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SCHOOL OF CIVIL AND ENVIRONMENTAL ENGINEERING

THE EFFECT OF METAKAOLIN ON THE COMPRESSIVE STRENGTH OF RECYCLED AGGREGATE CONCRETE

A Thesis in Structural Engineering

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A Thesis

Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Science

The undersigned have examined the thesis entitled '**The Effect of Metakaolin on the Compressive Strength of Recycled Aggregate Concrete**' presented by **Aron Berhanu**, a candidate for the degree of **Master of Science** and hereby certify that it is worthy of acceptance.

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ABSTRACT

Large quantity of construction and demolition waste has been produced due to the economic growth of the world. However, the most common way to dispose of such waste is by landfill. Without proper sorting and handling of it, as time passes, landfills will cause many environmental issues. Furthermore, the continuous utilization of natural aggregates will cause resource scarcity. The potential solution for these problems is the recycling of aggregates. Still, due to strength reduction associated with recycled aggregates, its usage is restricted for low strength concrete.

On the other hand, incorporation of metakaolin as a cement replacement in recycled aggregate concretes could pave a path towards a sustainable future. In addition, kaolin, the original material from which metakaolin is produced, is found in large quantities in Ethiopia which makes it an attractive option.

This research presents the effect of metakaolin as a cement replacement on the properties of fresh and hardened recycled coarse aggregate concrete as well as natural coarse aggregate concrete. The recycled coarse aggregate is extracted from first-hand cast laboratory cubes with known average compressive strength. Different types of concrete mixtures are tested: concrete made with natural coarse aggregate as a control concrete, concrete made with recycled coarse aggregate (0%, 25%, 50%, 75% and 100% replacement of natural coarse aggregate), concrete made with metakaolin as cement replacement (0%, 6%, 12%, 18%, and 24% replacement of cement), and concrete made with recycled coarse aggregate (0%, 25%, 50%, 75% and 100% replacement of natural coarse aggregate) with metakaolin as cement replacement (6%, 12%, and 18% replacement of cement). Additionally, a separate experiment has been conducted to check the production of metakaolin from locally found kaolin samples.

The outcomes of this research show that the use of metakaolin as a cement replacement improved the compressive strength of recycled coarse aggregate concrete and even resulted in a compressive strength almost equal to natural coarse aggregate concrete. In addition, this thesis has shown that metakaolin can be produced from locally occurring kaolin by thermal activation.

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LIST OF ABBREVIATIONS

ACI	American Concrete Institute
Al_2O_3	Aluminum Oxide
$Al_2Si_2O_5(OH)_4$ or ($Al_2O_3.2SiO_2.2H_2O$)	Kaolinite
aq.	Aqueous
ASTM	American Society of Testing Materials
C_2ASH_8	Hydrated Gehlenite
C_2S or (Ca_2SiO_4)	Belite or Dicalcium Silicate
C_3A or ($Ca_3Al_2O_6$)	Aluminate or Tricalcium Aluminate
C_3S or (Ca_3SiO_5)	Alite or Tricalcium Silicate
C_4AF or (Ca_2AlFeO_5)	Ferrite is Tetracalcium Aluminoferrite
$Ca(OH)_2$	Calcium Hydroxide or Portlandite
C-A-H or ($4CaO.Al_2O_3.13H_2O$)	Calcium Aluminate Hydrate
CaO	Calcium Oxide
CEM	Cement
CES	Compulsory Ethiopian Standard
CH	Calcium Hydrate
cm	Centimeter
CO_2	Carbon di-oxide
C-S-H or ($xCaO.ySiO_2$.aq.)	Calcium Silicate Hydrate
Cukα	Copper radiation source
D_{tg}	Degree of dehydroxylation
E	Modulus of elasticity
FA	Fly ash
Fe	Iron
Fe_2O_3	Iron Oxide
g	Gram

G	Shear modulus
GPa	Giga Pascal
H_2O	Water
hrs	Hours
ISSA	Incinerated sewage sludge ash
ITZ	Interfacial Transition Zone
K	Bulk modulus
K_2O	Potassium Oxide
Kg	Kilogram
KJ	Kilo Joule
Km	Kilometer
l	Liter
LOI	Loss on Ignition
m	Residual mass loss
m_0	Maximum mass loss
m^2	Square meter
m^3	Cubic meter
MgO	Magnesium Oxide
min	Minute
MK or ($Al_2O_3.2SiO_2$)	Metakaolin
ml	Milliliter
mm	Millimeter
mmol	Millimole
MnO	Manganese Oxide
MPa	Mega Pascal
Na_2O	Sodium Oxide
NAC	Natural aggregate concrete
NCA	Natural coarse aggregate

NIST	National Institute of Standards and Technology
OH	Hydroxide
OPC	Ordinary Portland Cement
P_2O_5	Phosphorous Oxide
PDF	Power diffraction file
PH	Power of hydrogen
PSD	Particle size distribution
RAC	Recycled aggregate concrete
RCA	Recycled coarse aggregate
SAI	Strength Activity Index
SEM	Scanning Electron Microscopy
SF	Silica fume
SiO_2	Silicon di-oxide
SSD	Saturated Surface dry condition
Ti	Titanium
TiO_2	Titanium Oxide
UK	United Kingdom
USA	United States of America
USD	United States Dollar
VCCTL	Virtual Cement and Concrete Testing Laboratory
XRD	X-ray Powder diffraction
ν	Poisson's ratio
^0c	Degree Celsius
µm	Micrometer

CHAPTER 1 INTRODUCTION

1.1 General background of the study

Demolition of structures due to change of purpose, structural degradation, expansion of traffic directions and increasing traffic load, natural disasters, rearrangement of a city, etc. and their replacement with new ones, is a common phenomenon today in a large part of the world. These demolished materials are commonly disposed to landfills. Consequently, a massive amount of construction waste is created and this, in turn, pollutes the environment on a large scale.

On the other hand, concrete being one of the most widely used material in the construction industry, its consumption is rapidly increasing each day. Proportionally the consumption of natural aggregate will increase since natural aggregate occupies the largest component of concrete. This trend leads to a question whether or not the appropriate resource utilization and preservation technique are being applied worldwide.

The key to solving these issues is the recycling of demolished concrete structures. For its environmental benefits, the reuse of concrete is necessary in order to reduce the demand for land for waste disposal and the effective use of resources. So far, a number of studies have been conducted on the mechanical properties of recycled aggregate concrete. one basic conclusion confirmed is, the compressive strength of concrete prepared with natural aggregates is higher than that of recycled aggregate concrete. Hence, due to this problem recycled aggregates are practically employed only in low strength concrete. Consequently, Engineers' concern has shifted to improving the strength of recycled aggregate concrete using material technology.

There are many ways to improve the compressive strength of recycled aggregate concrete, but the one that is chosen for this research is metakaolin as a cement replacement. Metakaolin is made from thermal activation of kaolin clay where there is plenty of kaolin raw material in Ethiopia. Further cement production is one of the major reasons for CO_2 emission which will be minimized in the utilization of metakaolin as cement replacement.

The main purpose of this work is to determine the basic properties of recycled coarse aggregate concrete with a metakaolin base. Concrete made of different percentages of recycled coarse aggregate and metakaolin are compared to the properties of concrete made with natural coarse aggregate-control concrete. Fine recycled aggregate will not be considered for recycled aggregate concrete production because its application in structural concrete is generally not recommended.

1.2 Statement of the problem

Although Recycled aggregate usage is limited in Ethiopia, leading countries in Recycle aggregate production like Germany and UK have shown that recycled aggregate is practical for low strength concrete, and to a restricted extent, for a few structural grade concretes. Hence it is necessary to improve the strength of recycled aggregate concrete for further and future application. Additionally, rather than searching for a solution, continuous utilization of natural aggregates will not only diminish the abundance of natural aggregate but also creates free demolished structural waste that is disposed of as a landfill which causes air and water pollution.

1.3 Objective of the study

1.3.1 General objective

Comparing the compressive strength of concrete made of natural coarse aggregate with that of concrete made of recycled coarse aggregate with metakaolin as a cement replacement.

1.3.2 Specific objective

- Investigating the effects of incorporating recycled coarse aggregate and metakaolin on the compressive strength of concrete.
- Checking whether metakaolin could be produced in the laboratory from kaolin samples available in Ethiopia.
- Comparing laboratory results with that of software simulations.

1.4 Scope of the study

A. Review and research of concrete properties made of recycled coarse aggregate and metakaolin.
B. Review and research of environmental and economic feasibility of recycled coarse aggregate and metakaolin.
C. Casting and testing of concrete specimens made of recycled coarse aggregate and metakaolin.
D. Review and research of metakaolin production and trying to produce metakaolin in Ethiopia.
E. Analysis of test results and recommendations for further research area.

1.5 Significance of the study

The successful integration of RCA into the construction industry can have a major impact on alleviating environmental problems, such as pollution associated with construction and demolition waste and the increased demand for land to dispose of such waste. In addition to this metakaolin, the material used to increase the compressive strength of the RCA concrete is used as a cement replacement. Using this material can have two basic advantages, one of which is the reduction of CO_2 emissions from cement production and the other is the opening of a potential market for the production of metakaolin in Ethiopia since there is a large amount of kaolin mineral from which metakaolin is produced.

1.6 Thesis structure

This research consists of seven chapters. The first chapter highlights the overall idea, major problems that will be investigated, objectives, scope, and significance of this research paper. Chapter two deals with previous works done on recycled aggregate and metakaolin. Chapter three briefly describes the materials used for experimentation. Chapter four explains the experimental methods and procedures used in this research. Chapter five discusses the results of the experiments, followed by analysis and interpretation. Chapter six compares the experimental results with that of simulations done using VCCTL software, which is developed by the National Institute of Standards and Technology. Finally, conclusions and basic recommendations for future works are drawn in chapter 8.

CHAPTER 2 LITERATURE REVIEW

2.1 Concrete

Concrete as a building material has been used for numerous years in the largest quantity. Concrete is defined as “a composite material that consists essentially of a binding medium within which are embedded particles or fragments of aggregate; in hydraulic cement concrete, the binder is formed from a mixture of hydraulic cement and water.” (ASTM C 125 2004).

Consumption of concrete worldwide is estimated to be around 11 billion metric tons per year. The reason for this can be found in the outstanding technical characteristics of concrete as well as in the economy of this material. The three primary reasons would be, one, because of its resistance to water making it an ideal building material for regulating, transporting and carrying water.

The second reason would be its versatility (ability to adapt a variety of shapes and sizes) mainly because of its plastic consistency that allows the material to flow into prefabricated shaping. And once the concrete has solidified the formwork could be removed.

The third reason would be because of its abundance as well as the material’s economy. The main components for making concrete are relatively inexpensive, namely aggregate, water, and Portland cement, and are commonly available (Mehta and Monteiro 2006).

2.1.1 Strength of concrete

The strength of a material is its capacity to withstand applied loads without failure or plastic deformation. The strength of concrete is highly crucial in concrete design and quality control and this is mainly because, compared to testing other properties of concrete, testing its strength is relatively easier. Furthermore, other properties are directly related to its strength and since concrete is more suitable in compressive load than tensile, the compressive strength of concrete is generally specified (Mehta and Monteiro 2006).

Concrete ingredient properties have a major impact on the properties of both fresh and hardened concrete. To begin with, aggregates, which consists of 60-80% of concrete, must fulfill certain requirements as well as being clean from chemicals or any other impurities that could affect the hydration process of concrete. Second, the properties of cement, a raw material composed of limestone, shale, clay and iron ore must have the right amount of lime, silica, alumina and iron components. The third factor that affects the strength of concrete would be the paste composed of cement, water, and entrained air, which in turn is affected by the water to cement ratio. Although lower water to cement ratio cold make the concrete stronger, it would also make it harder to work with. Thus, to attain the required strength while preserving workability, the correct balance must be achieved. The fourth factor affecting concrete would be water specifically the PH level. Almost all natural water used for drinking can be used for mixing but not vise-versa. It is generally preferable to use water that has a PH level between 4.5 to 8.5 (Fassara Wannan Shafi 2015).

2.1.2 Workability of concrete

Workability is the property of fresh concrete which indicates the ease of placement. Workable concrete implies concrete that can be positioned and readily compacted without separation. Once the mixing has been done, the concrete undergoes various processes like transportation, flow, finishing, and compaction. In all four procedures, concrete must be able to meet the requirements without difficulty.

One factor which affects the workability of concrete is the cement content in concrete. The more we add cement to the mix, the more the paste would be able to coat the surface of aggregate. This will assist in decreasing friction between aggregates and facilitate smooth movement of aggregates, making it more workable during mixing, transporting, placing and compacting of concrete.

On the other hand, adding water to the mix could either improve or decrease the workability of concrete. Not adding enough amount of water affects the mixing processes and makes it difficult to work with. While adding too much water could result in the segregation of the concrete and decrement of its workability. The type of cement and aggregates we use also affects its workability. Increasing the fineness of the cement requires additional water to make it more workable. And an increase in the size of aggregate makes it necessary to add in more cement to make it more workable.

Most standards specify a number of different tests, and the cause for this, of course, is that none of the tests is capable of dealing with the full range of workability that is of interest in practice (Tattersall 2014).

2.2 Recycled aggregate

The fast rise in building operations and the big quantity of construction and demolition waste make an enormous contribution to total solid waste. In the past, distinct materials have been recycled in distinct forms. In the latest past, the recycling of coarse aggregates, produced by demolished or destroyed waste concrete, has drawn scientists. Coarse aggregates, one of the most significant components of concrete, are becoming more expensive in terms of the growing price and accessibility of equipment. The recycling of large aggregates is therefore of the utmost significance to overcome this problem. The use of recycled aggregates is very essential in order to achieve sustainability in the building industry, as it decreases construction and demolition waste and thus decreases environmental pollution by preserving natural resources (Rao et al. 2019).

Recycled aggregates generally exhibit specific features such as higher porosity and absorption, and reduced density and strength than natural aggregates. In addition, some RAC studies show distinctions in the features of the interfacial transition areas between cement paste and aggregates (Casuccio et al. 2008). Furthermore, Tam et al. stated that the RAC was of low-quality due to greater water absorption, greater porosity, and weaker ITZ; in order to improve the ITZ, the strength and the mechanical behavior of RAC concrete, they altered the mixing method (Tam et al. 2005).

Padmini et al. summarized the effect RAC from investigation of many researches, as shown below:

- The crushing method of parent concrete has a major effect on the recycled aggregate;
- The particle shape of the recycled aggregate is more irregular than the natural aggregate and also has a thicker surface;
- For the same workability, RAC requires more water than normal concrete;
- The density, compressive strength and elasticity modulus of the RAC are comparatively smaller than that of the parent concrete;
- for the same water-cement ratio when compared to NAC risk of reinforcement corrosion, permeability, and rate of carbonation are higher;
- For a rational comparison, the maximum size of the parent and RAC aggregates should be kept the same (Padmini et al. 2009).

Therefore, in order to make more efficient use of recycled concrete aggregate as a concrete aggregate, it is essential to improve the quality and characteristics of the attached weak mortar. The treatment methods to enhance the characteristics of recycled concrete aggregates are directly linked to the attached mortar. Accordingly, Shaban et al. stated that the main techniques are classified into two primary categories, which are eliminating the attached mortar from recycled concrete aggregate using different methods and improving the properties of recycled concrete aggregate by improving the quality of the adhered mortar (Shaban et al. 2019). The full recycled concrete aggregate treatment techniques are presented in **Figure 2-1**.

