce moisture content and enhance grinding. 500g of the sun-dried sample was grinded to powder using a jaw crusher. 400g of the sample was taken for physical property tests while the remainder was further ground to finer particles and then used for the chemical composition tests. The sample was weighed and further dried in an oven at 110 for one hour to ensure moisture evaporation.

### 2.4.2. Determination of Properties

#### 2.4.2.1. Chemical Composition

A 01g mass of the sample was weighed into a Teflon crucible. It was moistened with aquilegia (mixture of hydrochloric and nitric acid in a ratio of 3:1 by volume). About 15ml of hydrofluoric acid was added and the mixture was covered, heated and digested in an oven set at 100oC until the solution became clear. The ratio of the clay sample to that of the additives is 9:1. The additives were burnt to ashes before mixing with the clay. After undergoing the heating process, the sample was allowed to cool and then transferred to a 250ml plastic volumetric flask and mixed with distilled water. This is the stock solution which was used in determining the Silicon, Aluminum and Iron oxides[25].

#### 2.4.2.2. Physical properties

##### A. Bulk Density

Bulk density is the weight per unit volume of the refractory material including the volume of open pore space. It could be determined using the direct volume measurement method [26]. A test specimen was cut from the core of the material

sample using a cut-off wheel. The dry and saturated weights of the sample were determined. The bulk density was then calculated using the relation 1:

$$\text{Bulk Density, } \rho = \frac{W_A}{W_C - W_B} \rho_W \dots\dots\dots (1)$$

Where  $W_A$ – weight of dry sample;  $W_B$ -weight of dry sample suspended in cold water;  $W_C$  - weight of soaked sample suspended in air; and  $\rho_W$  - density of water.

### B. Thermal shock resistance

The sample was placed in a muffle furnace preset at 1200°C minutes 10 minutes. It was then cooled outside the furnace for another 10 minutes and observed for cracks. The heating and cooling cycles are repeated until cracks are observed on the specimen. The number of these cycles undergone before cracking was recorded and this constitutes the thermal shock resistance of the material.

### C. Porosity

Porosity is the percentage relationship between the volume of pore spaces and the total volume of the refractory. For determination of porosity, a prepared clay sample was air-dried for 24 hours. The sample was then oven-dried for another 24 hours at 110°C. It was then fired at 1100°C, cooled and transferred into a desiccator and weighed to the nearest 0.01g (dried weight). The specimen was then transferred into a 250ml beaker in an empty vacuum desiccator. Water was then introduced into the beaker until the test sample was completely immersed. The specimen was allowed to soak in the boiled water for 30 minutes the set up being agitated from time to time to assist the release of trapped bubbles after which the specimen was transferred into an empty vacuum desiccator to cool. The soaked weight was then taken and recorded. The specimen was then weighed suspended in water using a beaker on a balance to obtain the suspended weight. The apparent porosity was calculated using:

$$\text{Apparent Porosity} = \frac{W - D}{W - S} \times 100\% \dots\dots\dots (2)$$

Where  $W$ - soaked weight;  $D$ - dried weight and  $S$ - suspended weight.

### D. Fired Shrinkage

The test piece was made into a standard slab and a line marked along the length of the slab. The distance between the two ends of the slab was measured using a

vernier caliper. The sample was air-dried for 24 hours and oven-dried at 110°C for another 24 hours. It was then fired at 1100°C for 6 hours. The test piece was cooled to room temperature and another set of measurement taken. The fired shrinkage was calculated using the relation:

$$\text{Fired Shrinkage} = \frac{DL - FL}{DL} \dots\dots\dots(3)$$

Where *DL* – initial fired length *FL* – final fired length

### E. Specific gravity

The specific gravity test is useful for determination of particle size distribution of any powdered material. It is defined as the ratio of the mass of the material to the mass of a quantity of water at 4°C which has a volume equal to the solid volume at the temperature of measurement to determine the specific gravity, a flask was filled to a given level with water; a weighed amount of suitably ground and dried sample material was poured into it and the increase in volume which occurred was noted. The specific gravity of the sample was then calculated using the relation below [27]:

*Specific gravity* =  $\frac{b-a}{d-a-c} \cdot g \dots\dots\dots(4)$  where: a – weight of bottle; b – weight of bottle plus sample; c – weight of bottle plus sample plus distilled water; d – weight of bottle plus distilled water and g – density of water.

### F. Refractoriness

Refractoriness is the measure of the fusibility of a material and indicates the temperature at which the material softens. The parametric cone equivalence (PCE) method was used to determine the refractoriness of Mangada clay. The clay sample was dried and ground to pass through a 30 mesh British standard (B.S 1610R1942) test sieve and 50g was further ground to pass a 72 mesh sieve. Sieving was frequent to avoid excess of very fine powder. The sample was then thoroughly mixed, made into a plastic mass with water and an organic binder of 0.5% maximum ash content was added. The test piece was then formed in a suitable mould and calcinated at 1000°C. The mould was shaped into a pyramid with a triangular base (of sides 1.27cm with a tolerance of not more than 0.16cm on each side) and having one edge of the pyramid perpendicular to its base and 3.81cm long. The test piece was mounted at the centre of a refractory plaque with



its edge vertical to the base and fixed with cement containing calcined alumina. British standard pyrometric cones were cemented to the plaque but oriented so that they would bend away from the test piece with the numbers facing inwards and the edges opposite the numbers vertical. The refractory plaque with test piece and the surrounding pyrometric cone was placed in the furnace and the temperature raised at the rate of 10 – 15°C per minute to an estimated temperature of 200°C below the equating temperature observing the temperature rise rate with an optical pyrometer. The test was continued until the tip of test cone had bent over level with the base. The plaque carrying the specimen was then removed and allowed to cool and test piece and pyrometric cones examined when cold. The refractoriness is taken as the number of pyrometric cone that has bent over to a large extent similar to the test cone. The temperature is then read off from the equivalent of the cone number.

## 2.5 Uses of Sawdust

Sawdust is a by-product of wood based actions. Since sawdust comes from wood and it is assumed to possess some of the properties of wood [28]. People have therefore tried to use sawdust in different ways and for various reasons, depending on the property of sawdust they want to utilize. This is in a bid to change it from waste to useful material.[29] stated other uses of sawdust to include fuel for cooking, particle board production, ceiling boards, wall tiles, floor tiles and partitioning panels. Chemical uses include ethyl alcohol, yeast, oxalic acid rinse, just to mention a few. [30], have also stated that the volume of wood waste generated in Nigeria increases yearly, and according to [31] this large volume of sawdust generated however, creates disposal problems which are of great concern to all wood industries and governments. As activities of sawmills continue to increase with the increase in the number of saw mill industries and other wood based industries, quantities of sawdust will continue to increase. [32], in a further research stated that as Nigeria had 1325 sawmills located in different parts of the country. This gave a total installed capacity of sawmills industry in Nigeria as 11.7 million m<sup>3</sup> of wood, but that the actual production was about 3.8 million m<sup>3</sup>. This they say represent the national capacity utilization of about 24.5%. This statistics shows an increase in the number of sawmills in Nigeria over what was earlier reported. It is noticed that sawdust production continues to increase at all times. This means that as long as there is wood in our forests and wood based industries and sawmilling continues to increase there will be increase