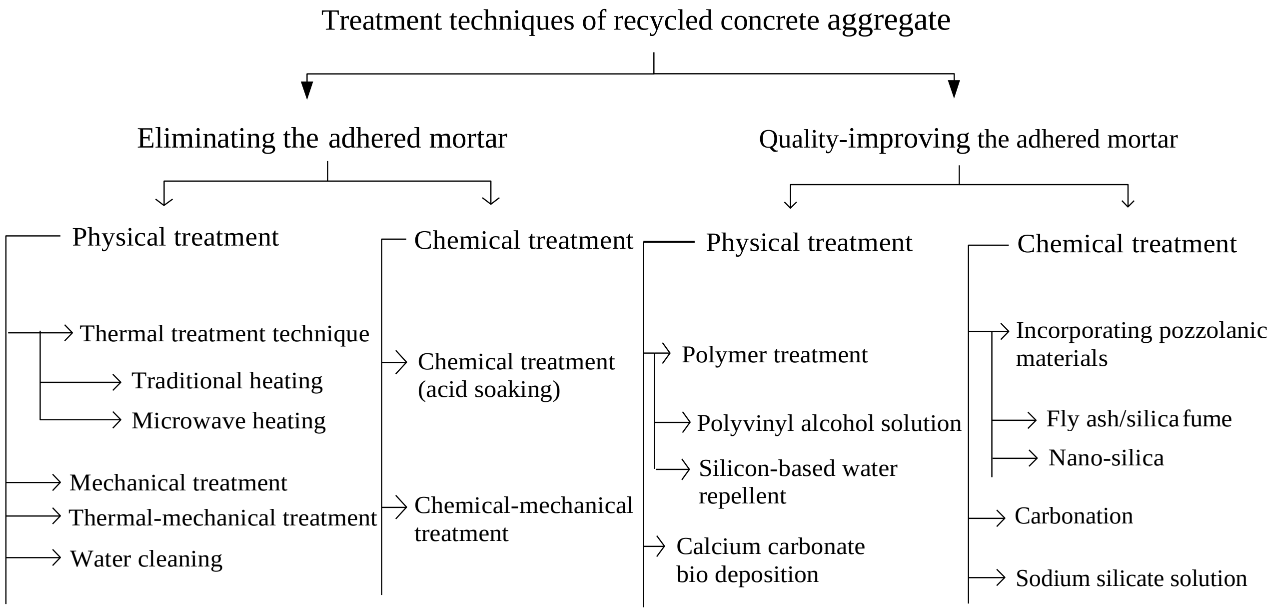

Figure 2-1: Treatment techniques of recycled concrete aggregate (Shaban et al. 2019)

2.2.1 Environmental impact of recycled aggregate

Due to the rapid growth of the construction industry, there is a rise in demand for sand and crushed stone. The over-exploitation of mountains for rocks and rivers for sand is severely destroying the ecological environment to meet this requirement. For instance, as one of the fastest-growing economies in the world, in latest years, China has encountered higher rates of natural aggregate depletion in some areas, leading in higher costs for rock and sand transport. Meanwhile, the generation of construction and demolition wastes is increasing and that it is estimated that there are presently around 200 million tons of waste concrete produced annually in Europe only, but only 30% of the waste is recycled (Shaban et al. 2019).

Waste recycling can significantly decrease damage to the environment caused by improper disposal, enhancing the helpful life of landfills and preserve natural resources. The primary benefit of recycling waste concrete from building and demolition is the reuse of substances that would otherwise be categorized as waste. In relation to shortened transport emissions, recycled aggregate can have lower embodied energy, particularly where recycled materials have been reused in close association with the processing site. In addition, recycling and monitored handling of concrete demolition waste will save land use and generate better possibilities to handle other types of waste (Rao et al. 2019).

2.3 Cement

Cement is a complicated combination of inorganic stages, consisting primarily of calcium silicates and aluminates. It is produced by heating a mixture of clay and limestone. The main stages of the typical Portland cement are alite, belite, aluminate and ferrite. Alite is essentially tricalcium silicate (Ca_3SiO_5, C_3S): it generally constitutes 50 - 70% of the total mass. It is the most significant stage in standard cement, in view of the fact that it affects the setting time and the growth of the short-term strength up to 28days.

Belite is dicalcium silicate (Ca_2SiO_4, C_2S) and makes up 15-30% of ordinary Portland cement clinkers. It contributes little for the first 28 days strength because it reacts slowly with water. It mainly contributes to later age strength.

Aluminate is a tricalcium aluminate ($Ca_3Al_2O_6$, C_3A) consisting of 5-10% of the most common Portland cement clinkers. It reacts quickly with water and it can cause undesirable fast setting unless a set-control agent, generally gypsum, is added.

Ferrite is tetracalcium aluminoferrite (Ca_2AlFeO_5, C_4AF) consisting of 5-10% of the most common Portland cement clinkers. The rate at which it reacts with water appears to be somewhat variable. It is high initially and intermediate between those alite and belite at later ages (Taylor 1990).

2.3.1 Hydration of cement

The exothermic chemical reactions that occur, when anhydrous cement and water mix, is commonly referred to as hydration. In other words, in the presence of water, silicates and aluminates form hydration products which, in time, produce a firm and hard mass - the hydrated cement paste.

There are two ways in which cement-type compounds can react with water: first, there is a direct addition of some water molecules, which is a true reaction of hydration. The second type of water reaction is hydrolysis, the nature of which can be explained using the C_3S hydration equation.

$3CaO.SiO_2 + H_2O = Ca(OH)_2 + xCaO.ySiO_2.aq.$ (calcium silicate hydrate)

The progress of hydration of cement can be determined by different methods:

- The amount of $Ca(OH)_2$ can be measured from the results of hydration of silicates;
- The heat released by hydration;
- The specific gravity of the paste;
- The amount of water combined chemically;
- The amount of cement present unhydrated (using XRD);
- Indirectly from the hydrated paste strength (Potgieter et al. 1999).

2.4 Pozzolanas

In the construction industry, the word ' pozzolana ' applies to all products that respond with lime and water providing calcium silicate and aluminum hydrates with cementing characteristics. As a result, all pozzolanas must be abundant in reactive silica or alumina plus silica. The list of pozzolanic materials contain many natural and artificial materials of different origins and compositions. And the good condition of many ancient constructions still in service today is the best evidence of the outstanding results of pozzolana binders. Pozzolanic binders have been very successful in the past due to some improved characteristics compared to other common binders, such as lime and plaster (Bensted and Barnes 2008).

2.4.1 Pozzolanic materials

Natural pozzolanas may be categorized according to their origination and their vital active constituent. On the primary basis of these criteria, natural pozzolanas are generally categorized as volcanic, tuffs, sedimentary, and diagenetic. Artificial pozzolans may be generated intentionally, e.g. by thermal energy activation of kaolin-clays for the manufacturing of metakaolin, or may be generated as waste or by-products from high-temperature processes such as fly ash from coal-fired electricity manufacturing (Bensted and Barnes 2008).

Donatello et al. compared five pozzolanic materials which are metakaolin (MK), silica fume (SF), fly ash (FA), incinerated sewage sludge ash (ISSA), and sand for their pozzolanic reaction. Accordingly, from the results, metakaolin was the most reactive pozzolanic material. The results of the Frattini test and strength activity index test are shown below (Donatello et al. 2010).

Table 2-1: Frattini test results (Donatello et al. 2010)

Material	(OH) mmol/l	(CaO) mmol/l	Theoretical max. (CaO) mmol/l	(CaO) reduction %
Control	57.4	8.2	8.3	0.6
Sand	53.8	9.9	9.0	-9.6
ISSA	61.3	8.1	7.6	1.6
FA	53.2	4.5	9.2	50.7
SF	35.4	11.5	17.2	33.1
MK	37.0	0.9	15.9	94.4

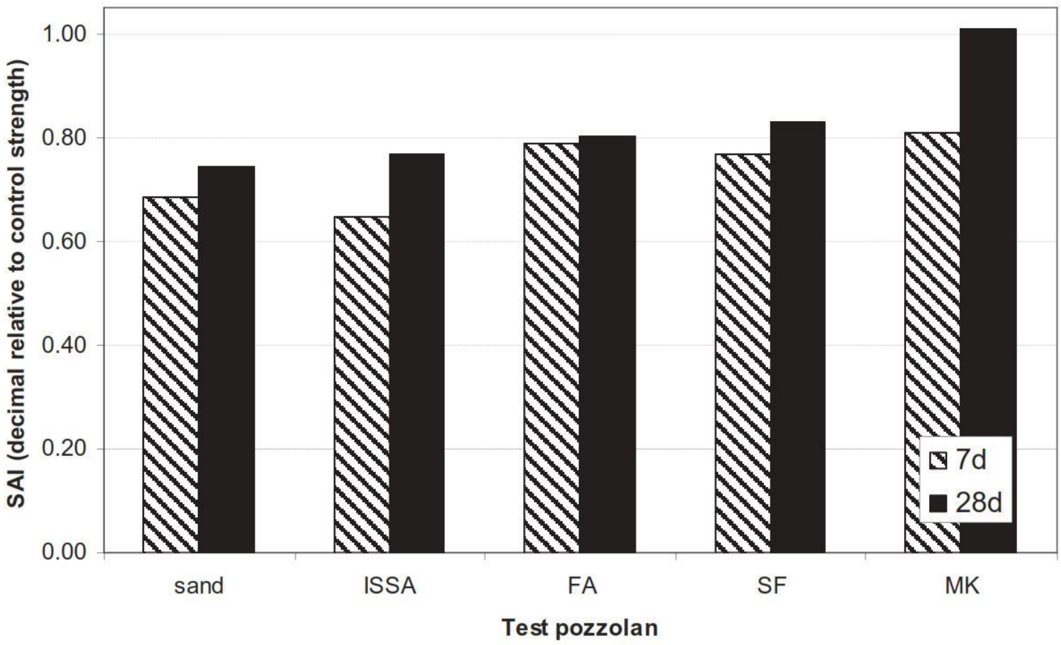

Figure 2-2: Strength activity index results (Donatello et al. 2010)

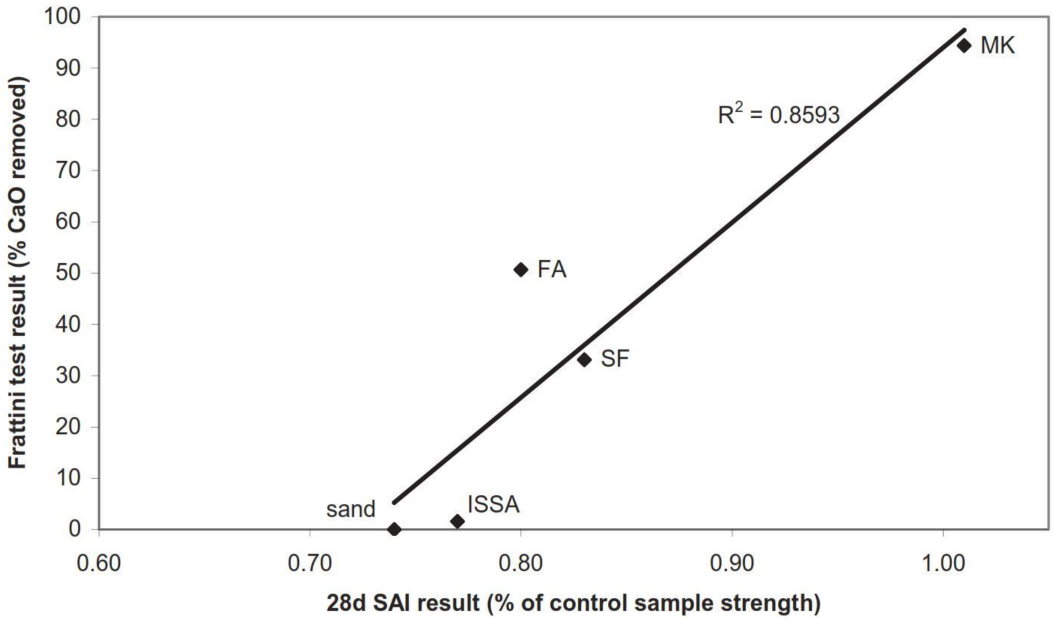

Figure 2-3: Correlation between measured pozzolanic activity using Frattini test and the strength activity index (Donatello et al. 2010)

2.4.2 Pozzolanic reaction

The term ' pozzolanic reaction ' involves all responses taking place in the reactive stages of pozzolana, lime, and water. Under the chemical thermodynamic laws, any mixture of silica or silicate with lime and water should offer rise to calcium silicate hydrates, but the response will only be efficient if the system is enabled or if it includes activated stages. For example, the first case is the reaction between quartz and lime, which only becomes effective when the mixture is autoclaved at an appropriate temperature. The second case is attainable at normal temperatures as the pozzolanas contain unstable phases.

Progress in the pozzolanic reaction is demonstrated by the increase in silica and alumina formed by the Florentin attack in the hardened paste and the decrease of $Ca(OH)_2$.

The primary hydrated phases arising from the reaction of lime with volcanic pozzolanas are:

- C-S-H;
- $4CaO.Al_2O_3.13H_2O$ ((C-A-H), more or less carbonated);
- C_2ASH_8 (hydrated gehlenite).

The proportion of lime mixed with pozzolanas varies between broad boundaries and depends on variables linked to the nature of the pozzolana and the features of the mixture.

The term ' pozzolana nature ' involves the chemical and mineral structure as well as the specific surface of the pozzolana, while the term ' mix features ' refers to the circumstances under which the pozzolana response occurs. The main factors affecting the pozzolanic reaction are:

- nature and composition of the active phases;
- their content in pozzolana;
- their specific surface
- the lime/pozzolana ratio of the mix;

The rate of pozzolanic reaction can be enhanced by using a variety of means: grinding and firing of pozzolanas, compacting mixtures and adding some chemicals to the binder output (Bensted and Barnes 2008).

2.5 Metakaolin

The development of building products offering technical and environmental advantages is the primary challenge of the new millennium. One such material is metakaolin, which is categorized as a fresh generation of additional cementitious material. Supplementary cement materials are finely ground materials used to replace parts of the clinker in a cement or cement in a concrete mix. Metakaolin is distinctive in that it is neither a by-product of an industrial process nor is it completely natural; it is obtained from a naturally occurring mineral and is produced specifically for cement purposes. Metakaolin is generally generated by thermal treatment, i.e. by calcination of kaolin clays within a certain temperature range. Kaolinite ($Al_2Si_2O_5(OH)_4$) is the most ordinary kaolin mineral with chemical composition: 46.54% SiO_2, 39.50% Al_2O_3, and 13.96% H_2O.

The heating method dissolves water from the mineral kaolinite ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$), the primary component of kaolin clay, and dissolves the material structure resulting in amorphous aluminosilicate ($Al_2O_3 \cdot 2SiO_2$), metakaolinite. The equation for this dihydroxylation process is shown below:

$$Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O \rightarrow Al_2O_3 \cdot 2SiO_2 + 2H_2O\uparrow$$

The heat conversion of kaolinite, which was the topic of a big amount of inquiries, has shown that heating parameters, such as temperature, heating rate and time, as well as cooling parameters (cooling rate and ambient circumstances), have a significant impact on the dehydroxylation method (Ilić et al. 2010).

The degree of dehydroxylation (D_{tg}) is the main quantitative criterion for estimating the performance kaolinite by thermal treatment which is calculated as $D_{tg} = m/m_0$, where m and m_0 are residual and maximum mass loss respectively (Rahier et al. 2000).

The growth of pozzolanic characteristics in fired clays relies primarily on the type and quantity of clay minerals in the raw resources, the calcination conditions and the fineness of the end product. The quality of the metakaolin depends, therefore, on the quality and fineness of the kaolin in which it is made from. There are three basic variables that affect the contribution that metakaolin makes to concrete strength when it partly replaces cement in concrete. “These are the filler effect, the acceleration of OPC hydration and the pozzolanic reaction of MK to CH. The filler effect is immediate, the acceleration of OPC has its major impact within the first 24 hours and the maximum effect of the pozzolanic reaction occurs between 7 and 14 days” (Wild et al. 1996).

2.5.1 Environmental impact of metakaolin

Since metakaolin is used as a cement replacement, its environmental impact is directly related to the reduction of CO_2 emission due to cement production. The use of Portland cement in concrete has a major effect on greenhouse gas where the production of each ton of Portland cement produces roughly 0.9 tons of CO_2 emissions (Spot and Wojtarowicz 2003). In addition, around 5% of worldwide man-made CO_2 emissions are generated by cement producers, which is why new regulations and laws are mandating reductions in greenhouse gas emissions. This, in turn, drives cement manufacturers and concrete contractors to seek alternatives such as less CO_2 intensive supplementary cementitious materials in order to decrease the carbon footprint of their activities (Little 2010). The replacement of cement with metakaolin will have a significant impact on the reduction of CO_2 emissions and will, therefore, pave the way for a sustainable future.

2.6 Interfacial transition zone (ITZ)

Concrete is a composite material consisting of a binding medium and aggregate particle. It can be created in several kinds and properties. It may consist of three stages: cement paste, aggregate and interfacial transition area (ITZ).

In the ITZ, in terms of morphology, composition, and density, the structure of the cement paste is different from that of the bulk paste. It has less unhydrated cement, less C-S-H, bigger calcium hydroxide crystals, higher ettringite concentration, and higher porosity (reduced density) compared to bulk-paste (Hilal 2016).

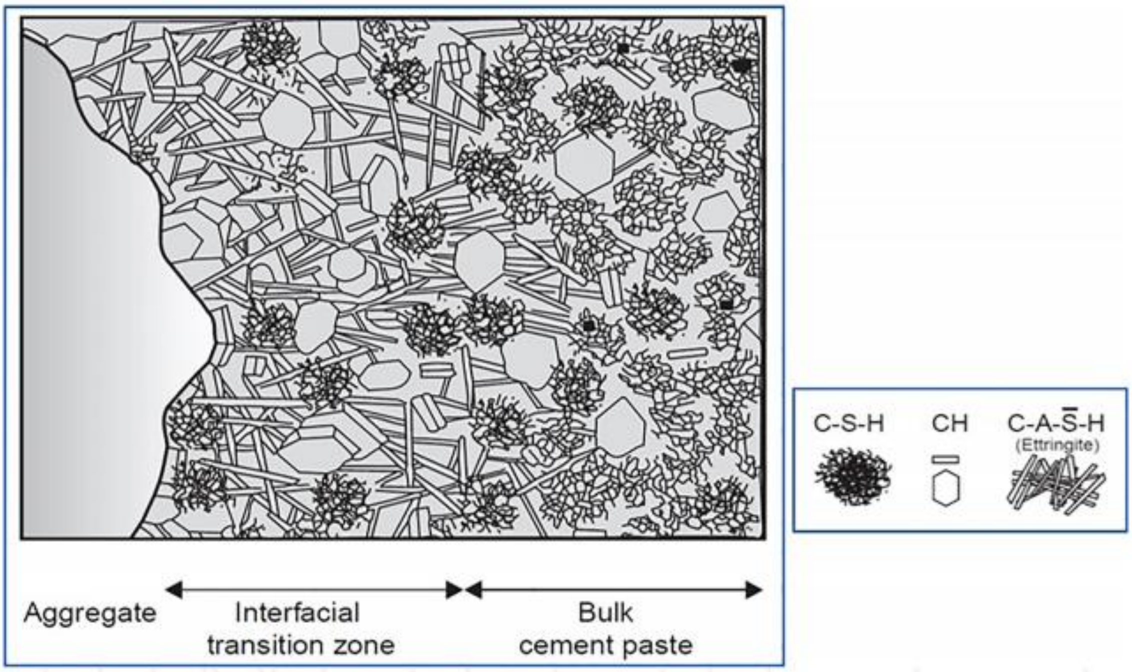

Figure 2-4: Schematic diagram of ITZ in concrete (Hilal 2016)

The ITZ, approximately 50 μm thick, plays a part in the rigidity, strength, and permeability of cemented products comprising aggregate owing to its usually reduced density and strength compared to cement paste found elsewhere. Due to its weakness, which results in the quasi-brittle nature of concrete, microcracks may occur within the ITZ when the material is subjected to loads (Hilal 2016).

“Recycled Aggregate Concrete (RAC) behavior and characteristics are strongly conditioned and influenced by the “source” waste aggregates, the adopted concrete mix design, the operative conditions to realize the mix, and the intrinsic RAC attributes (i.e. mineralogical composition, surface status, morphological and morphometrical properties)” (Bonifazi et al. 2015). The cohesive interface properties arising from RAC usage, properties highly related to the features of the Interfacial Transition Zone (ITZ), parent mortar quality and the parent mortar content of the initial concrete play an important part in the final concrete properties (Bonifazi et al. 2015).

2.7 Analysis of cementitious materials using XRD

X-ray diffraction (XRD) is one of the most prominent analytical methods used to characterize fine-grained, crystalline materials such as cement and other cementitious materials. It has a wide range of applications in phase identification, lattice parameters determination, crystal structure determination, and crystal structure refinement and quantitative phase analysis.

X-ray diffraction by a crystalline material results in a pattern composed of peaks at distinctive diffraction corners of variable intensities. The diffraction angle or position of the peaks is determined through the law of Bragg by the symmetry and size of unit cell crystalline material, whereas the intensities of the peaks are related to the nature and disposition of the atoms in the unit cell of the material (Scrivener et al. 2018).

XRD is used in qualitative phase identification. Qualitative phase analysis is done by comparing peaks in a measured XRD pattern to a database of peak patterns of predefined phases. The most commonly used general-purpose database is the powder diffraction file (PDF) published by the International Centre for Diffraction Data and using such kind of database along with chemical or categorical filters will minimize the number of possible patterns (Scrivener et al. 2018).

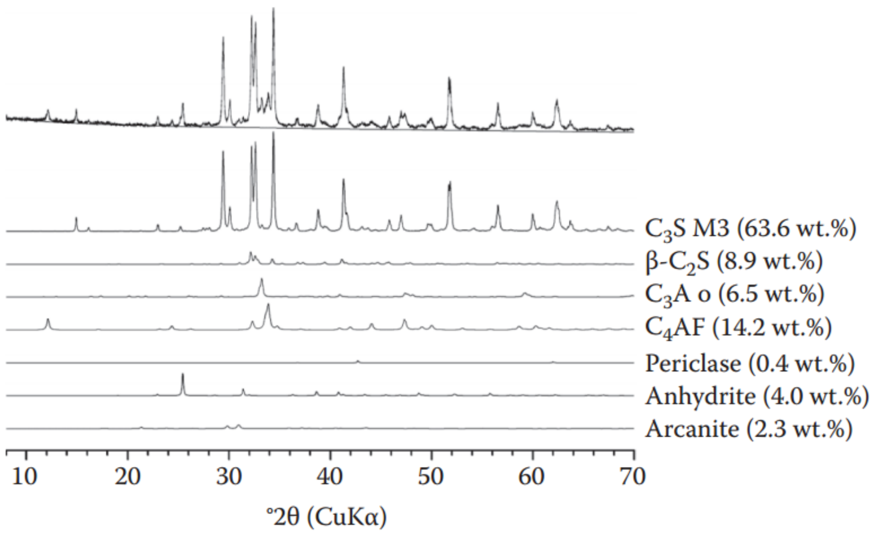

Figure 2-5: XRD of cement (CEM I 52.5N) sample (Scrivener et al. 2018)

To retrieve information from XRD output, the following methods can be used (Speakman 2011).

- Basic XRD Data Analysis using High Score Plus
- Profile Fitting and Crystallite Size Determination
- Rietveld Refinement

2.8 Abundance of kaolin in Ethiopia

2.8.1 Occurrence of kaolin

Kaolin is a white, soft, non-plastic clay consisting primarily of fine-grained platy minerals and kaolinite, usually obtained from the transformation of alkali feldspars and micas. Kaolin is mined and manufactured in many countries. In its extremely sophisticated state, it is one of the few industrial minerals of adequate importance to be commonly exported to international trade throughout the industrialized world. While in Ethiopia, according to the industrial mineral occurrence map of Ethiopia, there are six major locations where kaolin could be found in abundance. These are Bombowha, Awzet, Debretabor, Gypsite Mariam, Kerker, and kombolcha (Kadir 2007). For further information, the map is attached in APPENDIX D.

In addition, a research made by Mammo and Negassa discussed Kombolcha, Belesea and Ansho kaolin occurrences in detail (Mammo and Negassa 2011).

2.8.1.1 Kombolcha occurrences

The town of Kombolcha lies just over 18 km north of Harar on a road from Harar to Ejersa Goro. Kaolinized granite findings are sited within a 4-10 km radius of the center of Kombolcha. There are four kaolin deposits: Gende Errer, Gende Moli, and two other localities.

Figure 2-6: Kaolin occurrences south of kombolcha (Mammo and Negassa 2011)

Kombolcha Kaolin is regarded to be the weathering product of granites. The gradual rise in alkali and the decline in percent of clay size fractions with depth suggest that Kombolcha kaolin is a residual deposit with comparatively unaltered products occupying the lesser horizons. The Kombolcha Kaolin deposit is defined by a comparatively greater alkali and iron content and a low clay content. It, therefore, limits its implementation for fine ceramics without benefit. It is, however, an appropriate raw material for the manufacturing of wall and floor tiles.

2.8.1.2 Belesa occurrences

Belesa Kaolin site is located 10 km north-east of Hossaina town, on the primary highway to Addis Ababa, and the kaolin deposits are located 5 km east of Belesa, linked by dry weather highways. Kaolinite is a predominant clay chemical compound in Belesa kaolin. Quartz is the only non-clay mineral which is found in small quantity in this kaolinite. The existence and lack of quartz in the kaolinite had no effect on its the crystal lattice. Kaolin has no impurities, such as iron coating, Fe, Ti, mica, and feldspar.

2.8.1.3 Ansho occurrences

The occurrence of ansho kaolin is situated in the Hadiya zone. Ansho is situated in Gimbicho, 50 km southwest of Hossaina. The kaolin rich areas are found about 18 km south of Gimbicho and could be reached via a dry road. The deposits are interlay of sand, silt, and clay with signs of about 40 cm thick dark brown fissile oil shale and two-meter sedimentary kaolin layer that becomes sandy downwards. The kaolinite is characterized by its quartz-bearing and non-quartz varieties.

Figure 2-7: Ansho kaolin occurrence (Mammo and Negassa 2011)

2.9 The future of metakaolin production

Metakaolin is manufactured in small quantities in Africa and its only output comes from newly opened businesses in South Africa. Although metakaolin is not yet manufactured in Ethiopia, the production of kaolin is growing owing to the latest opening of big ceramic factories. As a result of the abundance of kaolin and the latest emergence of ceramic factories, metakaolin could readily be manufactured in Ethiopia as well as open a prospective market in Africa.

In contrast, metakaolin production is already booming up worldwide and the latest studies indicate that the kaolin market is set to reach 6.5 billion USD and that the metakaolin market is expected to cross 150 million USD by 2024 (Hegde 2017).

CHAPTER 3 MATERIALS AND PROPERTIES

In this chapter, types of materials used for this research and their properties are described. The materials are prepared and tested in the Construction Material Laboratory in Addis Ababa Institute of Technology. The XRD results are taken from Addis Ababa University, College of Natural and Computational Science and the complete silicate analysis results are taken from Geological Survey of Ethiopia. Additionally, the materials are checked whether they meet the requirements set in different standards.

3.1 Cement

Cement used for this experimental study is Dangote Ordinary Portland Cement (CEM I\42.5R) compatible with (CES-28 2013).

3.2 Metakaolin

The sample of metakaolin used in this experimental study is Pozzofilz Metakaolin obtained from Zigma International, a company located in India. This Metakaolin has been approved and recommended in large projects like Burj Khalifa, the tallest building in the world.

Figure 3-1: Metakaolin (Pozzofilz)

The chemical and physical properties of metakaolin and cement are presented in **Table 3-1**. The full chemical analysis of cement and metakaolin results obtained from Geological survey of Ethiopia is attached in APPENDIX C. In addition, the particle size distribution of cement and metakaolin is determined using hydrometer analysis and Sieve analysis. The hydrometer analysis works based on Stokes' law which is used to determine the terminal velocity of a falling sphere in a liquid. This method depends upon variations in the density of suspended particles contained in a 1000ml graduated cylinder. The density of suspension is measured with a hydrometer at determined time intervals; then the coarsest diameter of particles in suspension at a given time and the percentage of particles finer than the coarsest suspended particle diameter are computed (Murthy 2002). The hydrometer analysis test and procedures are done according to (ASTM D 422 2007). Sieve analysis is used for coarser particle sizes which cannot be determined using hydrometer analysis. The PSD curve of cement and metakaolin is presented in **Figure 3-2**. The hydrometer and sieve analysis results of cement and metakaolin are attached in APPENDIX A. Furthermore, the XRD analysis results of cement and metakaolin are presented in APPENDIX E.

Table 3-1: Chemical and physical properties of cement and metakaolin

Chemical Composition	Cement %	Metakaolin %
SiO_2	22.28	62.36
Al_2O_3	6.88	34.12
Fe_2O_3	3.26	1.9
CaO	60.66	0.26
MgO	1.16	0.1
Na_2O	<0.01	<0.01
K_2O	<0.01	0.28
MnO	0.04	<0.01
P_2O_5	0.11	0.09
TiO_2	0.22	0.64
H_2O	0.73	0.36
LOI	3.26	0.65
Blaine fineness (m^2/kg)	350	491
Specific gravity	3.15	2.54
Physical form	Fine powder	Fine Powder
Color	Grey	Off white to Ivory

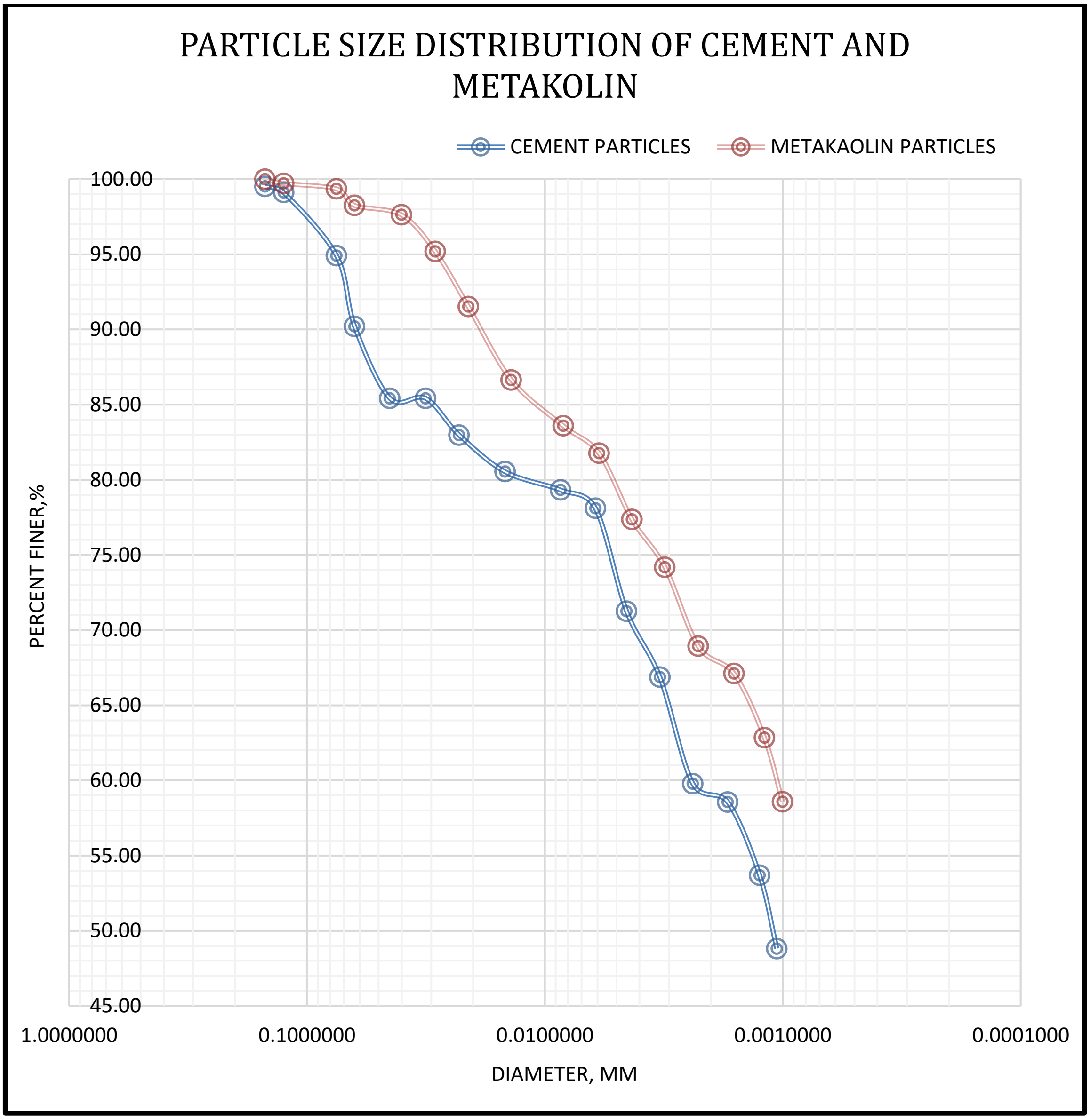

Figure 3-2: PSD of cement and metakaolin using hydrometer and sieve analysis

3.3 Aggregate

Aggregates play a major role in concrete production since they make up for around 60% to 80% of the volume of concrete. In this research, the aggregate was supplied from the same source in order to eliminate any variations arising from using aggregates from a different source. Additionally, the aggregate was washed before test to remove impurities. Then the aggregate was stored in a double-layered plastic bag in order to minimize moisture differences.

Figure 3-3: Aggregates packed in a double-layered plastic bag

3.3.1 Fine aggregate

The type of fine aggregate used for experimentation is natural sand. The fine aggregate was mainly washed to remove excess silt. Hence, the fine aggregate was washed to minimize its silt content from 3.0% (original silt content) to 1.67%. The PSD and the basic properties of fine aggregate are presented below.