in sawdust production, hence its availability. Many studies have also been carried out on the use of sawdust in building materials.[33] in their study on “limestone dust and wood sawdust as brick materials” undertook a parametric experimental study to investigate the potential use of wood sawdust waste (WSW) and Limestone Powder Waste (LPW) combination to produce a low-cost and light weight composite as a building material. Some of the physical and mechanical properties of concrete mixes having high levels of WSW and LPW, like compressive strength, flexural strength, unit weight, ultrasonic pulse velocity (UPV) and water absorption values were investigated. It was then found that the values of the physical and mechanical properties investigated satisfy the relevant international standards. This parametric experimental study revealed that the effect of high level replacement of WSW with LPW does not exhibit a sudden brittle fracture even beyond the failure loads. It shows high energy absorption capacity, dramatically reduces the unit weight and introduces a smoother surface compared to the current concrete bricks in the market. This experiment reveals that the mixture of WSW and LPW has potential to be used for walls, wooden boards' substitute, economically alternative to the concrete blocks, ceiling panels and sound barrier panels.

One of the aims of this study was to utilize the abundant waste materials of sawdust and limestone powder which cause serious environmental problems and health hazards. [34], established that 18-20% of log volume in Uganda is sawdust. They also noted that sawdust is one of the major-under-utilized by products from sawmilling operations in Uganda. They therefore suggest that finding an appropriate use of sawdust would help to offset production costs and increase the profitability of saw milling operations in Uganda's plantation forests. This suggestion will help Nigerian saw millers too, if economic use of sawdust is found. This project therefore seeks to explore ways of finding economic use of sawdust in Nigeria and the world, in building materials. In a study carried out by [35], on the production of composite bricks from sawdust using Portland cement as binder, whose specific objectives were: (i) To make composite bricks using varying sawdust to cement ratios and (ii) Determine the density, mass and compressive strength of the composite, 48 bricks of cement to sawdust mix ratios of 3:2 and 2:1 by volume were produced. The bricks sizes were 50mm x 50mm x 50mm (small) and 100mm x 100mm x 100mm (big). The sawdust particles were between 2.5 – 3.5mm in diameter. The sawdust was soaked in water for 24 hours to reduce the amount of water soluble sugars and tannins. This was then dried to

5% moisture content. The structural integrity of the brick was assessed by dropping the bricks from a height of 1m. Mass was measured using a weighing balance, while density was calculated from mass and volume of the bricks. A universal testing machine was used to test for compressive strength values which were found to be 1.61 Nmm<sup>2</sup> and 1.986 Nmm<sup>2</sup> for composite of 3:2 and 2:1 respectively. The compressive strength value of 100mm x 100mm x 100mm composite with sawdust to cement ratios of 3:2 and 2:1 was found to be 1.778 Nmm<sup>2</sup> and 2.21Nmm<sup>2</sup> respectively. The statistical tool used was analysis of variance (ANOVA) which indicated significant difference ( $P < 0.05$ ) in strength values of the two composites. [36] Therefore concluded that use of wood/cement composite bricks can reduce overall weight of the construction, since their densities and weight are generally low. They however found the composite bricks unfit for paving and medium heavy load construction. [37] al; however recommended that due to the light weight of the composites, they can be imparted with decorative mosaics and can be used for interior wall paneling and decoration, where minimum wetting is experienced. These bricks are unfit to be used externally due to dimensional instability during wetting. The study contributed to the improvement of the environment in Uganda through reduction of waste.[38] however, has laid a good base for this work where similar things are don.

## 2.6. Uses of Rice Husk

The use of rice husk has however been tried by some persons. The [39] used rice husk to produce, particle boards and found out that it possessed excellent mechanical properties like, internal bond strength, elasticity, dimensional stability, screw and nail holding capacity, abrasion resistance, and surface hardness. With these qualities discovered by [40], it means then that rice husk has promise in building materials. [41], also asserts that rice husk ash gives a good castable insulating and semi-refractory blocks when mixed with cement and water (2:1 – 3:1), while [42] stipulates that rice husk ash (RHA) significantly improves the durability of concrete and substantially replaces silica fumes as additive at lower cost. Rice husk is a class ‘A’ insulating material. The silicon it produces when burnt provides excellent thermal insulation. [43], stated that rice husk had been used directly, without burning to ashes, in many industrial applications. It has been used directly as fuel in power plants. Rice husk has been used also as source of raw materials for synthesis and development of new phases

and compounds. Rice husk has been used in industrial sectors as fuel in power plant, in formation of activated Carbon, as a source of Silica and Silicon compounds, to get porous SiO<sub>2</sub> /C composite, for insulating fire brick.[44], in their study on “effects of the combined use of rice husk and fly ash on concrete properties” investigated the characteristics of concrete in the case where rice husk ash is mixed in concrete of low water binder ratio, using a large volume of fly ash. In the investigation, the compressive strength, freezing and thawing, as well as pore size distribution, were specifically studied. It was therefore discovered through tests that, the compressive strength of concrete with rice husk, tested at 3 days and 365 days became 1.1-1.3 times more than concrete without rice husk ash.

It was also discovered that concrete with rice husk ash offered similar resistance to freezing and thawing when compared with concrete without rice husk ash. Furthermore, the study measured pore size distribution and found out that total volume in concrete with rice husk ash decreased thereby leading to an increase of strength. The study by [45] centred on the compressive strength, freezing and thawing as well as pore size distribution. This study however concentrated on determination of compressive strength of the bricks. This study also uses rice husk in un-burnt form while they used rice husk ash. [46] Undertook a research on the influence of particle size and firing temperature in burnt properties of rice husk/clay mix. Their main objective was to investigate the scope of the use of rice husk in brick making. The effect of the rice husk/clay mix ratios for the different particle sizes of the rice husks and temperature of the formed bricks were also studied in the context of their effects on the compressive strength and water absorption.[47] molded composite bricks containing various additions of rice husks grouped into four particle size ranges. These bricks were molded under 5 MN/m<sup>2</sup> compaction pressure, dried and fired at 500oC, 600oC, 700oC, 800oC respectively. During the measurement of compressive strength, water absorption and density, it was discovered that small additions ( $\leq 1\%$ ) of rice husks led to improved compressive strength and a decrease in the amount of water absorbed. The addition of higher percentages of rice husk reduced the compressive strength and density of the bricks and increased the amount of water absorption. This work further asserted that particle size ranges of 425 – 600 $\mu$ m gave best improvement of the brick properties. The disparity of [48] work is their use of clay mix with rice husk and the firing of the bricks while this study uses rice husk and sawdust as aggregate and does not subject them to firing. In another study, [49], used

rice husk ash with cement to produce low cost blocks. They first tested the compressive strengths of some commercial sandcrete blocks in Minna, Niger State, of Nigeria, and found them to be deficient in strength. The possibility of addition of rice husk ash was therefore explored experimentally to augment the high cost of cement which was the reason for its insufficient use which led to the loss of strength of the blocks. Hollow blocks of size 150mm x 450 containing various percentages of rice husk ash replacements (0, 10, 20, 30, 40, and 50%) were cast, cured and crushed at 1, 3, 7, 14, 21, and 28 days. The mixture ratio used was 1.8 (one part of binder to eight parts of sand) at different levels of OPC and rice husk ash (RHA). For each replacement level about 18 blocks samples were cast. Three sample blocks were crushed each at 1, 3, 7, 14, 21, and 28 days at different replacement levels using compressive testing machine. In this work of [50].

The tests conducted included chemical analysis of rice husk ash, particle size of sand, specific gravity test on RHA and sand, bulk density test on rice husk and sand, silt content test on sand, constituency tests, setting time test, free moisture content test and slump test as well as the determination of compressive strength and density. Mix design was carried out by absolute volume method to select most suitable materials of cement, RHA, sand and water, to produce blocks with desired properties. The maximum value of the compressive strength of the blocks obtained in Minna was 0.97 N/mm<sup>2</sup>, which does not comply with the minimum standard of 3.5 N/mm<sup>2</sup>. Bulk density of RHA and sand were 530kg/ m<sup>3</sup> for compacted and 460 kg/m<sup>3</sup> for un-compacted RHA, while compacted densities of sand was 1600 kg/ m<sup>3</sup> and un-compacted density of sand was 1500 kg/ m<sup>3</sup> . The difference in value is attributed due to the sample disturbance. It was found out that at 28 days hydration period, blocks made with 10% and 20% RHA replacement met the minimum stipulated compressive strength of 3.5 N/mm<sup>2</sup> as they were 4.1N/mm<sup>2</sup> and 3.65 N/mm<sup>2</sup> respectively. Although other replacement levels fell below standard of 3.5 N/mm<sup>2</sup>, they were higher than local blocks produced in Minna, which had 0.97 N/mm<sup>2</sup>, except for 50% replacement which had compressive strength of 0.59 N/mm<sup>2</sup> at 28 days. [51] Concluded that blocks using RHA as partial replacement to OPC will be cheaper to produce and thus improve the quality of blocks in Minna. The use of sand and rice husk ash in this work deviates from the intent of this work which eliminates sand to reduce weight and uses sawdust and rice husk with OPC. Basic test on compressive strength are carried out. Many other tests as mentioned above are not used for the intended

project. Furthermore, rice husk and sawdust are used in their natural state, not burned to ashes. Sawdust, another agro-waste material, is a by-product of wood, produced from sawing of wood. Sawdust is those small pieces of wood that are left when one has been cutting wood. [52,] has observed that large quantities of sawdust can be found in Nigeria around sawmills and wood based industries. Sawdust is largely seen as waste and so is not utilized. According to [53], the non-utilization of the sawdust creates disposal problems, which are burdensome. [54], therefore, observed that in order to dispose of the large sawdust hills around sawmills, saw millers resort to burning. The burning thus produces smoke and offensive gases like carbon dioxide and carbon monoxide, which are hazardous to human health and contribute to ozone layer depletion as well as environmental pollution.

# Chapter Three: Experimental work

## Chapter Three: Experimental work

### Introduction

In this chapter of the research, we will study the effect of adding rice husk and sawdust on the physical and chemical properties of clay, as well as preparing samples and devices used, as well as testing properties such as density, porosity, thermal shock resistance and specific density.

### 3.1 Experimental procedure

In this chapter of the research, we will study the effect of adding sawdust and rice husk in different proportions on the physical and chemical properties of refractory clay properties.

### 3.2 Material preparation

The clay sample was dug from the deposit site using a hand-held hoe. The sample was sun-dried to reduce moisture content and enhance grinding. 500g of the sun-dried sample was grinded to powder using a milling device.



Figure 1. Milling device

400g of the sample was taken for physical property tests while the remainder was further ground to finer particles





Figure 2. Ball mill

Then used for the chemical composition tests. The sample was weighed and further dried in an oven at  $110^{\circ}\text{C}$  for one hour to ensure moisture evaporation.



Figure 3 .firing furnace

### 3.3 Test

### 3.3.1 Bulk Density

Bulk density is the weight per unit volume of the refractory material including the volume of open pore space. It could be determined using the direct volume measurement method.

A test specimen was cut from the core of the material sample using a cut-off wheel. The dry and saturated weights of the sample were determined. The bulk density was then calculated using the relation ..... (1)

Where  $WA$ – weight of dry sample;  $WB$ -weight of dry sample suspended in cold water;  $WC$ - weight of soaked sample suspended in air; and  $\rho W$ - density of water.

### 3.3.2 Thermal shock resistance

The sample was placed in a muffle furnace preset at 1200°C minutes 10 minutes. It was then cooled outside the furnace for another 10 minutes and observed for cracks. The heating and cooling cycles are repeated until cracks are observed on the specimen. The number of these cycles undergone before cracking was recorded and this constitutes the thermal shock resistance of the material.

### 3.3.3 Porosity

Porosity is the percentage relationship between the volume of pore spaces and the total volume of the refractory. For determination of porosity, a prepared clay sample was air-dried for 24 hours. The sample was then oven-dried for another 24 hours at 110°C . It was then fired at 1100°C, cooled and transferred into a desiccator and weighed to the nearest 0.01g (dried weight). The specimen was then transferred into a 250ml beaker in an empty vacuum desiccator. Water was then introduced into the beaker until the test sample was completely immersed. The specimen was allowed to soak in the boiled water for 30 minutes the set up being agitated from time to time to assist the release of trapped bubbles after which the specimen was transferred into an empty vacuum desiccator to cool. The soaked weight was then taken and recorded.

The specimen was then weighed suspended in water using a beaker on a balance to obtain the suspended weight. The apparent porosity was calculated using:

$$\text{Apparent porosity} = \frac{w-d}{w-s} \times 100\% \dots\dots\dots(2)$$

Where W-- dried weight and S- suspended weight.

### 3.3.4 Fired Shrinkage

The test piece was made into a standard slab and a line marked along the length of the slab. The distance between the two ends of the slab was measured using a vernier caliper. The sample was air-dried for 24 hours and oven-dried at 110°C for another 24 hours. It was then fired at 1100°C for 6 hours. The test piece was cooled to room temperature and another set of measurement taken. The fired shrinkage was calculated using the relation:

$$\text{Fired Shrinkage} = \frac{DL - FL}{DL} \dots\dots\dots(3)$$

Where *DL* – initial fired length *FL* – final fired length

### 3.3.5 Specific gravity

The specific gravity test is useful for determination of particle size distribution of any powdered material. It is defined as the ratio of the mass of the material to the mass of a quantity of water at 4°C which has a volume equal to the solid volume at the temperature of measurement. To determine the specific gravity, a flask was filled to a given level with water; a weighed amount of suitably ground and dried sample material was poured into it and the increase in volume which occurred was noted. The specific gravity of the sample was then calculated using the relation below:

$$\text{Specific gravity} = \frac{b - a}{(d - a) - (c - b)} * g \dots\dots\dots(4)$$

where: a – weight of bottle; b – weight of bottle plus sample;

c– weight of bottle plus sample plus distilled water; d – weight of bottle plus distilled water and g – density of water

# Chapter four :

## Results and discussion

### Chapter four: Results and discussion

#### Introduction

In this chapter of the research, we will study the results of the tests and study the effect of rice husk and sawdust additives on the physical properties of clay, i.e. through the method of making Takoji (comparing the results with the tables below) we will get the effect of those additives.