Table 3-2: The basic properties of fine aggregate

No.	Test description		Test result
1.	Silt content		1.67%
2.	Moisture content		1.57%
3.	Absorption capacity		3.09%
4.	Finesse modulus		2.83%
5.	Unit weight		1574Kg/m^3
6.	Specific gravity	Bulk	2.487
		Bulk (SSD)	2.564
		Apparent	2.69

Table 3-3: The PSD of fine aggregate

Sieve size (mm)	Percent passing (%)	ASTM C 33 Standard passing range (%)
9.5	100.0	100
4.75	100.0	95-100
2.36	93.0	80-100
1.18	75.0	55-85
0.60	39.0	25-60
0.30	8.0	5-30
0.15	2.0	0-10
Pan	-	0

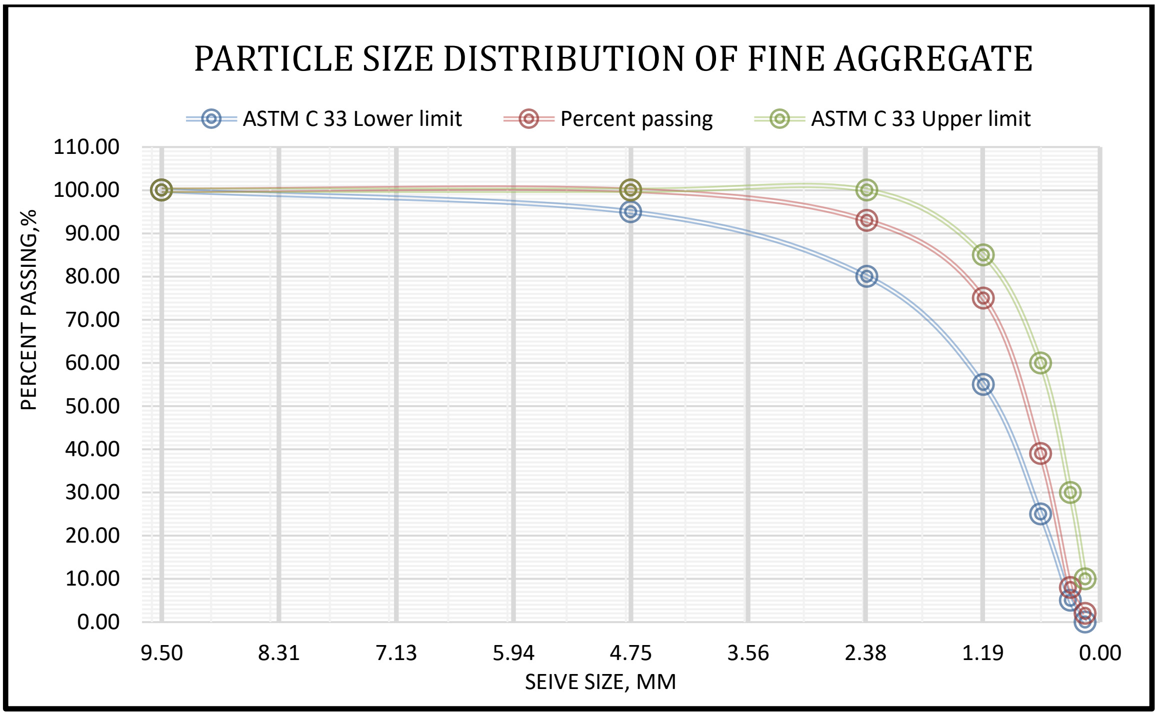

Figure 3-4: The PSD curve of fine aggregate

3.3.2 Natural coarse aggregate

The NCA has a nominal size of 25mm graded according to (ASTM C 33 2017) requirement. The size number of the aggregate is 56 which ranges between 25mm to 4.75mm sieve sizes (ASTM C 33 2017).

Figure 3-5: NCA retained on the specified sieve sizes

Figure 3-6: RCA retained on the specified sieve sizes

3.3.2.1 Well-graded natural coarse aggregate type 1

These aggregates are taken from the whole batch (stockpile) according to (ASTM D 75 1995) sampling of aggregates. The PSD and the basic properties of these aggregates are presented below.

Table 3-4: The basic properties of NCA type 1

No.	Test description		Test result
1.	Nominal size		25mm
2.	Moisture content		1.27%
3.	Absorption capacity		1.52%
4.	Unit weight		1585Kg/m^3
5.	Specific gravity	Bulk	2.61
		Bulk (SSD)	2.65
		Apparent	2.72

Table 3-5: The PSD of NCA type 1

Sieve size (mm)	Percent passing (%)	ASTM C 33 Standard passing range (%)
37.5	100.0	100
25	77.17	90-100
19	49.9	40-85
12.5	16.7	10-40
9.5	7.4	0-15
4.75	0.2	0-5
Pan	0	0

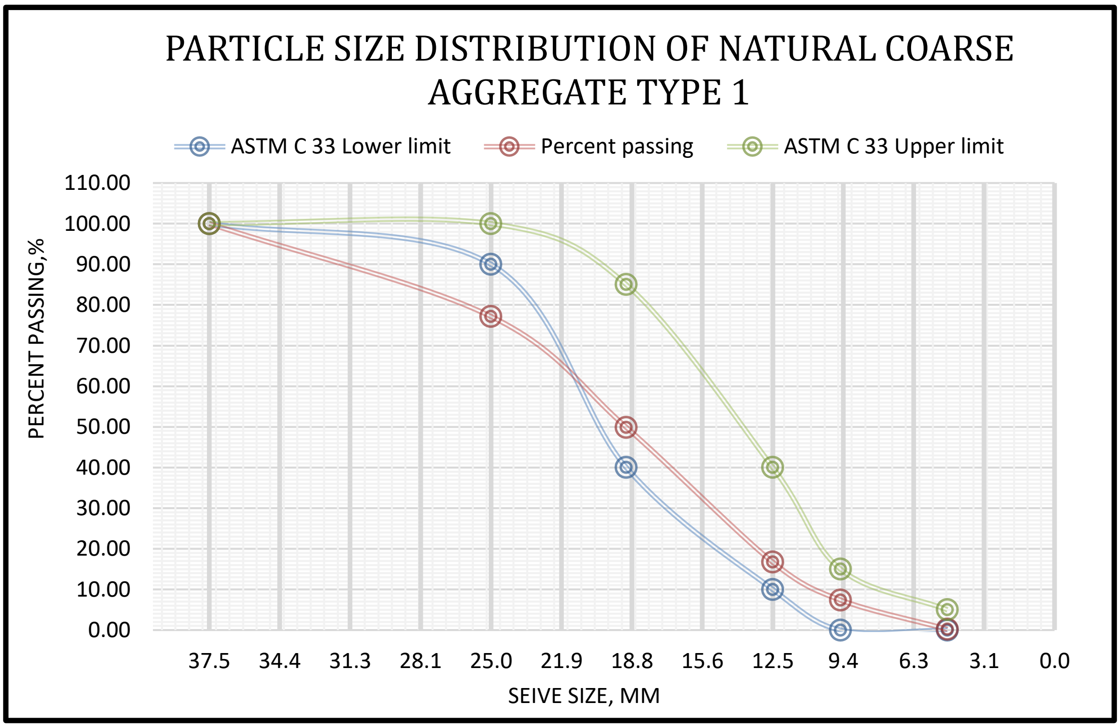

Figure 3-7: The PSD curve of NCA type 1

3.3.2.2 Well-graded natural coarse aggregate type 2

A principal assumption in every research is that we can draw inference about the whole batch based on the evaluation of a representative sample. However, in drawing such an inference there is going to be some error (not significant) due to material property differences. Therefore, to further minimize this error, these aggregates have a fixed gradation, and the weighted moisture content and absorption capacity are taken after the moisture contents and absorption capacities are done individually for every sieve size. The PSD, the weighted absorption capacity, the weighted moisture content and the basic properties of these aggregates are presented below.

Table 3-6: The PSD of NCA type 2 and RCA

Sieve size (mm)	Percent passing (%)	ASTM C 33 Standard passing range (%)
37.5	100.0	100
25	95.0	90-100
19	70.0	40-85
12.5	25.0	10-40
9.5	10.0	0-15
4.75	0	0-5
Pan	0	0

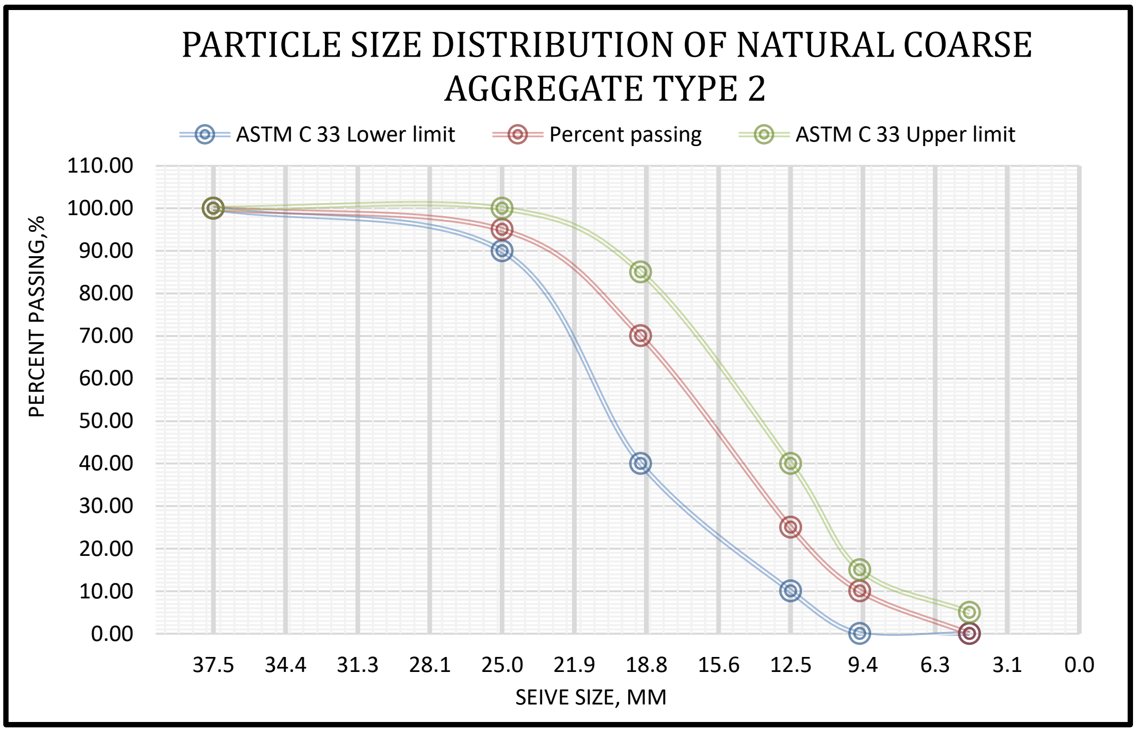

Figure 3-8: The PSD curve of NCA type 2 and RCA

Table 3-7: The weighted absorption capacity of NCA type 2

Sieve size (mm)	Percent retained (%)	Absorption capacity (%)	Weighted absorption capacity (%)
37.5	-	-	
25	5.0	1.215	0.061
19	25.0	1.215	0.304
12.5	45.0	2.041	0.914
9.5	15.0	2.669	0.400
4.75	10.0	2.669	0.267
Total	100.0		1.95

Table 3-8: The weighted moisture content of NCA type 2

Sieve size (mm)	Percent retained (%)	Moisture content (%)	Weighted moisture content (%)
37.5	-		
25	5.0	1.874	0.094
19	25.0	1.874	0.469
12.5	45.0	2.690	1.211
9.5	15.0	2.690	0.404
4.75	10.0	3.799	0.380
Total	100.0		2.56

Table 3-9: The basic properties of NCA type 2

No.	Test description		Test result
1.	Nominal size		25mm
2.	Moisture content		2.56%
3.	Absorption capacity		1.95%
4.	Unit weight		1627Kg/m^3
5.	Specific gravity	Bulk	2.67
		Bulk (SSD)	2.72
		Apparent	2.81

3.3.3 Recycled coarse aggregate

RCA properties highly depend on the parent material strength where these aggregates are taken from. In order to control strength variations from parent material, concrete cubes were cast firsthand then, these cubes were crushed using two separate procedures. First, these cubes were crushed using a compression machine then, the recycled aggregates are further crushed using different kinds of hammers.

Figure 3-9: Crushing concrete cubes using a compression machine and its result

Figure 3-10: Crushing of recycled aggregates using different kinds of hammers

These recycled aggregates have the same grading as NCA type 2 and contain varying amounts of paste percentages which are difficult to quantify. Thus, recycled coarse aggregates for different sieve sizes with varying paste percentages are presented in **Figure 3-11**. Additionally, the moisture content and absorption capacity are weighted similar to NCA type 2. The weighted absorption capacity, the weighted moisture content and the basic properties of these aggregates are presented below.

Figure 3-11: RCA with increasing paste percentage

Table 3-10: The weighted absorption capacity of RCA

Sieve size (mm)	Percent retained (%)	Absorption capacity (%)	Weighted absorption capacity (%)
37.5	-	-	
25	5.0	10.86	0.543
19	25.0	8.70	2.174
12.5	45.0	7.30	3.283
9.5	15.0	9.17	1.376
4.75	10.0	10.86	1.086
Total	100.0		8.460

Table 3-11: The weighted moisture content of RCA for experiment 2

Sieve size (mm)	Percent retained (%)	Moisture content (%)	Weighted moisture content (%)
37.5	-		
25	5.0	8.249	0.41
19	25.0	7.089	1.77
12.5	45.0	5.955	2.68
9.5	15.0	8.483	1.27
4.75	10.0	10.424	1.04
Total	100.0		7.18

Table 3-12: The weighted moisture content of RCA for experiment 4

Sieve size (mm)	Percent retained (%)	Moisture content (%)	Weighted moisture content (%)
37.5	-		
25	5.0	9.440	0.47
19	25.0	8.268	2.07
12.5	45.0	7.121	3.20
9.5	15.0	9.678	1.45
4.75	10.0	11.640	1.16
Total	100.0		8.36

Table 3-13: The basic properties of RCA

No.	Test description		Test result	
1.	Nominal size		25mm	
2.	Moisture content		7.18%	8.36%
3.	Absorption capacity		8.46%	
4.	Unit weight		1430Kg/m^3	
5.	Specific gravity	Bulk	2.21	
		Bulk (SSD)	2.39	
		Apparent	2.71	

3.4 Water

The water used for this research is an underground water found in the Construction Material Laboratory in Addis Ababa Institute of Technology.

3.5 Kaolin

There are two kaolin samples collected for testing from Di Yuan Ceramic Factory which is found in the Eastern Industry Zone located in Dukem, Ethiopia. The first sample has its origin around Mojo and due to its bright color, it is called white kaolin while the second sample which originated around Arsi (Dera) is rather dark in color and known as red kaolin. The chemical and physical properties of both kaolin samples are presented in **Table 3-14**. The full chemical analysis of white kaolin and red kaolin results obtained from Geological survey of Ethiopia is attached in the APPENDIX C.

Figure 3-12: Red kaolin

Figure 3-13: White kaolin

Table 3-14: Chemical and physical properties of white and red kaolin

Chemical Composition	White kaolin %	Red kaolin %
SiO_2	60.68	57.82
Al_2O_3	23.10	20.28
Fe_2O_3	0.66	1.46
CaO	1.34	<0.01
MgO	<0.01	<0.01
Na_2O	<0.01	<0.01
K_2O	0.28	0.96
MnO	<0.01	<0.01
P_2O_5	0.07	0.05
TiO_2	0.51	0.39
H_2O	4.55	5.86
LOI	10.22	11.87
Blaine fineness (m^2/kg)	400	405
Specific gravity	2.51	2.51
Kaolin content	74	63
Physical form	Fine powder	Powder
Color	Off white to bright orange	Ivory to Dark orange

CHAPTER 4 EXPERIMENTAL PROGRAM

The main aim of this research is to perform an experimental study to investigate the effect of metakaolin on the compressive strength of recycled coarse aggregate concrete. To carry out this study, all experimental programs are done at the Construction Material Laboratory in Addis Ababa Institute of technology.

Five different experimental programs were conducted. These include the testing of NCA concrete for the production of recycled aggregate, testing of RCA concrete, testing of NCA concrete with metakaolin, testing of RCA concrete with metakaolin and finally determining the optimum heating temperature of kaolin clay to produce metakaolin.

4.1 Test types and specimens

Much of this research work is related to concrete testing. Fresh and hardened concrete tests were done. Compressive machines were used to test hardened concrete properties while slump tests were done to see the properties of fresh concrete. All compression tests are done on $15cm \times 15cm \times 15cm$ concrete cubes. A total of 293 cubes: 110 cubes for NCA concrete testing, 30 cubes for RCA concrete testing, 45 cubes for NCA concrete with metakaolin testing and 108 cubes for RCA concrete with metakaolin testing. Moreover, there is a separate test to produce metakaolin from kaolin using a muffle furnace. A total of 33 kaolin powder: 15 samples for white kaolin while 18 samples of red kaolin powder were heated at different temperatures and durations for the fabrication of metakaolin.

4.2 Mix design

Mix design is the process of finding the right proportions of coarse aggregate, fine aggregate, water and cement for a pre-required concrete strength. Although there are lots of papers and standards for determining mix design, the perfect mix design can only be mastered through repetition of trial mixes and following the correct path which results in a continuous error reduction, when compared with the anticipated result. The main cause for iteration could be material property differences across different places (The origins of the materials used on those papers and standards) but this does not mean the papers and the different standards are wrong because they are published with thousands of data to justify the cause.