Table 1: Composition of red clay and additives mixture

Starting material batch no.	Red clay %	Additives	
		sawdust	Rice husk
1,2	98	2	2
3,4	96	4	4
5,6	94	6	6
7,8	92	8	8
9,10	90	10	10
11,12	80	20	20
13,14	70	30	30

Table 2a: Result of physical analysis for a water filter disc with sawdust by slip casting.

Addition Ratio	2%	4%	6%	8%	10%	20%	30%
Physical Properties							
Porosity %	47.5	51.6	55	55	58.4	68.4	84.6
Density g/cm <sup>3</sup>	1.38	1.32	1.28	1.13	1.09	0.94	0.69
Permeability Cm/min	$3 \cdot 10^{-7}$	$4 \cdot 10^{-7}$	$1 \cdot 10^{-6}$	$2 \cdot 10^{-6}$	$2 \cdot 10^{-6}$	$1 \cdot 10^{-5}$	$6 \cdot 10^{-5}$

Table 2a: Result of physical analysis for a water filter disc with sawdust by slip casting.

Addition ratio	2%	4%	6%	8%	10%	20%	30%
Physical properties							
Porosity %	48.5	44.8	47.1	50	54.6	65	66.6
Density g/cm <sup>3</sup>	1.65	1.63	1.60	1.43	1.26	1.13	1.09
Permeability Cm/min	2*10 <sup>-7</sup>	2*10 <sup>-7</sup>	3*10 <sup>-7</sup>	4*10 <sup>-7</sup>	2*10 <sup>-6</sup>	3*10 <sup>-6</sup>	5*10 <sup>-6</sup>

#### 4.1 Results of Taguchi Design

Table No. 1 was analyzed using the Takuji method, and after conducting the analyzes and tests, we will get the ability of the additives to influence with clay components and probability ratios. Table No. 2 includes the physical analysis of the ceramic filter with additives of rice husk and sawdust by slip casting.

##### Taguchi Orthogonal Array Design

L9(3<sup>2</sup>)

Factors: 2

Runs: 9

Columns of L9(3<sup>4</sup>) Array

1 2

##### Regression Analysis: A versus B

The regression equation is

$$A = 2.000 + 0.0000 B$$

$$S = 0.925820 \quad R-Sq = 0.0\% \quad R-Sq(adj) = 0.0\%$$

##### Analysis of Variance

Source	DF	SS	MS	F	P
--------	----	----	----	---	---

Regression	1	0	0.000000	0.00	1.000
------------	---	---	----------	------	-------

Error 7 6 0.857143

Total 8 6

### Test for Equal Variances: A versus B

Method

Null hypothesis all variances are equal

Alternative hypothesis At least one variance is different

Significance level  $\alpha = 0.05$

95% Bonferroni Confidence Intervals for Standard Deviations

B	N	StDev	CI
1	3	1	(0.0131425, 376.665)
2	3	1	(0.0131425, 376.665)
3	3	1	(0.0131425, 376.665)