There were two separate mix design procedures used for this experiment to achieve the required characteristic compressive strength of 25MPa which translates to a specified average compressive strength of 33MPa according to ACI 214R-11. The first mix design procedure used was the (ACI 211 2002) which gave specified compressive strengths much larger than the expected 33MPa. The second mix design procedure used was the binary method. The basis for the binary method of mix design is the smallest possible volumetric ratio of aggregates (volume of fine aggregate to volume of total aggregates) should be taken for proportioning. Apart from this, the procedure is rather similar to (ACI 211 2002). Accordingly, the mix proportions obtained from the binary method are used as the main mix design in this research.

The mix designs, as well as recycled coarse aggregate and metakaolin replacements, were done volumetrically. There were two major mix designs. The first mix design is used only for the production of NCA concrete which was made for the production of RCA, while the second mix design was used for the remaining concrete tests. The rationale behind the two mix designs difference is PSD, in other words, different PSD means different specific gravity. The mix designs are presented below.

Table 4-1: The major mix proportions

	Mix design 1		Mix design 2	
Composition	Volume (l)	Mass (Kg)	Volume (l)	Mass (Kg)
Water	190.00	190.00	190.00	190.00
Cement	104.17	328.15	104.17	328.15
Fine aggregate	308.62	791.30	298.64	765.71
Coarse aggregate	377.21	999.60	387.19	1053.15
Air	20.00		20.00	
Total	1000.00		1000.00	

4.3 Experiments

All concretes were mixed in the laboratory using the mixer shown in **Figure 4-2**. The coarse aggregate was added to the mixer first followed by cement and fine aggregate, then it was dry blended for about one minute. Two-thirds of water was added and the mixing was continued for an additional one minute. The remaining water is then added, and the total mixing time was three minutes. Slump tests of fresh concrete were done immediately after mixing to check workability and consistency. Finally, the concrete was placed in $15cm \times 15cm \times 15cm$ cube molds and vibrated for 30 seconds. After 24hrs the cubes were taken out of their molds and put in a water tank for curing as shown in **Figure 4-3** until the test date.

Figure 4-2: Mixer

Figure 4-1: Concrete placed in 15cm cube molds

Figure 4-3: Curing of concrete cube samples

4.3.1 Experiment 1

This experiment is done to produce recycled coarse aggregates from strength-controlled parent concretes. 11 mixes were done with the same mix proportion (mix design 1), each producing 10 cubes, for a total of 110 cubes. These cubes were crushed after determination of the 28^{th}-day compressive strength.

4.3.2 Experiment 2

This experiment is done to compare the compressive strengths of different percentages of RCA concretes with NCA concrete. The RCA was used to replace the NCA by 25, 50, 75 and 100%, respectively. The RCA was pre-soaked before casting to a level where the moisture content, at the time of casting, is less than 1.28% of the absorption capacity. Three cube samples were used to check both the 7-day and 28-day strengths.

4.3.3 Experiment 3

This part of the experiment is done to find the optimum metakaolin percentage, used as a cement replacement, and also to compare the compressive strengths of NCA concrete incorporating different percentages of metakaolin, as cement replacement, with that of NCA concrete. Cement is replaced with metakaolin by 6, 12, 18 and 24%, respectively. Three cube samples were used to check the 7-day, 28-day, and 56-day strengths.

Additionally, to check the workability of NCA concrete with metakaolin, especially for higher replacement levels (18 and 24%), the consistency of both NCA concrete with and without metakaolin was done in accordance with (ASTM C 187 2004). Consistency is performed to determine the water content needed to produce a cement paste of standard consistency. However, for this experiment, consistency is done to see the water requirement difference between NCA concrete with and without metakaolin. Accordingly, if the water requirement difference is high, admixture must be introduced to enhance the low workability of the mixtures.

4.3.4 Experiment 4

This experiment is done to compare the compressive strengths of RCA concretes with metakaolin, to that of RCA concretes and NCA concretes. For every RCA replacement additionally, cement is replaced with metakaolin by 6, 12 and 18%, respectively. For example, for 25% RCA replacement cement is also replaced with metakaolin by 6, 12 and 18%, respectively. The same goes for 50, 75 and 100% RCA replacements. The notations of concrete mixtures and the mixture proportions of the concrete series are presented in **Table 4-2** and **Table 4-3** respectively for ease of reference.

Table 4-2: The notation of mixtures, expansion of notation, mix design and series

Series	Mix design	Notation	Expansion of notation
N	1	1N	Concrete containing NCA
	2	2N	Concrete containing NCA
		2NM6	Concrete containing NCA and 6% Metakaolin
		2NM12	Concrete containing NCA and 12% Metakaolin
		2NM18	Concrete containing NCA and 18% Metakaolin
		2NM24	Concrete containing NCA and 24% Metakaolin
R25	2	2R25	Concrete containing 25% RCA
		2R25M6	Concrete containing 25% RCA and 6% Metakaolin
		2R25M12	Concrete containing 25% RCA and 12% Metakaolin
		2R25M18	Concrete containing 25% RCA and 18% Metakaolin
R50	2	2R50	Concrete containing 50% RCA
		2R50M6	Concrete containing 50% RCA and 6% Metakaolin
		2R50M12	Concrete containing 50% RCA and 12% Metakaolin
		2R50M18	Concrete containing 50% RCA and 18% Metakaolin
R75	2	2R75	Concrete containing 75% RCA
		2R75M6	Concrete containing 75% RCA and 6% Metakaolin
		2R75M12	Concrete containing 75% RCA and 12% Metakaolin
		2R75M18	Concrete containing 75% RCA and 18% Metakaolin
R100	2	2R100	Concrete containing 100% RCA
		2R100M6	Concrete containing 100% RCA and 6% Metakaolin
		2R100M12	Concrete containing 100% RCA and 12% Metakaolin
		2R100M18	Concrete containing 100% RCA and 18% Metakaolin

Table 4-3: Concrete mixture proportions

Series	Mix design	Notation	Constitution (liter)						
			Composition of binder		Water	Fine aggregate	NCA	RCA	Air
			Cement	Metakaolin					
N	1	1N	104.17	0	190	308.62	377.21	0	20
	2	2N	104.17	0	190	298.64	387.19	0	20
		2NM6	97.92	6.25	190	298.64	387.19	0	20
		2NM12	91.67	12.5	190	298.64	387.19	0	20
		2NM18	85.42	18.75	190	298.64	387.19	0	20
		2NM24	79.17	25.00	190	298.64	387.19	0	20
R25	2	2R25	104.17	0	190	298.64	290.39	96.80	20
		2R25M6	97.92	6.25	190	298.64	290.39	96.80	20
		2R25M12	91.67	12.5	190	298.64	290.39	96.80	20
		2R25M18	85.42	18.75	190	298.64	290.39	96.80	20
R50	2	2R50	104.17	0	190	298.64	193.59	193.59	20
		2R50M6	97.92	6.25	190	298.64	193.59	193.59	20
		2R50M12	91.67	12.5	190	298.64	193.59	193.59	20
		2R50M18	85.42	18.75	190	298.64	193.59	193.59	20
R75	2	2R75	104.17	0	190	298.64	96.80	290.39	20
		2R75M6	97.92	6.25	190	298.64	96.80	290.39	20
		2R75M12	91.67	12.5	190	298.64	96.80	290.39	20
		2R75M18	85.42	18.75	190	298.64	96.80	290.39	20
R100	2	2R100	104.17	0	190	298.64	0	387.19	20
		2R100M6	97.92	6.25	190	298.64	0	387.19	20
		2R100M12	91.67	12.5	190	298.64	0	387.19	20
		2R100M18	85.42	18.75	190	298.64	0	387.19	20

4.3.5 Experiment 5

This experiment is done to produce metakaolin. Metakaolin is produced by heating kaolin clay. This heating process drives off water from kaolin clay which results mass loss. The material formed after maximum mass loss is metakaolin while, further heating after this point will result in dead burnt, nonreactive refractory called mullite (Said-mansour et al. 2011). Kaolin clay finer than 32μm sieve were taken for this experimentation. 25g samples of kaolin clay were heated at different temperatures of 600, 650, 700, 750, 800 and 850 ^{0}C in a muffle furnace for different durations of 45, 90 and 135 min. The samples were then cooled at room temperature to avoid the crystallization of amorphous metakaolin. Finally, the mass of the samples after heating was measured to calculate the mass loss. Additionally, the degree of dehydroxylation was calculated to counter check metakaolin formation.

CHAPTER 5 RESULTS AND DISCUSSION

In this section, the test results of the experimental programs performed are presented and analyzed.

5.1 Test on fresh concrete

5.1.1 Slump test

Slump test is carried out to check the consistency and workability of freshly made concretes. Slump tests conducted for all experiments are presented below.

5.1.1.1 Experiment 1

In this experiment, slump tests were done to check the consistency of mixes from batch to batch. **Table 5-1** shows the results of the slump test.

Table 5-1: Slump test results for experiment 1

Mix (1N)	1	2	3	4	5	6	7	8	9	10	11
Slump (mm)	50	50	50	45	65	50	120	70	70	100	60

Result inconsistencies may arise from variations in characteristics and proportion of ingredients, variations in mixing, variations in placing and variations in mixing temperature and humidity (ACI Committee 2011). However, due to the same mixing time and speed, variations arising from mixing could be neglected and due to the usage of the same operator across all mixes variations in placing could also be neglected. Therefore, the inconsistencies occurred as a result of variations in characteristics of ingredients (Coarse aggregates, fine aggregates, and cement) and variations in mixing temperature and humidity.

5.1.1.2 Experiment 2

In this experiment, slump tests were done to check workability as different percentages of RCA were used. The results of the slump test are presented in **Table 5-2.**

Table 5-2: Slump test results for experiment 2

Mix Notation	2N	2R25	2R50	2R75	2R100
Slump (mm)	25	35	125	155	175

According to the results, slump increased with an increase in replacement percentage of RCA. This can be attributed to additional water for moisture adjustment. Although RCA was pre-soaked up to 7.18% moisture content, additional water was needed to reach its SSD moisture content of 8.46%. Furthermore, this additional adjustment water increased as the replacement percentage of RCA increased.

The main point noted in this experiment is:

- The workability of RCA concrete rises with an increase in RCA replacement due to initial free water in the fresh mix added to mitigate deviations of RCA from SSD condition. This point is also mentioned in previous research work (Kou et al. 2011).

5.1.1.3 Experiment 3

Consistency test was done before slump test to predict the workability of metakaolin concrete. Accordingly, the water to cement ratio of NCA concrete with and without metakaolin was 0.3 and 0.26 respectively. This shows for the same amount of water NCA concrete with metakaolin has low workability when compared to NCA concrete without metakaolin. Additionally, there is no need for using admixture because consistency results are close.

Slump results for this experiment are presented in **Table 5-3.** The results indicate as the replacement percentage of metakaolin increased the workability of the mixes decreased. This can be supported by consistency test and the points discussed above.

Table 5-3: Slump test results for experiment 3

Mix notations	2N	2NM6	2NM12	2NM18	2NM24
Slump (mm)	75	60	50	50	45

The main point noted in this experiment is:

- The workability of concrete containing metakaolin as cement replacement declines with an increase in metakaolin content due to higher specific surface area of metakaolin resulting in portions of water being absorbed on the surface. This decrease in workability is also seen in previous literature (Dinakar et al. 2013).

5.1.1.4 Experiment 4

In this experiment, slump tests were done to check both the workability and consistency of the mixes since different percentages of RCA and metakaolin were used. In order to minimize high workability conditions due to increased percent replacements of RCA, as presented in experiment 2, the RCA was pre-soaked to a moisture content of 8.36%.

Table 5-4: Slump test results for experiment 4

Mix notations	2R25M6	2R25M12	2R25M18	2R50M6	2R50M12	2R50M18	2R75M6	2R75M12	2R75M18	2R100M6	2R100M12	2R100M18
Slump (mm)	70	65	50	55	45	40	35	55	40	45	40	40

The results in **Table 5-4** show two main findings. The first is for higher percent replacements of metakaolin the workability becomes low. The second is high workability conditions of RCA concrete can be controlled by pre-soaking RCA.

The main point noted in this experiment is:

- To control the high workability condition of RCA concrete, the RCA must be pre-soaked to a level where the difference between moisture content and absorption capacity is small rather than the accustomed 24hr pre-soaking in water before casting. This point is also found in previous literature (Rao et al. 2019).

5.2 Test on hardened concrete

5.2.1 Compressive strength

The average compressive strength test results are presented in this section. Furthermore, the detailed results of the compressive strength test are provided in APPENDIX B.

Figure 5-1: Compressive strength testing machine

5.2.1.1 Experiment 1

The 28th-day average compressive strength test results obtained from ten representative samples are given in **Table 5-5**.

Table 5-5: Average compressive strength results of experiment 1

Mix (1N)	1	2	3	4	5	6	7	8	9	10	11
Compressive strength (MPa)	35.72	34.98	33.12	35.51	34.35	33.52	32.98	33.96	32.59	32.95	33.18

The average compressive strength of the above 11 results which is 33.88MPa, was taken as the strength of the parent concrete in which RCA is extracted. Additionally, this result conforms to the expected strength result from the trials which was 33MPa.

5.2.1.2 Experiment 2

The 7th and 28th-day average compressive strength test results obtained from three representative samples are given in **Table 5-6**.

Table 5-6: Average compressive strength results of experiment 2

Mix notations	Average compressive strength (MPa)	
	7th day	28th day
2N	21.01	32.04
2R25	19.29	31.13
2R50	18.08	28.29
2R75	16.07	26.72
2R100	15.66	25.02

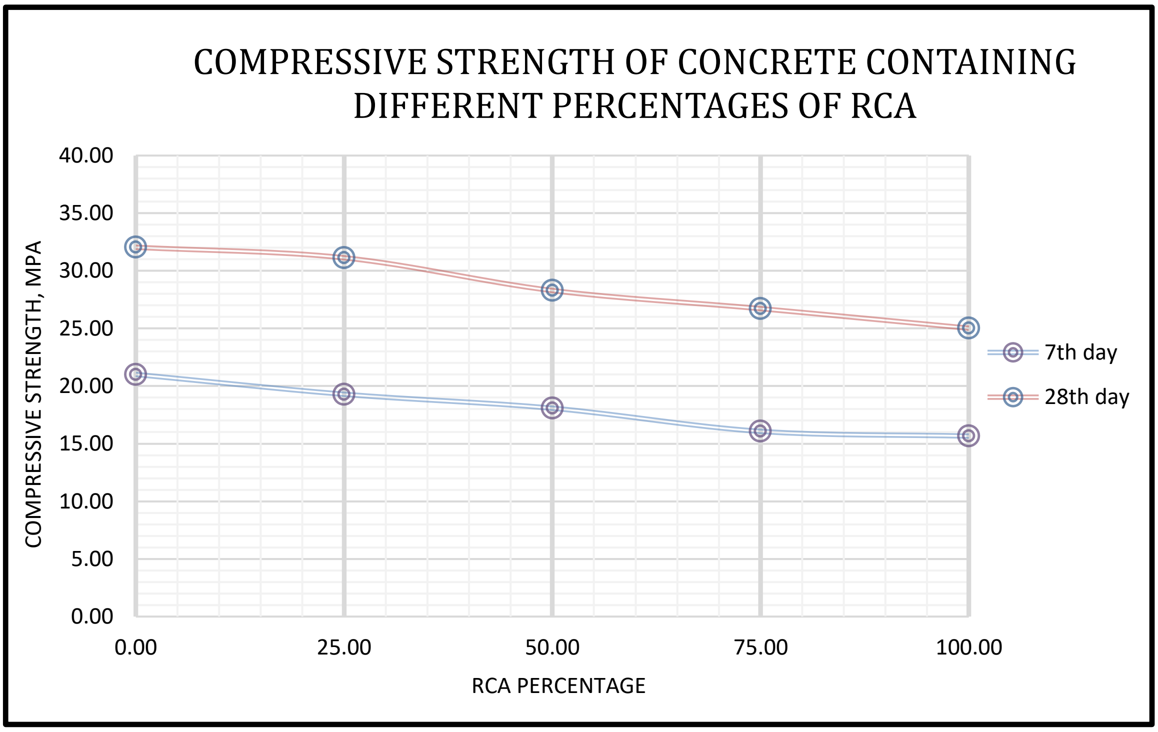

Figure 5-2: Average compressive strength of concrete containing RCA

It can be seen from the results that the average compressive strength decreased with increasing percentage of RCA. The 7th day average compressive strength of concrete containing 25%, 50%, 75% and 100% RCA declined by 8.2%, 13.9%, 23.5% and 25.5%, respectively, while the 28th day declined by 2.8%, 11.7%, 16.6% and 21.9% respectively.