Individual confidence level = 98.3333%

Tests

Test

Method	Statistic	P-Value
Multiple comparisons	—	1.000
Levene	0.00	1.000

### Test for Equal Variances: A vs B

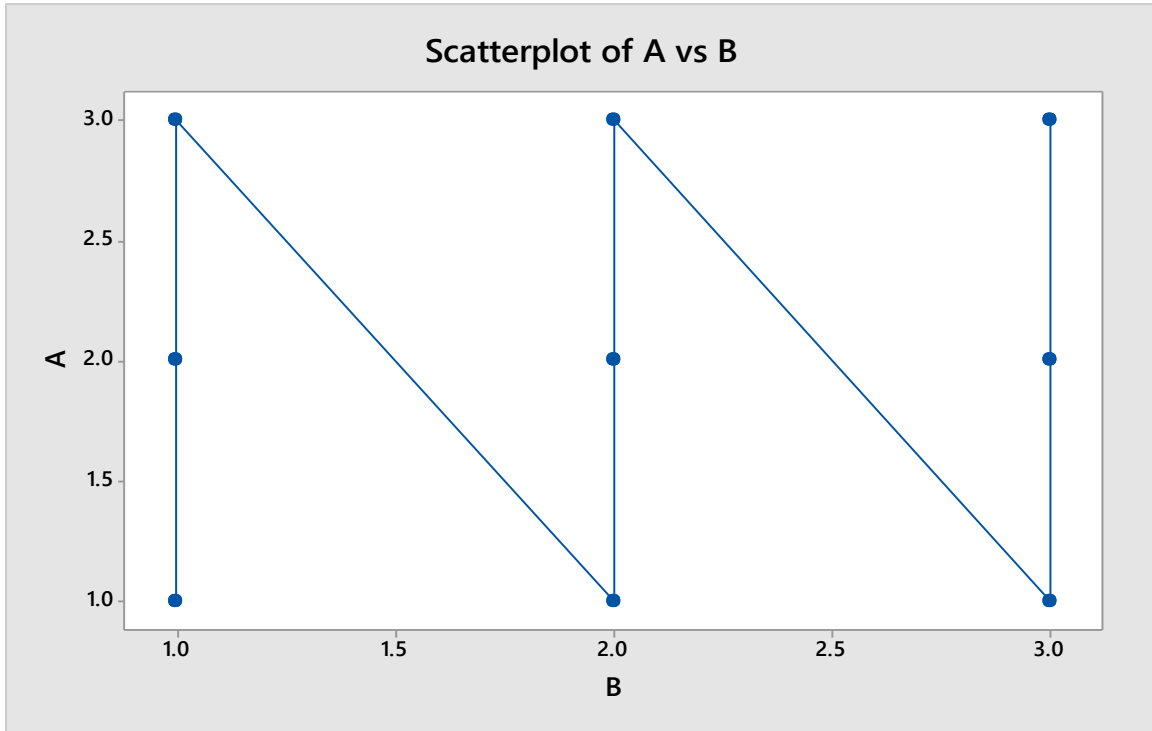


Figure (1) Scatterplot of A vs B

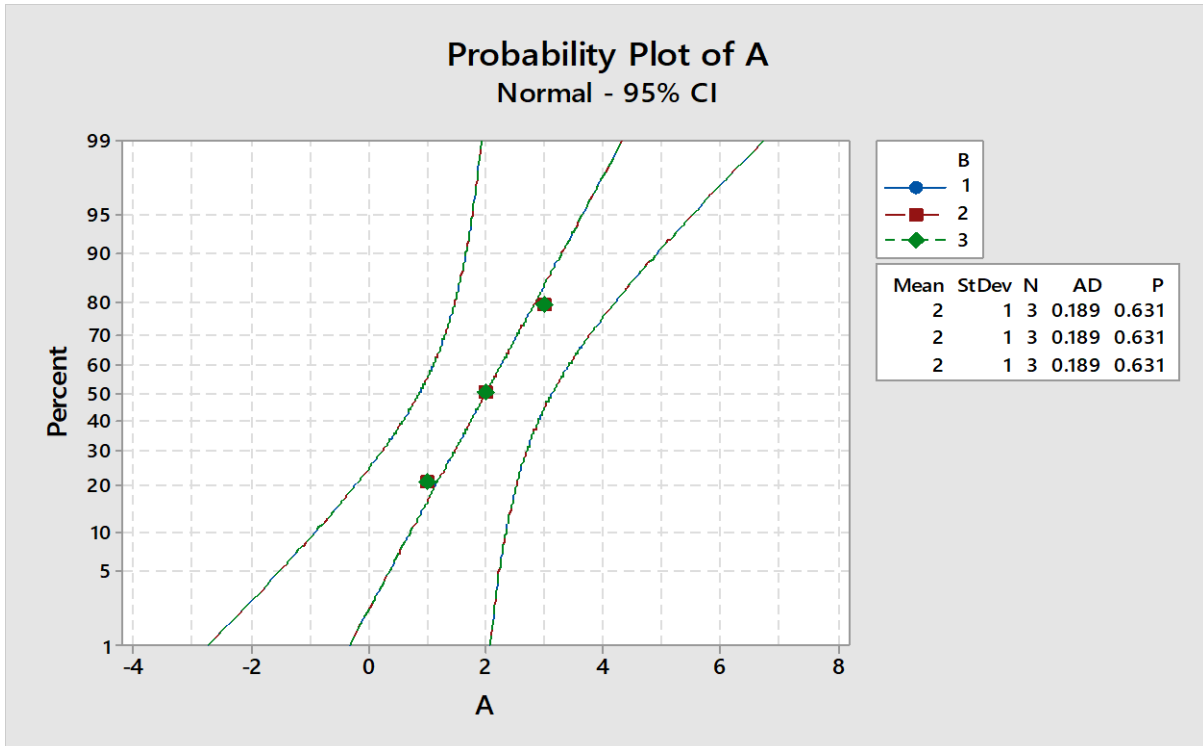
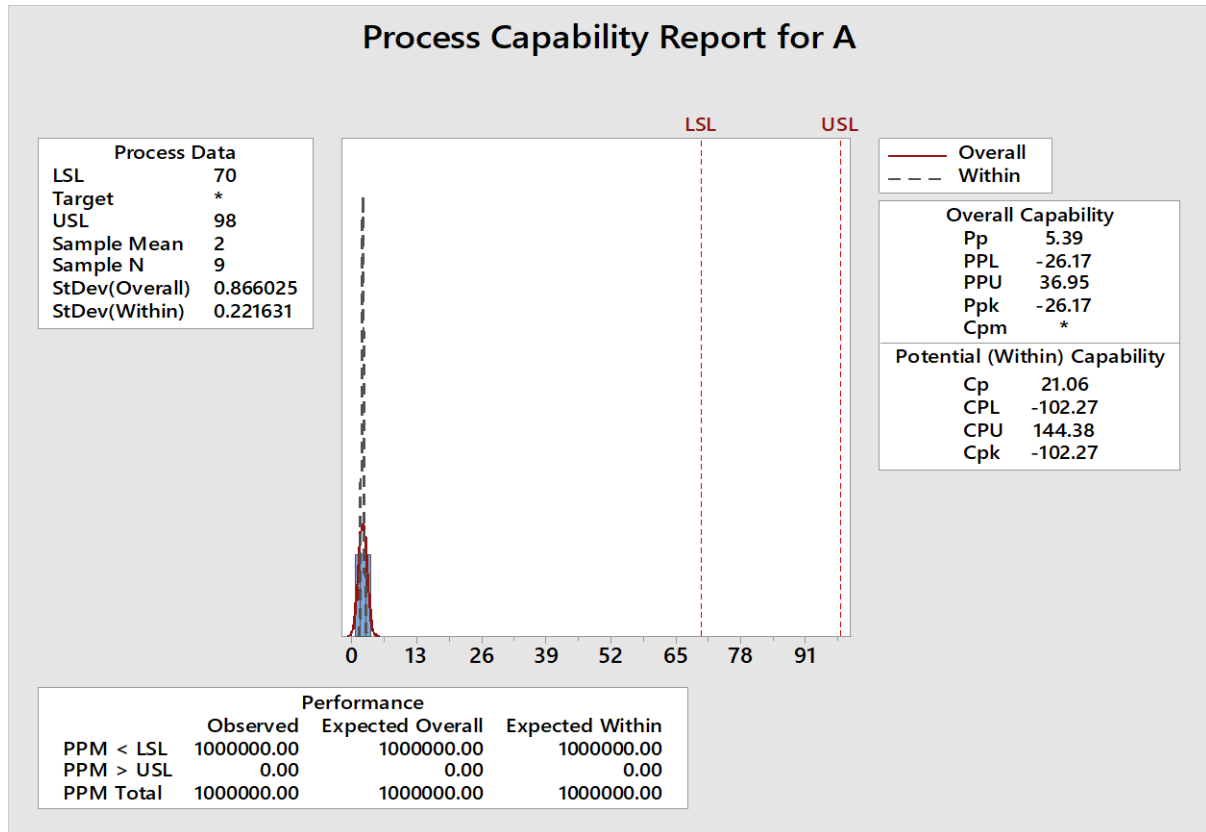


Figure (2) Probability Plot of A





**Figure (3) Process Capability Report for A**

All results above from table 1 for Composition of red clay and additives mixture ( sawdust and Rice husk)

### Result of physical analysis for a water filter disc with sawdust by slip casting

#### Taguchi Design

Taguchi Orthogonal Array Design

L27(3<sup>2</sup>)

Factors: 2

Runs: 27

Columns of L27(3<sup>13</sup>) Array

1 2

## Results for: Central Composite Design

Factors: 2 Replicates: 1

Base runs: 14 Total runs: 14

Base blocks: 2 Total blocks: 2

Two-level factorial: Full factorial

Cube points: 4

Center points in cube: 3

Axial points: 4

Center points in axial: 3

$\alpha$ : 1.41421

It is noting that the distribution could not be fit. The number of distinct rows of data;

- \* in porosity (for addition ratio = 2.00%) must be greater than or
- \* Equal to the number of estimated distribution parameters.

As well, it can see in the porosity that the distribution could not be fit. The number of distinct rows of data because of the following reasons;

- \* In porosity (for addition ratio = 4.00%) must be greater than or
- \* Equal to the number of estimated distribution parameters.

While, in the addition ratio, the study is showing that the distribution could not be fit. The number of distinct rows of data;

- \* in porosity (for addition ratio = 6.00%) must be greater than or
- \* Equal to the number of estimated distribution parameters.

Whilst, the distribution could not be fit. The number of distinct rows of data

- \* In porosity (for addition ratio = 8.00%) must be greater than or
- \* Equal to the number of estimated distribution parameters.

However, the distribution could not be fit. The number of distinct rows of data

- \* In porosity (for addition ratio = 10.00%) must be greater than or
- \* Equal to the number of estimated distribution parameters.

Also, the distribution could not be fit. The number of distinct rows of data

- \* in porosity (for addition ratio = 20.00%) must be greater than or
- \* Equal to the number of estimated distribution parameters.

Then the distribution could not be fit. The number of distinct rows of data

- \* in porosity (for addition ratio = 30.00%) must be greater than or
- \* Equal to the number of estimated distribution parameters.

Whereas, the distribution could not be fit. The number of distinct rows of data

- \* In density (for addition ratio = 2.00%) must be greater than or
- \* Equal to the number of estimated distribution parameters.

Accordingly, the distribution could not be fit. The number of distinct rows of data

- \* In density (for addition ratio = 4.00%) must be greater than or
- \* Equal to the number of estimated distribution parameters.

Consequently, the distribution could not be fit. The number of distinct rows of data

- \* In density (for addition ratio = 6.00%) must be greater than or
- \* Equal to the number of estimated distribution parameters.

Subsequently, the distribution could not be fit. The number of distinct rows of data

- \* In density (for addition ratio = 8.00%) must be greater than or
- \* Equal to the number of estimated distribution parameters.

Then, the distribution could not be fit. The number of distinct rows of data

\* In density (for addition ratio = 10.00%) must be greater than or

\* Equal to the number of estimated distribution parameters.

Thus, the distribution could not be fit. The number of distinct rows of data

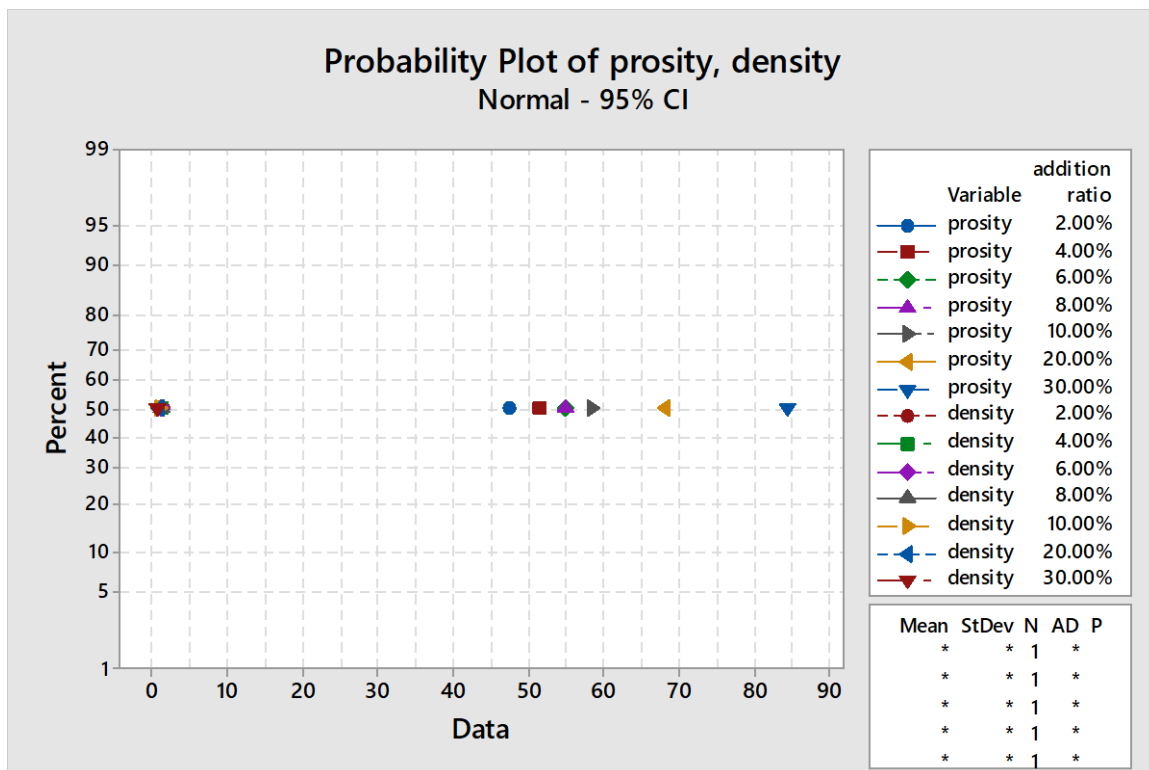
\* In density (for addition ratio = 20.00%) must be greater than or

\* Equal to the number of estimated distribution parameters.

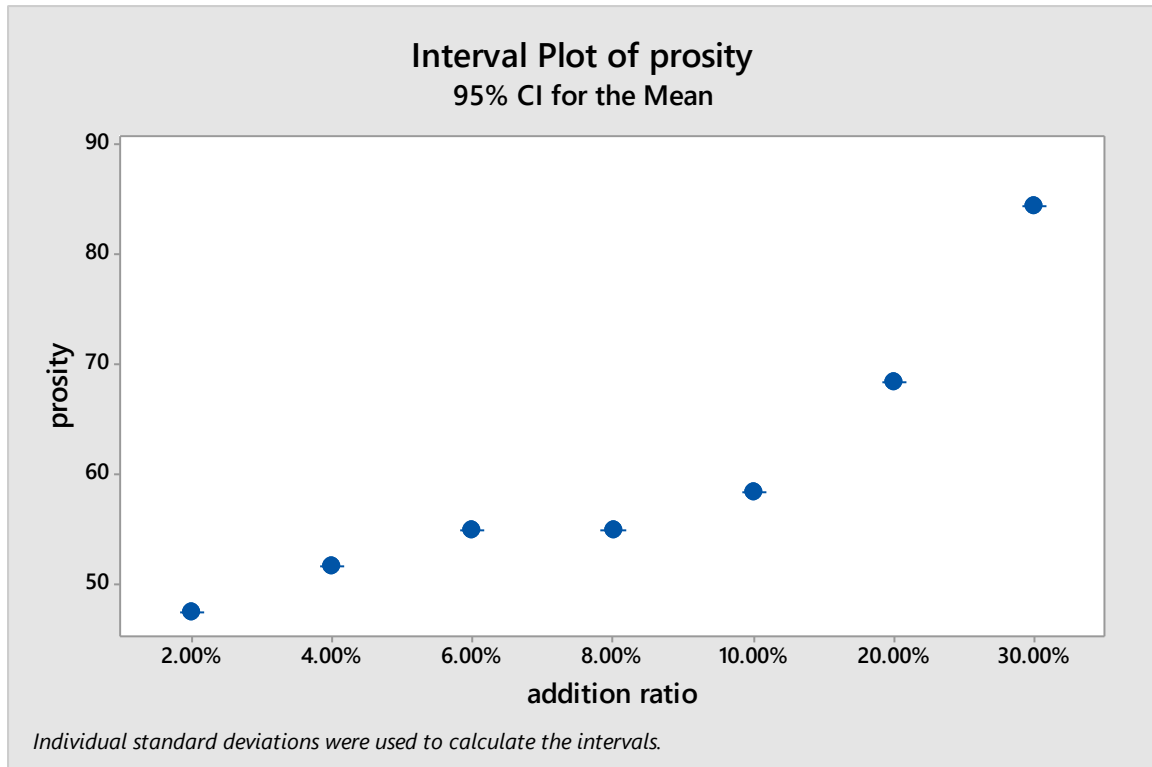
Finally, the distribution could not be fit. The number of distinct rows of data