The main point noted in this experiment is:

- Incorporation of RCA decreases the compressive strength of concrete as much as 21.9% for 100% replacement of NCA and 26.2% when compared with the compressive strength of parent concrete respectively. This is due to the existence of old mortar on the RCA surface. This old mortar forms a weak link in RCA which contains many cracks and pores. Consequently, these cracks and pores increase water consumption, leading to a shortage of water at the recycled aggregate concrete ITZ (Xiao et al. 2012).

5.2.1.3 Experiment 3

The 7th, 28th and 56th-day average compressive strength test results obtained from three representative samples are given in **Table 5-7**.

Table 5-7: Average compressive strength results of experiment 3

Mix notations	Average compressive strength (MPa)		
	7th day	28th day	56th day
2N	20.87	31.69	37.99
2NM6	21.11	31.86	38.05
2NM12	24.19	33.55	38.30
2NM18	21.65	30.78	37.33
2NM24	20.58	29.83	37.19

It can be seen from the results that the average compressive strength is maximum for concrete containing 12% metakaolin as cement replacement. The 7th-day average compressive strength is enhanced by 1.1%, 15.9% and 3.7% for 6%, 12%, and 18% metakaolin replacements respectively, but for 24% metakaolin replacement the compressive strength declined by 1.4%. In addition, the 28th-day average compressive strength rose by 0.5% and 5.9% for 6% and 12% metakaolin replacements respectively, and the 56th-day average compressive strengths rose by 0.2% and 0.8% for 6% and 12% metakaolin replacements respectively. However, the 28th and 56th-day average compressive strength declined for 18% and 24% metakaolin replacements.

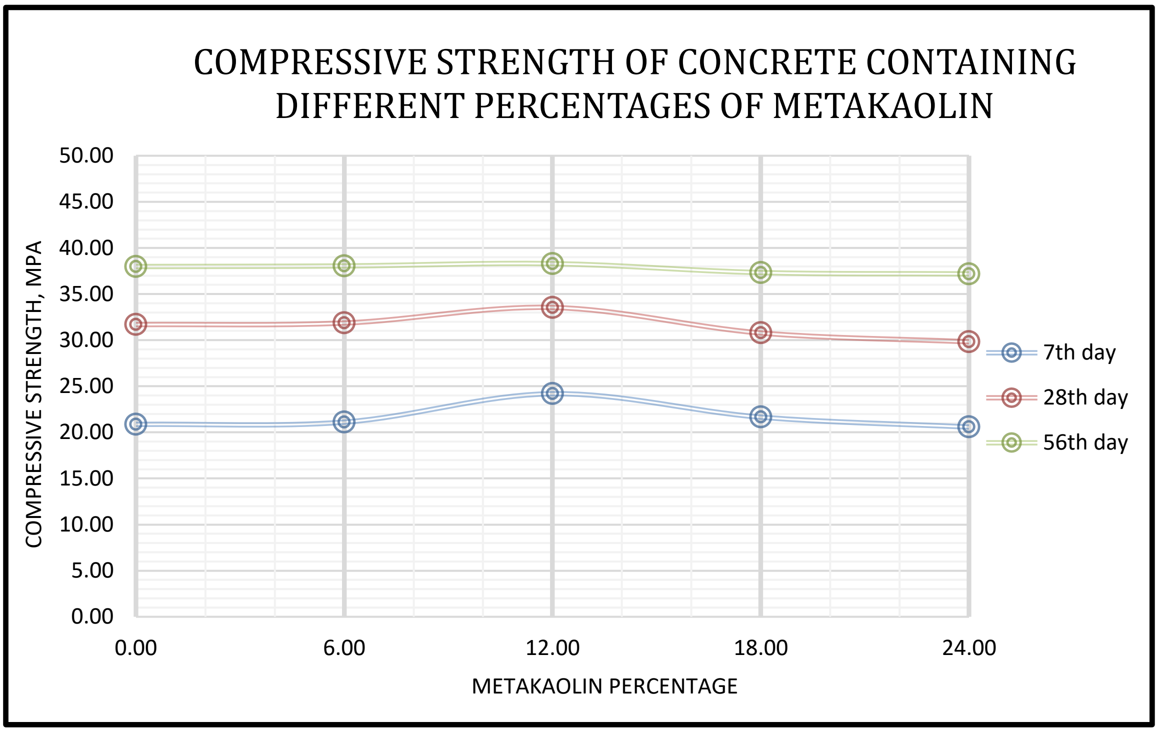

Figure 5-3: Average compressive strength of concrete containing Metakaolin

The main points noted in this experiment are:

- The compressive strength of NCA concrete is enhanced up to 12% metakaolin as cement replacement regardless of the curing ages. Therefore 12% metakaolin is the optimum replacement for NCA concrete.
- The compressive strength of NCA concrete is enhanced by the incorporation of metakaolin. This is due to three basic factors which are filler effect, acceleration of OPC hydration and pozzolanic reaction. The pores between larger OPC particles are filled with smaller particles of metakaolin which leads to a better particle size packing at the ITZ of aggregate paste interface as well as the entire microstructure of concrete. Additionally, the pozzolanic reaction of metakaolin which occurs between calcium hydroxide of cement and, silicate and aluminate generated from metakaolin produces more CSH gel that contributes to strength development (Said-mansour et al. 2011).
- The compressive strength enhancement rate of metakaolin for NCA concrete is more during early ages (7 and 28 days) than later age (56 days). This is due to the completion of pozzolanic reaction which prevents further reactions due to the reduction of calcium hydroxide or refinement of pores which retards this process.

Furthermore, XRD analysis results of NCA concrete and NCA with optimum metakaolin (2NM12) are presented in APPENDIX E.

5.2.1.4 Experiment 4

The 7th and 28th-day average compressive strength test results obtained from three representative samples are separately presented for 25%, 50%, 75% and 100% RCA replacements with different metakaolin content as cement replacement respectively, while the 56th-day average compressive strength test result is presented showing all RCA and metakaolin replacements. Furthermore, the average compressive strength results of concrete with NCA is compared with concretes with different percentages of RCA and metakaolin.

Table 5-8: Average compressive strength results of experiment 4 for 25% RCA

Mix notations	Average compressive strength (MPa)	
	7th day	28th day
2R25	19.29	31.13
2R25M6	22.06	31.78
2R25M12	21.26	31.31
2R25M18	19.48	30.36

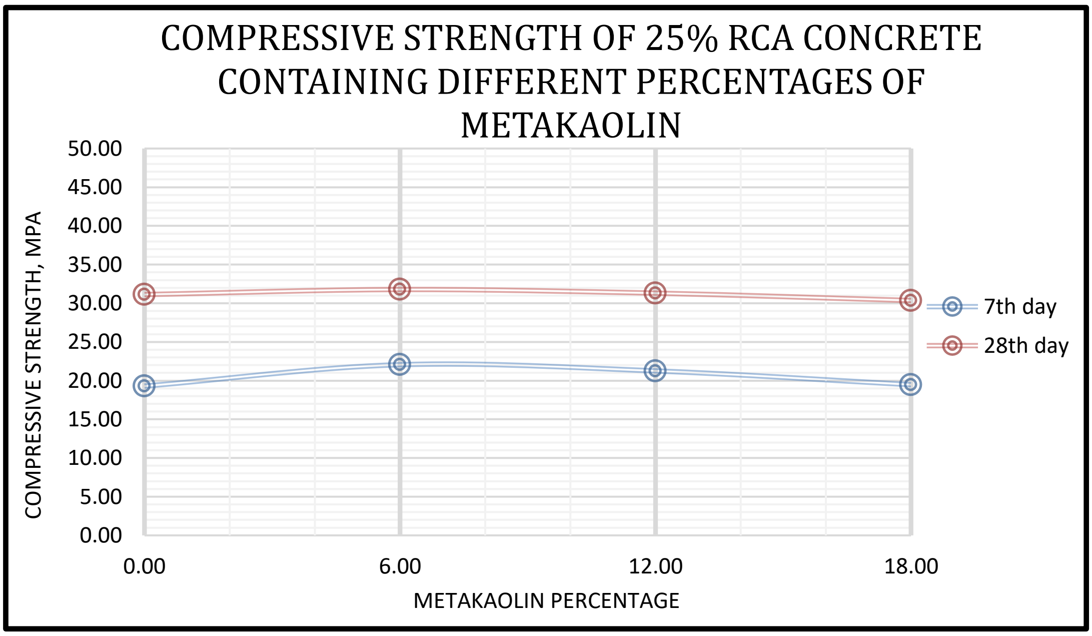

Figure 5-4: Average compressive strength of 25% RCA concrete with Metakaolin

Table 5-9: Average compressive strength results of experiment 4 for 50% RCA

Mix notations	Average compressive strength (MPa)	
	7th day	28th day
2R50	18.08	28.29
2R50M6	21.78	30.90
2R50M12	22.16	31.93
2R50M18	21.14	31.15

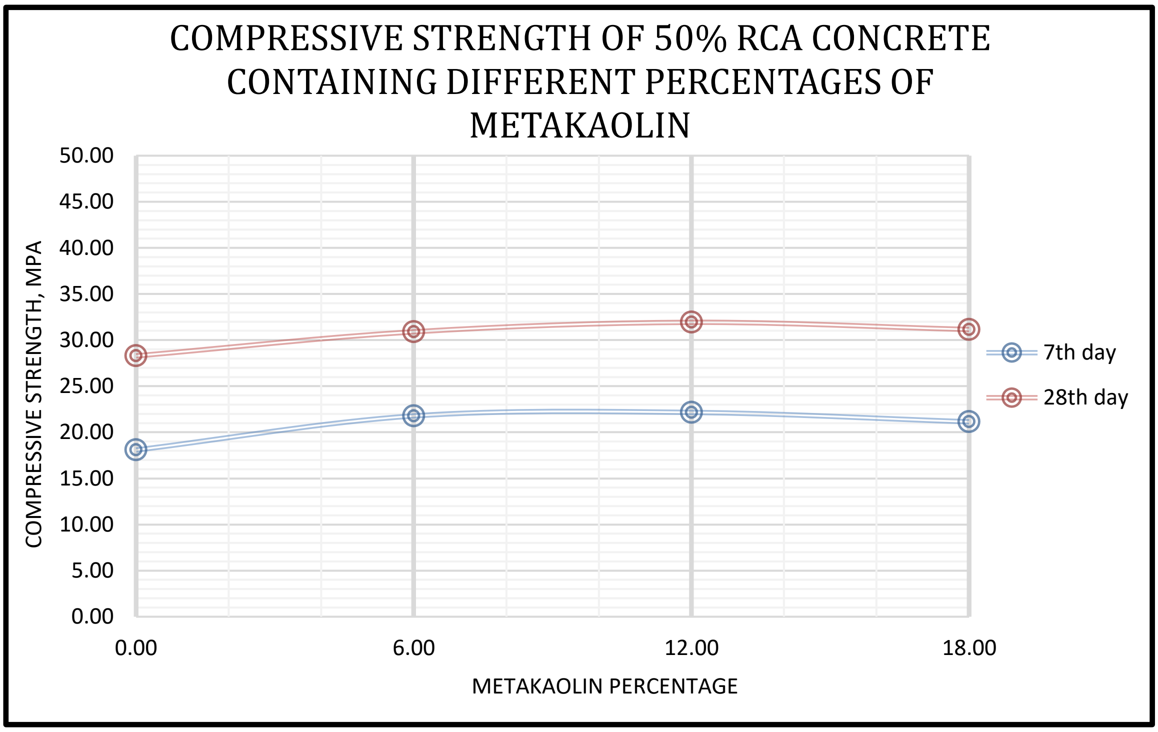

Figure 5-5: Average compressive strength of 50% RCA concrete with Metakaolin

Table 5-10: Average compressive strength results of experiment 4 for 75% RCA

Mix notations	Average compressive strength (MPa)	
	7th day	28th day
2R75	16.07	26.72
2R75M6	22.49	32.71
2R75M12	20.87	31.11
2R75M18	18.73	30.75

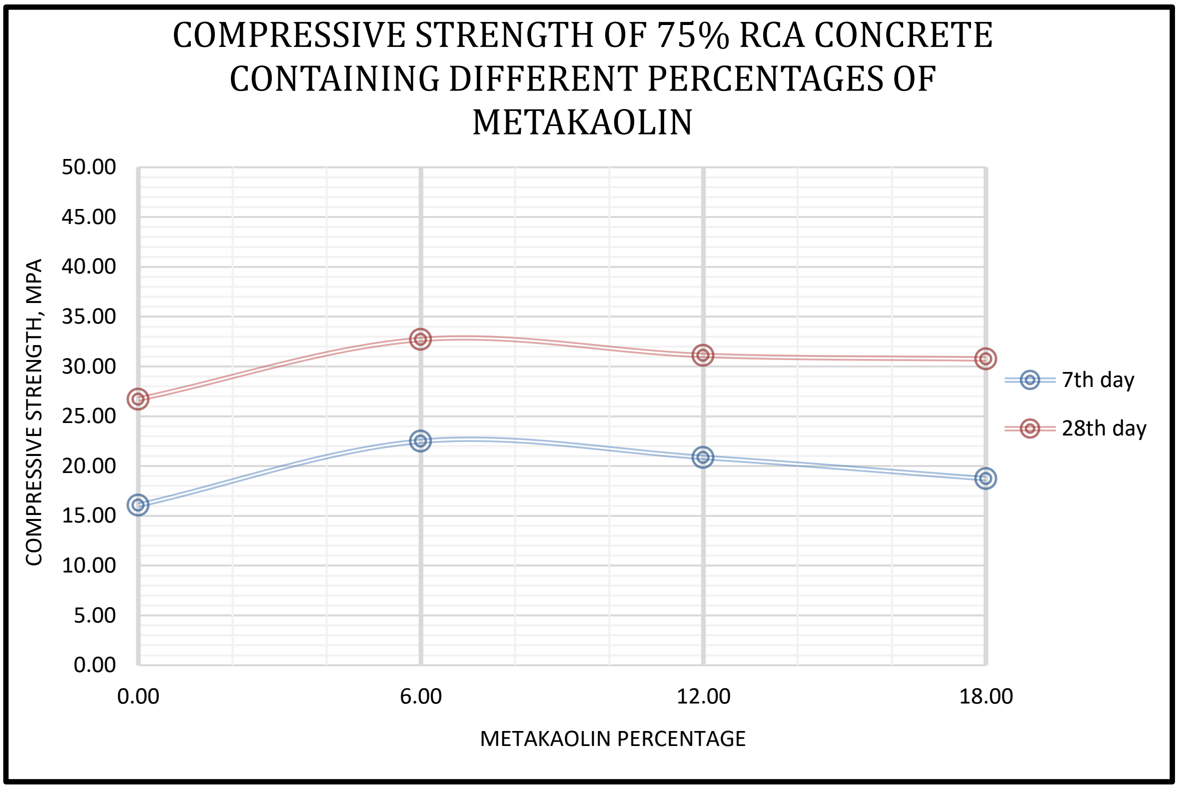

Figure 5-6: Average compressive strength of 75% RCA concrete with Metakaolin

Table 5-11: Average compressive strength results of experiment 4 for 100% RCA

Mix notations	Average compressive strength (MPa)	
	7^{th} day	28^{th} day
2R100	15.66	25.02
2R100M6	22.94	34.48
2R100M12	21.42	31.84
2R100M18	20.95	31.33

It can be seen from the results that the average compressive strength of RCA concrete is increased with the incorporation of metakaolin as a cement replacement for all cases. Additionally, the maximum average compressive strengths are attained for 6% metakaolin replacements for concretes with 25%, 75% and 100% RCA replacements. However, the maximum average compressive strength for 50% RCA replacement is achieved with 12% metakaolin replacement.

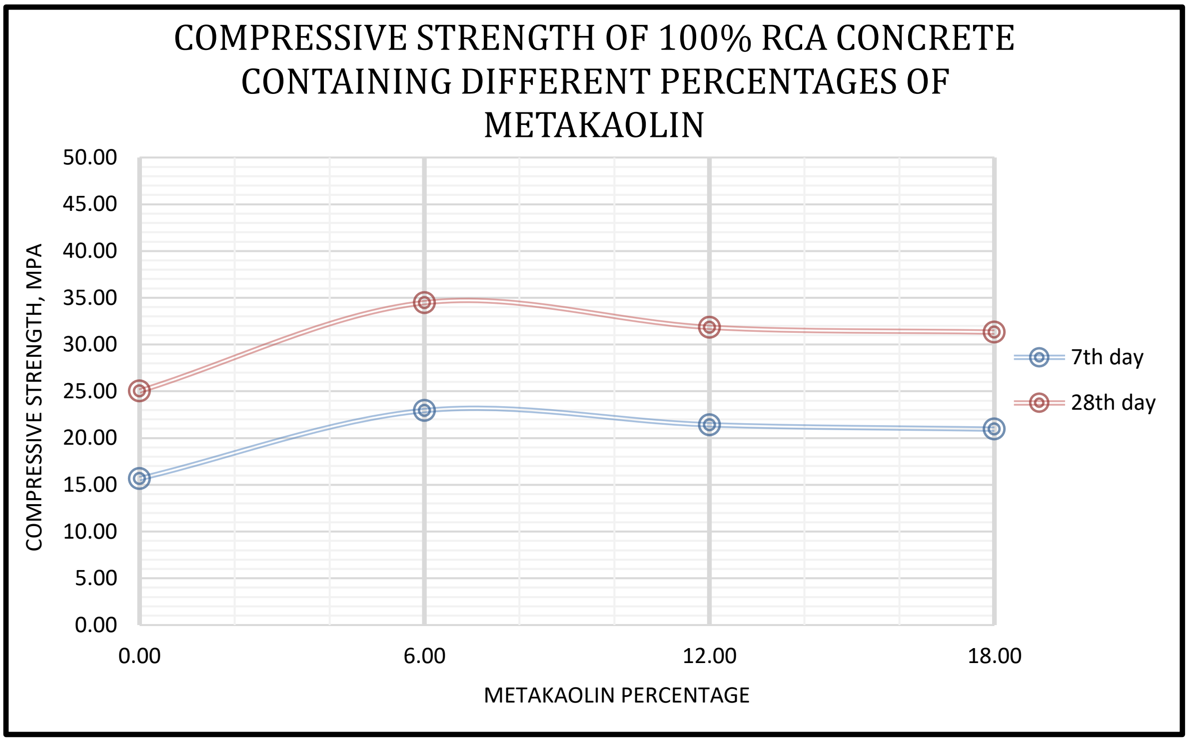

Figure 5-7: Average compressive strength of 100% RCA concrete with Metakaolin

Overall, the 7th day average compressive strength of 25%, 50%, 75% and 100% RCA replacements is increased by 14.4% (from 19.29MPa to 22.06MPa), 22.6% (from 18.08MPa to 22.16MPa), 40% (from 16.07MPa to 22.49MPa) and 46.5% (from 15.66MPa to 22.94MPa) respectively, and the 28th day average compressive strength of 25%, 50%, 75% and 100% RCA replacements can be increased by 2.1% (from 31.13MPa to 31.78MPa), 12.9% (from 28.29MPa to 31.93MPa), 22.4% (from 26.72MPa to 32.71MPa) and 37.8% (from 25.02MPa to 34.48MPa) respectively, with incorporation of metakaolin.

Table 5-12: 56th-day average compressive strength results of experiment 4

Mix notations	56th-day average compressive strength (MPa)		
	6% Metakaolin	12% Metakaolin	18% Metakaolin
2N	38.05	38.30	37.33
2R25	36.70	35.23	31.34
2R50	36.75	36.96	32.45
2R75	36.42	34.48	32.48
2R100	36.10	35.53	32.63

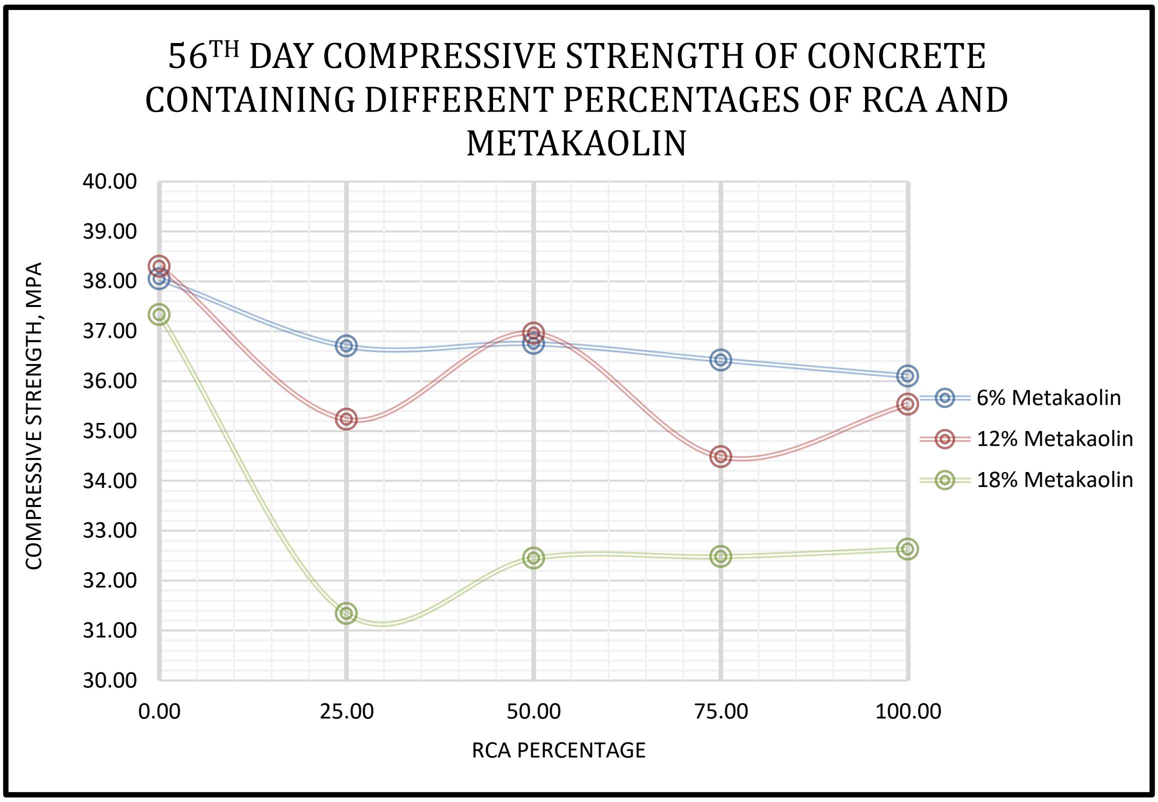

Figure 5-8: 56th-day average compressive strength of concrete containing RCA and metakaolin

It can be seen from the result that maximum average compressive strength is achieved for 6% metakaolin replacements for incorporation of 25%, 75% and 100 % RCA in place of NCA respectively, while 12% metakaolin is found to be optimum for 0% and 50% RCA replacements respectively. Additionally, maximum average compressive strength is attained for 0% RCA replacement (100% NCA) followed by 50% RCA replacement then 25%, 75% and 100% labeled in decreasing order respectively.

Finally, a comparison of the average compressive strength of NCA concrete (taken from experiment 3) with that of the maximum average compressive strength of RCA concrete achieved from the different metakaolin percentages (optimum metakaolin content) used is presented below.

Table 5-13: Comparison of average compressive strength of NCA concrete with RCA concrete containing optimum metakaolin

Mix notations	Average compressive strength (MPa)		
	7th day	28th day	56th day
2N	20.87	31.69	37.99
2R25 (6%MK)	22.06	31.78	36.70
2R50 (12%MK)	22.16	31.93	36.96
2R75 (6%MK)	22.49	32.71	36.42
2R100 (6%MK)	22.94	34.48	36.10

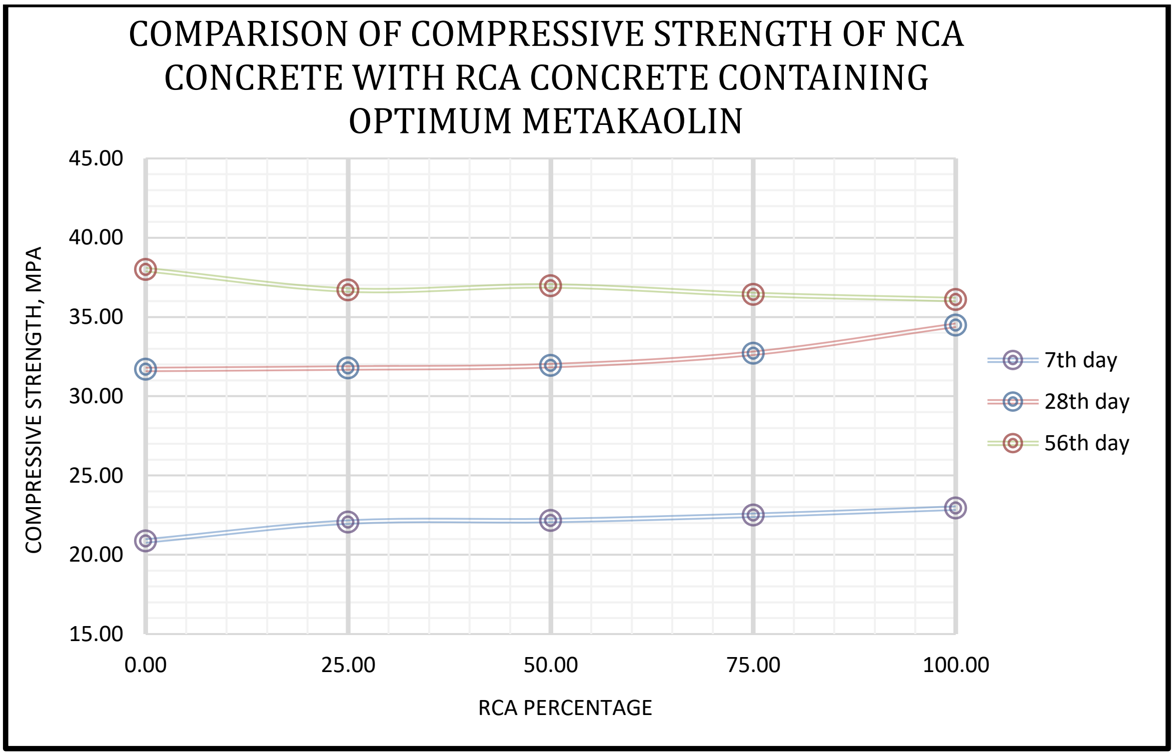

Figure 5-9: Comparison of average compressive strength of NCA concrete with RCA concrete containing optimum metakaolin

It can be seen from the results that the 7th and 28th-day average compressive strength of NCA concrete is enhanced by using metakaolin as a cement replacement for every RCA replacement. In contrast, the 56th-day average compressive strength of NCA concrete is reduced when compared with concretes containing RCA and metakaolin. The 7th day average compressive strength of NCA concrete is enhanced by 5.7%, 6.2%, 7.8% and 9.9% for 25%, 50%, 75%, 100% RCA concretes with metakaolin respectively, and the 28th day average compressive strength of NCA concrete is enhanced by 0.3%, 0.8%, 3.2% and 8.8% for 25%, 50%, 75%, 100% RCA concretes with metakaolin respectively. However, the 56th-day average compressive strength of NCA concrete is reduced by 3.4%, 2.7%, 4.1% and 5.0% for 25%, 50%, 75%, 100% RCA concrete with metakaolin respectively.

The main points noted in this experiment are:

- The compressive strength of RCA concrete is enhanced up to 6% metakaolin as cement replacement for 25%, 75% and 100% of RCA replacements respectively, while it is enhanced up to 12% metakaolin for 50% RCA. Therefore 6% metakaolin is the optimum replacement level for 25%, 75% and 100% RCA replacements respectively, whereas 12% metakaolin is optimum for 50% RCA replacement.
- The compressive strength of RCA concrete is enhanced by the incorporation of metakaolin as cement replacement and even exceeded the 7th and 28th-day compressive strengths of NCA concrete.
- The strength enhancement effect of metakaolin is more pronounced in higher RCA replacements than lower RCA replacements. This is due to the structure of RCA which contains more pores and microcracks. Consequently, more metakaolin can enter the pores and microcracks to enhance the ITZ between aggregates and cement paste.
- The compressive strength enhancement rate of metakaolin for RCA concrete is more during early ages (7 and 28 days) than later age (56 days) for the same reasons mentioned in NCA concrete containing metakaolin.

5.2.2 Weight of concrete

The 28th day dry $15cm \times 15cm \times 15cm$ concrete cube average masses, measured for different contents of RCA and Metakaolin are presented below.

Table 5-14: 28th-day average weight (Kg) of concrete cubes

Concrete cube with	0% RCA	25% RCA	50% RCA	75% RCA	100% RCA
0% Metakaolin	8.16	8.02	7.71	7.71	7.60
6% Metakaolin	8.14	7.84	7.86	7.81	7.62
12% Metakaolin	8.05	7.79	7.78	7.71	7.71
18% Metakaolin	8.10	7.81	7.71	7.69	7.72

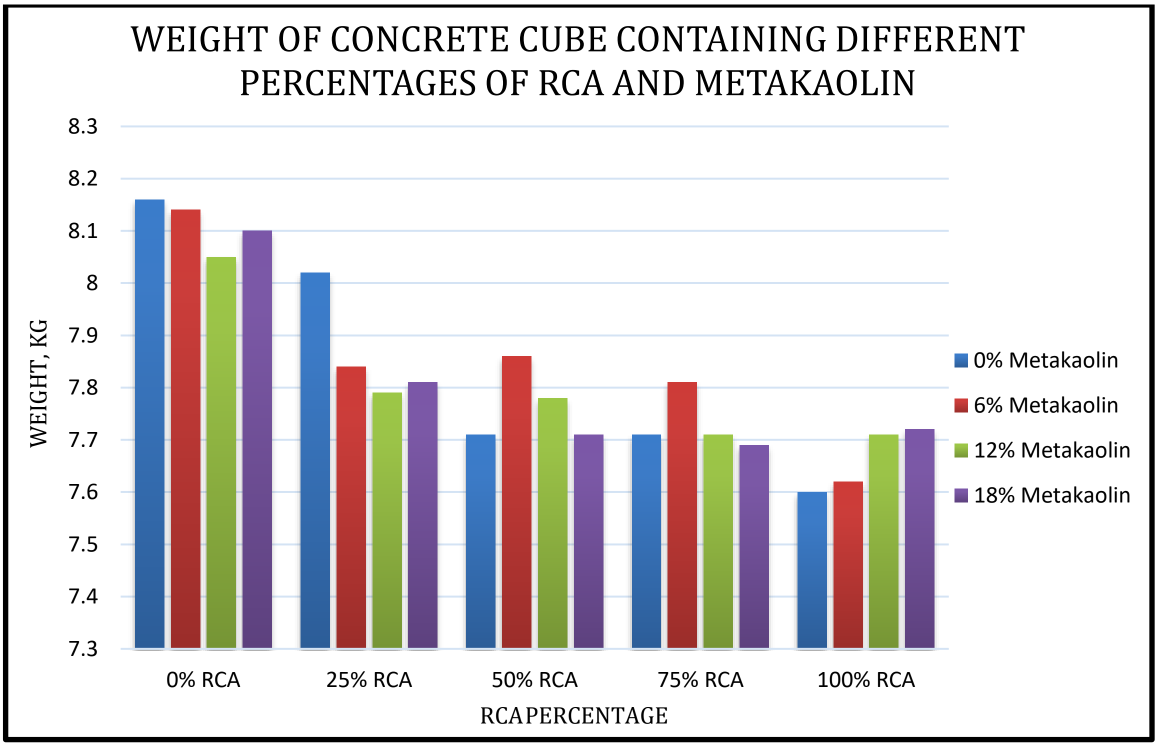

Figure 5-10: 28th-day average weight of concrete cube containing RCA and metakaolin

It can be seen from the results that the weight of concrete declines with increasing RCA content. The weight of concrete containing 25%, 50%, 75% and 100% RCA declined by 1.7%, 5.5%, 5.5% and 6.9% respectively, when compared to NCA concrete. Additionally, the incorporation of metakaolin has a varied outcome on the weight of concrete. However, to see the effects of metakaolin on the weight of concrete it is necessary to take the maximum two weights of concrete for every RCA replacement from **Table 5-14**. As such the maximum two weights of concrete containing 0% RCA are 0% and 6% metakaolin, 25% RCA are 0% and 6% metakaolin, 50% RCA are 6% and 12% metakaolin, 75% RCA are 6% and 12% metakaolin, and 100% RCA are 18% and 12% metakaolin respectively.

The main points noted in this experiment are:

- The weight of RCA concrete decreases as the percentage of RCA replacement increases. This is due to the presence of mortar attached to the RCA surface.
- Maximum weights can be achieved for higher RCA replacements by using higher metakaolin content as a cement replacement, while for lower RCA replacements, maximum weight can be achieved by using lower metakaolin content as a cement replacement. This is due to the presence of higher amount of pores and microcracks in higher RCA replacements in which more metakaolin could enter.

5.3 Metakaolin production

5.3.1 Thermal activation of kaolin clay

Figure 5-11: Muffle furnace used for heating at different temperatures

Figure 5-12: Kaolin samples before heating (Red on the left and White on the right)

Figure 5-13: Kaolin samples after heating (Red on the left and White on the right)

The mass loss results of white and red kaolin clay, which were subjected to thermal treatments at different temperatures and times, are presented below.