\* in density (for addition ratio = 30.00%) must be greater than or

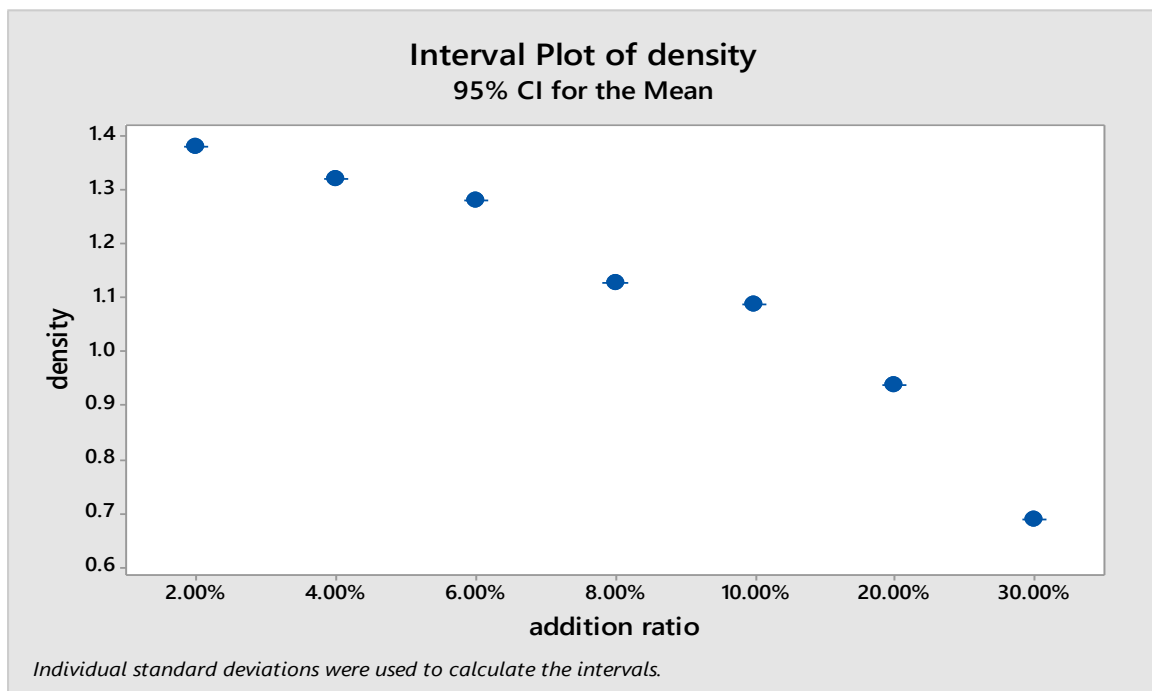
\* Equal to the number of estimated distribution parameters.



**Figure (4) Results for: Probability Plot of porosity, density**



**Figure (5) Interval Plot of porosity**



**Figure (6) Interval Plot of density**

According to the Interval Plot of density analysis , the following accrued notes achieved;

1. The distribution could not be fit. The number of distinct rows of data
  - \* in porosity (for addition ratio = 2.00%) must be greater than or
  - \* Equal to the number of estimated distribution parameters.
2. The distribution could not be fit. The number of distinct rows of data
  - \* In porosity (for addition ratio = 4.00%) must be greater than or
  - \* Equal to the number of estimated distribution parameters.
3. The distribution could not be fit. The number of distinct rows of data
  - \* in porosity (for addition ratio = 6.00%) must be greater than or
  - \* Equal to the number of estimated distribution parameters.
4. The distribution could not be fit. The number of distinct rows of data
  - \* In porosity (for addition ratio = 8.00%) must be greater than or
  - \* Equal to the number of estimated distribution parameters.
5. The distribution could not be fit. The number of distinct rows of data
  - \* In porosity (for addition ratio = 10.00%) must be greater than or
  - \* Equal to the number of estimated distribution parameters.
6. The distribution could not be fit. The number of distinct rows of data
  - \* In porosity (for addition ratio = 20.00%) must be greater than or
  - \* Equal to the number of estimated distribution parameters.
7. The distribution could not be fit. The number of distinct rows of data
  - \* In porosity (for addition ratio = 30.00%) must be greater than or
  - \* Equal to the number of estimated distribution parameters.
8. The distribution could not be fit. The number of distinct rows of data
  - \* In density (for addition ratio = 2.00%) must be greater than or
  - \* Equal to the number of estimated distribution parameters.

9. The distribution could not be fit. The number of distinct rows of data
  - \* In density (for addition ratio = 4.00%) must be greater than or
  - \* Equal to the number of estimated distribution parameters.
10. The distribution could not be fit. The number of distinct rows of data
  - \* In density (for addition ratio = 6.00%) must be greater than or
  - \* Equal to the number of estimated distribution parameters.
11. The Distribution could not be fit. The number of distinct rows of data
  - \* In density (for addition ratio = 8.00%) must be greater than or
  - \* Equal to the number of estimated distribution parameters.
12. The distribution could not be fit. The number of distinct rows of data
  - \* In density (for addition ratio = 10.00%) must be greater than or
  - \* Equal to the number of estimated distribution parameters.
13. The distribution could not be fit. The number of distinct rows of data
  - \* In density (for addition ratio = 20.00%) must be greater than or
  - \* Equal to the number of estimated distribution parameters.
14. The distribution could not be fit. The number of distinct rows of data
  - \* in density (for addition ratio = 30.00%) must be greater than or
  - \* equal to the number of estimated distribution parameters.

### **Empirical CDF of porosity, density**

The analysis of empirical cumulative distribution functions is using to understand “distributions”, or more specifically, “probability distributions”. If it sum those probabilities we get 1. And this represents the “probability distribution” for our event. Formally this event follows a Binomial distribution because the events are independent, there are a fixed number of trials (3), the probability is the same for each flip (0.5), and our outcome is the number of “successes” in the number of trials. In fact what we just demonstrated is a binomial distribution with 3 trials

and probability equal to 0.5. This is sometimes abbreviated as  $b(3,0.5)$ . We can quickly generate the probabilities in R using the `dbinom` function:

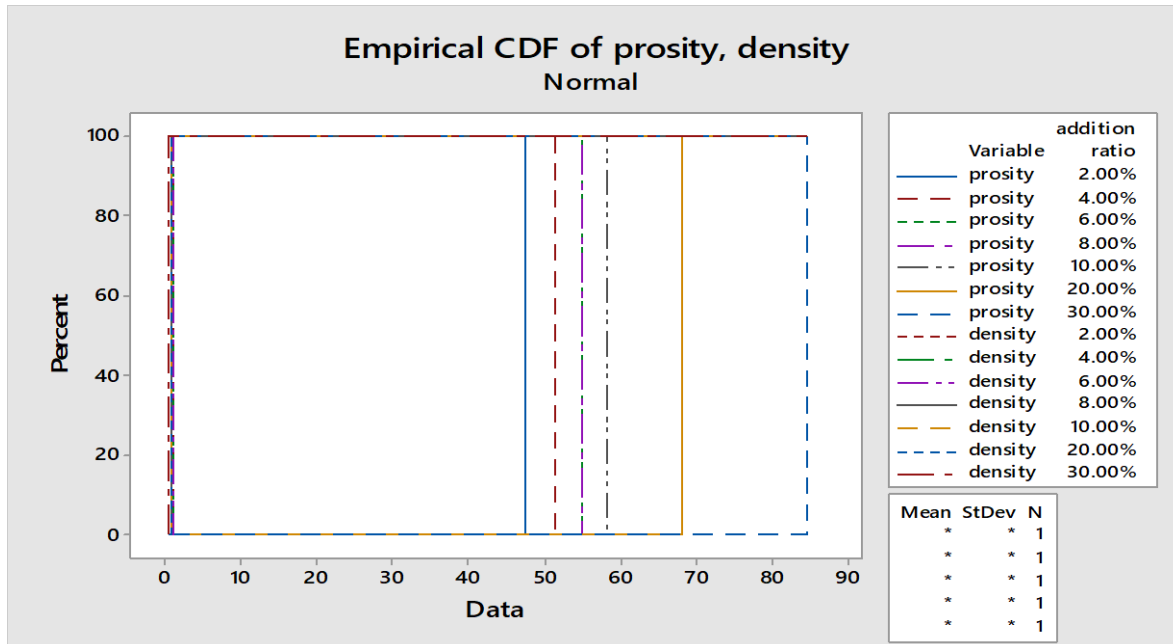


Figure 7. CDF of porosity, density

These are probabilities that accumulate as it move from left to right along the x-axis in our probability distribution. Looking at the distribution plot above.

## 4.2 Discussion

According to the Taguchi method, compared to Table 1 (Forming a mixture of red clay, sawdust additives and rice husks), as this method involves several steps, including regression analysis and the equal difference test, where the results showed:

### 4.2,1- Process capacity report for A

Which includes the comprehensive ability and latent ability of process. Table 1 showed the effect of adding sawdust and rice husk on the porous ceramic filter. The results showed that the porosity increases with the increase in the percentage of addition Both sawdust and rice husk. As for Table No. 2, we note that as the percentage of addition increases (2% , 4% , 6%, 8%, .....30%), we notice the difference in the density of the ceramic filter, that is, it decreases gradually because the additions of rice husk and sawdust lead to an increase in the porosity of the ceramic filter. therefore the density value decreases (Porosity is inversely proportional to density). according to results Takuji method with Table No. 2,



where it shows us in the drawing whenever The percentages of addition increase as the density decreases, and vice versa .As for the porosity of Table No. 2, the higher the percentages of The addition leads to a gradual increase in porosity. As for the results of fit plot porosity and density when combined Percentage of materials, different grades will appear for each Density and porosity.

#### 4. 2. 2. Taguchi design steps:

##### I. Taguchi Method Design of Experiments

1- Define the process objective, or more specifically, a target value for a performance measure of the process. This may be a flow rate, temperature, etc. The target of a process may also be a minimum or maximum; for example, the goal may be to maximize the output flow rate. The deviation in the performance characteristic from the target value is used to define the loss function for the process.

2- Determine the design parameters affecting the process. Parameters are variables within the process that affect the performance measure such as temperatures, pressures, etc. that can be easily controlled. The number of levels that the parameters should be varied at must be specified

3- Create orthogonal arrays for the parameter design indicating the number of and conditions for each experiment. The selection of orthogonal arrays is based on the number of parameters and the levels of variation for each parameter, and will be expounded below.

4- Conduct the experiments indicated in the completed array to collect data on the effect on the performance measure.

5- Complete data analysis to determine the effect of the different parameters on the performance measure

##### II. Taguchi Loss Function:

The goal of the Taguchi method is to reduce costs

##### III. Determining Parameter Design Orthogonal Array

The effect of many different parameters on the performance characteristic in a condensed set of experiments can be examined by using the orthogonal array

experimental design proposed by Taguchi. Once the parameters affecting a process that can be controlled have been determined, the levels at which these parameters should be varied must be determined. Determining what levels of a variable to test requires an in- depth understanding of the process, including the minimum, maximum, and current value of the parameter. If the difference between the minimum and maximum value of a parameter is large, the values being tested can be further apart or more values can be tested. If the range of a parameter is small, then less values can be tested or the values tested can be closer together

#### IV. Array Selector

#### V. Links to Orthogonal Arrays

L4 Array

L8 Array

L9 Array

L12 Array

L16 Array

L18 Array

L25 Array

L27 Array

L32 Array

L'32 Array

L36 Array

L50 Array

#### VI. Important Notes Regarding Selection + Use of Orthogonal Arrays

Note 1: The array selector assumes that each parameter has the same number of levels. Sometimes this is not the case. Generally, the highest value will be taken or the difference will be split

Note 2 : If the array selected based on the number of parameters and levels includes more parameters than are used in the experimental design, ignore the additional parameter columns.

## VII. Analyzing Experimental Data

Once the experimental design has been determined and the trials have been carried out, the measured performance characteristic from each trial can be used to analyze the relative effect of the different parameters. To demonstrate the data analysis procedure, the following L9 array will be used, but the principles can be transferred to any type of array.

.The Taguchi method was used in this test, which includes orthogonal rows (orthogonal matrix) and this method is carried out according to the following steps:

- 1- Regression analysis, that is, through the regression equation
- 2- Analysis of variance
- 3- Testing of equal differences and then we run the tests, the statistical method test. (Equal differences test to get the probability chart which is a scatter plot and then the process ability report.)

## Conclusion

1- The local raw material red clay, sawdust and rice, husk it observes that bulk density and strength decrease with increase sawdust that decreases due to higher porosity and the porosity of the sample could be controlled by varying the percentage sawdust mixture porosity.

2- The pores of sawdust are larger which gives high porosity and low density rice husk pores.

3- Also, the sample is slightly affected due to the presence of two additives. However, the examined clay samples are suitable for use in lining heat treatment furnaces, as well as clay samples with additives can be used as ceramic materials.

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