Table 5-15: Mass loss (%) of white kaolin clay

Heating time, min	Temperature, ^{0}c				
	600	650	700	750	800
45	13.6	15.6	16.0	16.4	16.4
90	15.2	16.0	16.4	16.4	16.4
135	15.6	16.0	16.4	16.4	16.4

Table 5-16: Mass loss (%) of red kaolin clay

Heating time, min	Temperature, ^{0}c					
	600	650	700	750	800	850
45	12.4	14.4	15.2	15.6	16.4	17.6
90	14.0	14.8	15.6	16.0	17.6	18.0
135	14.4	14.8	16.4	17.2	18.4	18.4

It can be seen from the results that the mass loss of white kaolin increased up to 700^{0}c for a heating time of 90 min, from this point on increasing the temperature and heating time has no effect on mass loss. In contrast, the mass loss of red kaolin increased up to 800^{0}c for a heating time of 135 min, from this point on increasing the temperature has no effect on mass loss as long as the heating time remained 135 min.

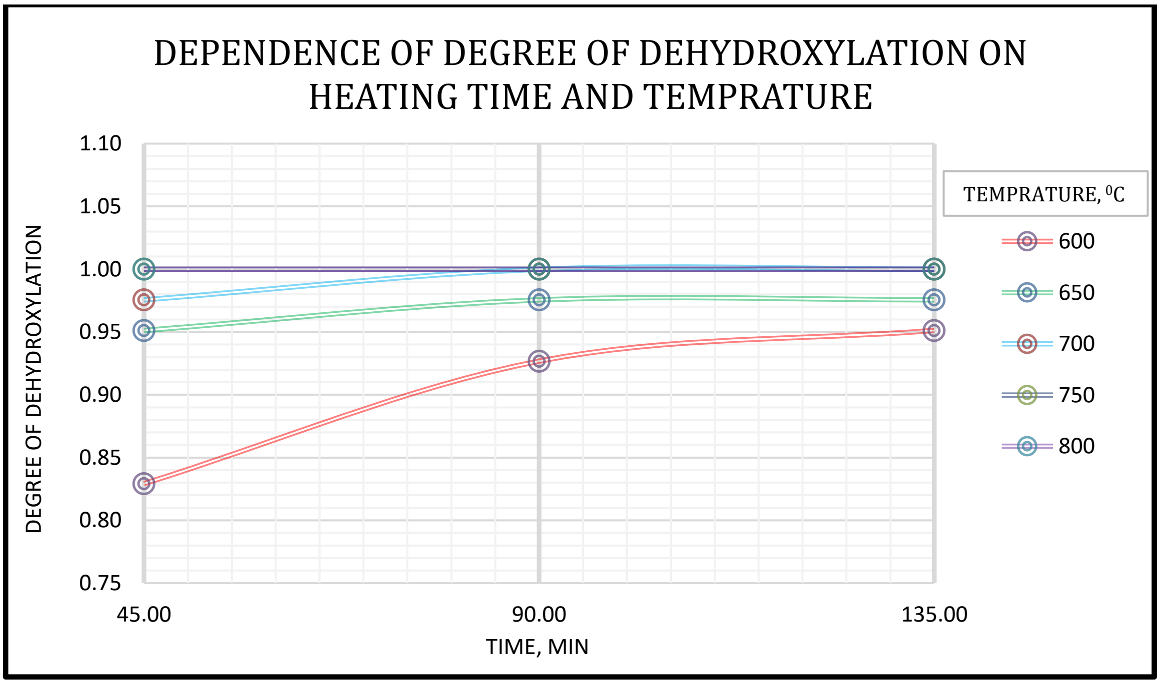

Figure 5-14: Dependence of degree of dehydroxylation on heating time and temperature for white kaolin

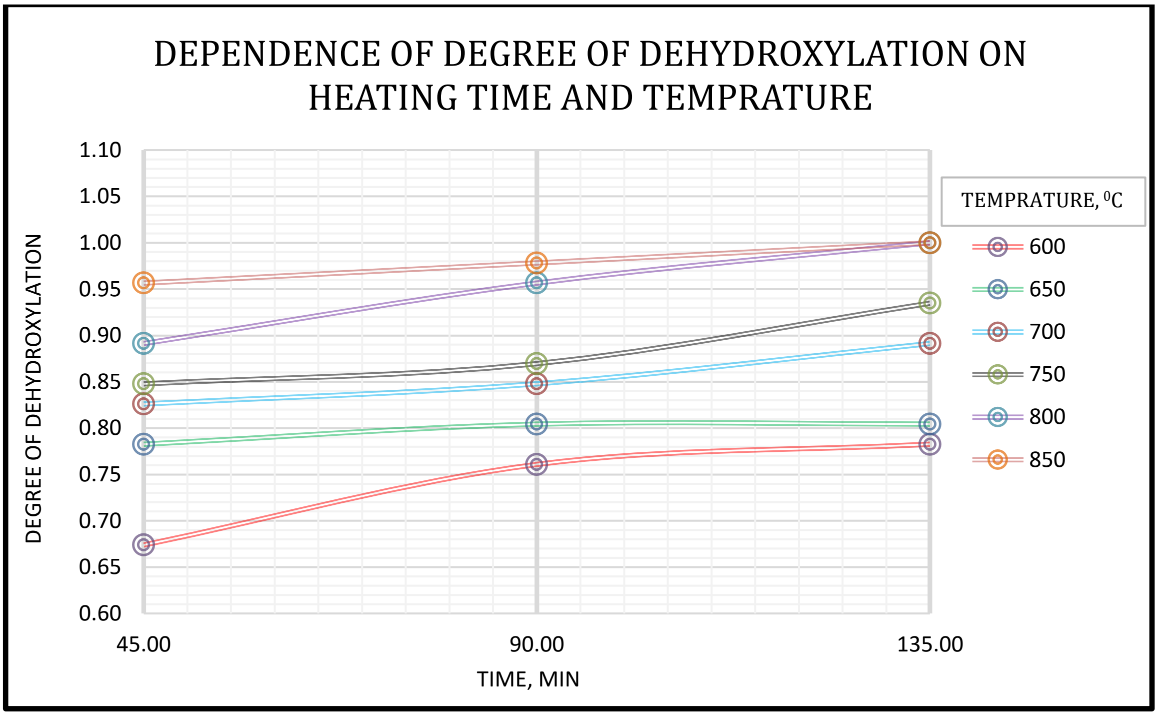

Figure 5-15: Dependence of degree of dehydroxylation on heating time and temperature for red kaolin

The degree of dehydroxylation (D_{tg}) is the main quantitative criterion for estimating the performance kaolinite by thermal treatment which is calculated as $D_{tg} = m/m_0$, where m and m_0 are residual and maximum mass loss respectively (Rahier et al. 2000). Accordingly, maximum mass losses of white kaolin and red kaolin are 16.4 and 18.4 respectively, while the residual mass losses are the particular mass losses depicted in **Table 5-15** and **Table 5-16.**

The first full dehydroxylation ($D_{tg} = 1$) of white kaolin occurred at 700^0c with heating time of 90 min and 750^0c with heating time of 45 min respectively, while it occurred at 800^0c with heating time of 135 min for red kaolin. These points are presented in **Figure 5-14** and **Figure 5-15.**

Furthermore, after thermal treatment, the XRD patterns of beginning clay and calcined clay were compared to check the disappearance of kaolinite peaks. The XRD analysis results of both white and red kaolin samples are presented from **Figure 5-16** up to **Figure 5-21**.

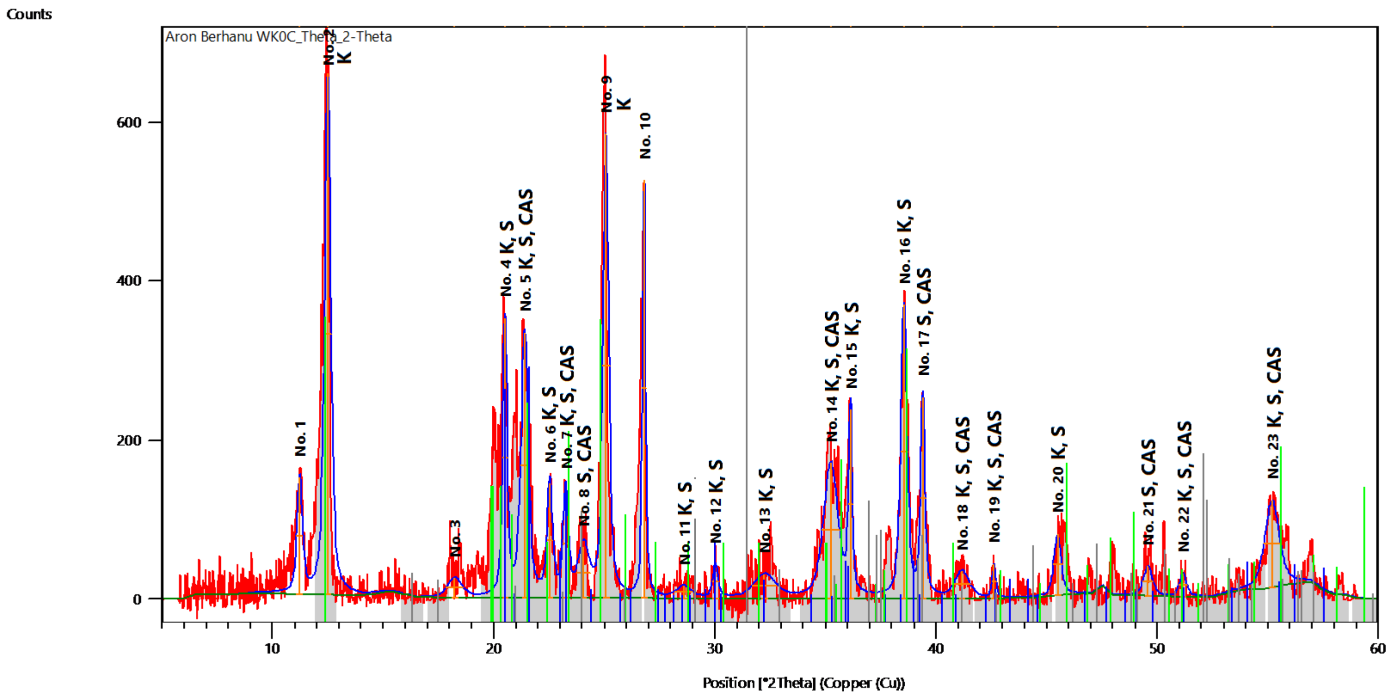

Figure 5-16: XRD pattern of white kaolin without thermal treatment

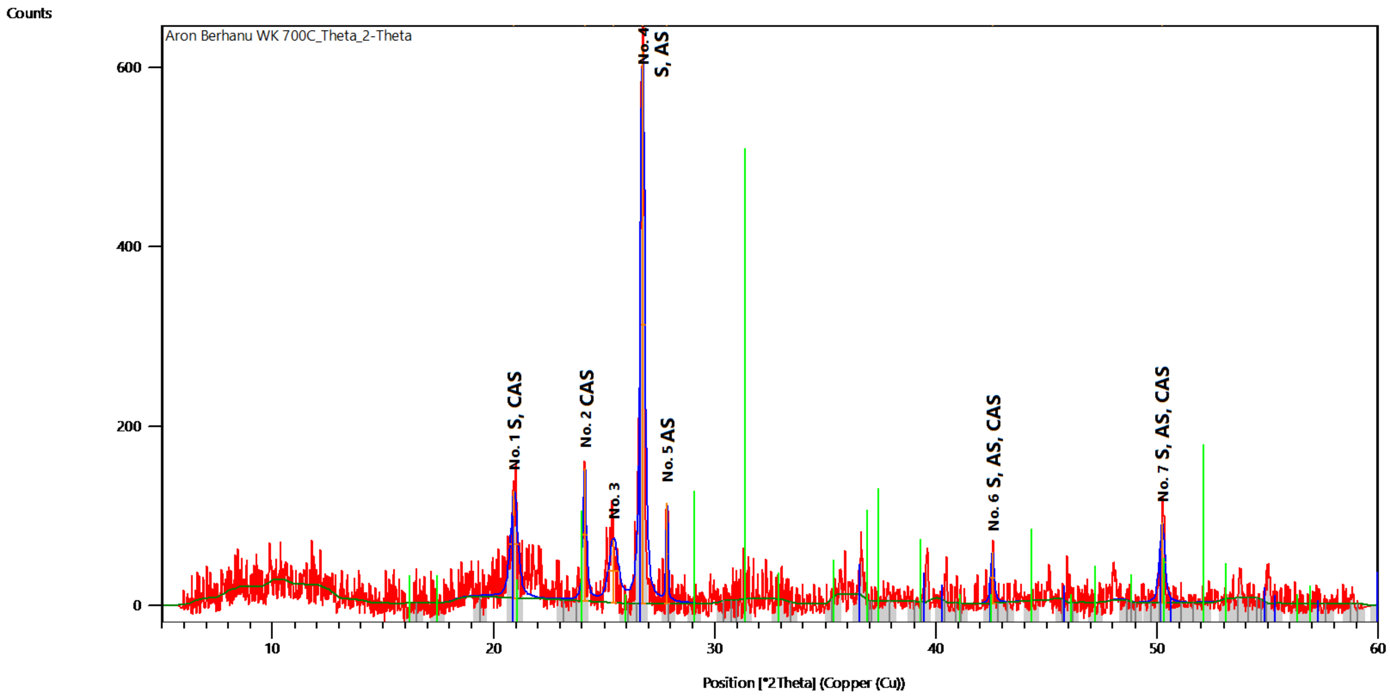

Figure 5-17: XRD pattern of white kaolin thermally treated at 700 ^{0}C for 90min

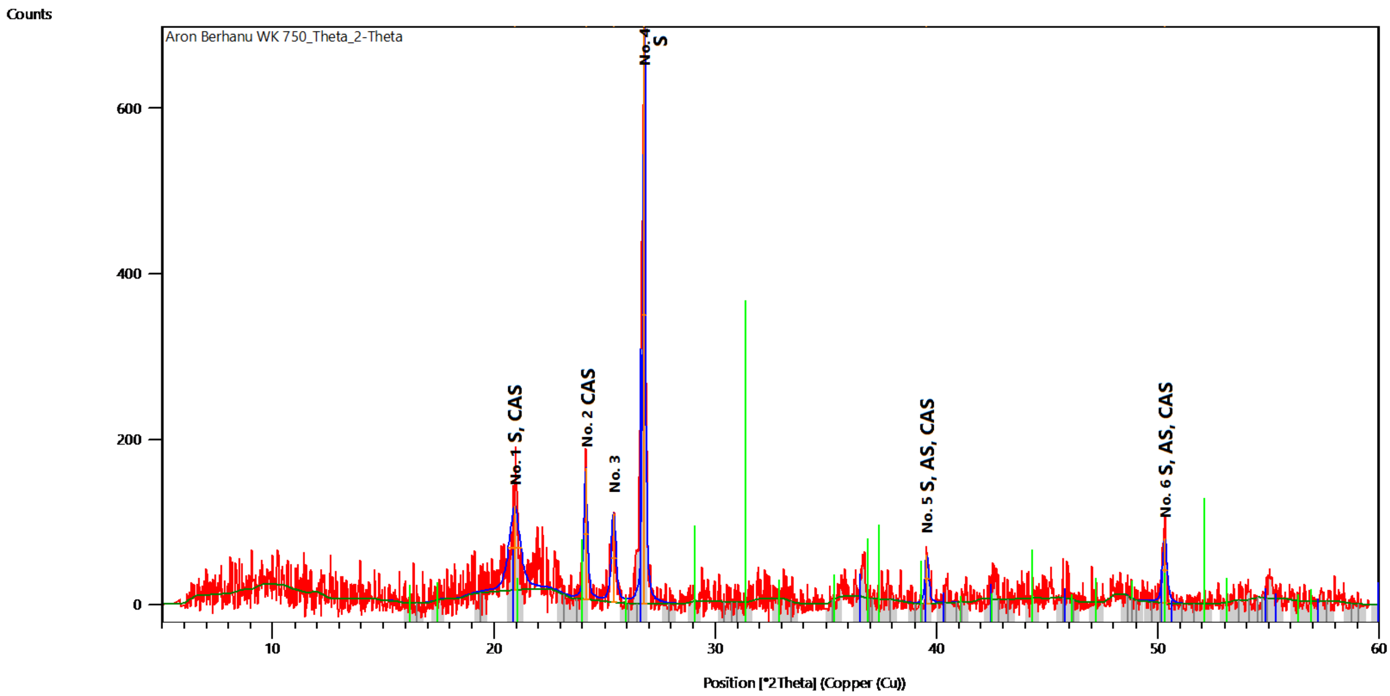

Figure 5-18: XRD pattern of white kaolin thermally treated at 750 ^{0}C for 45min

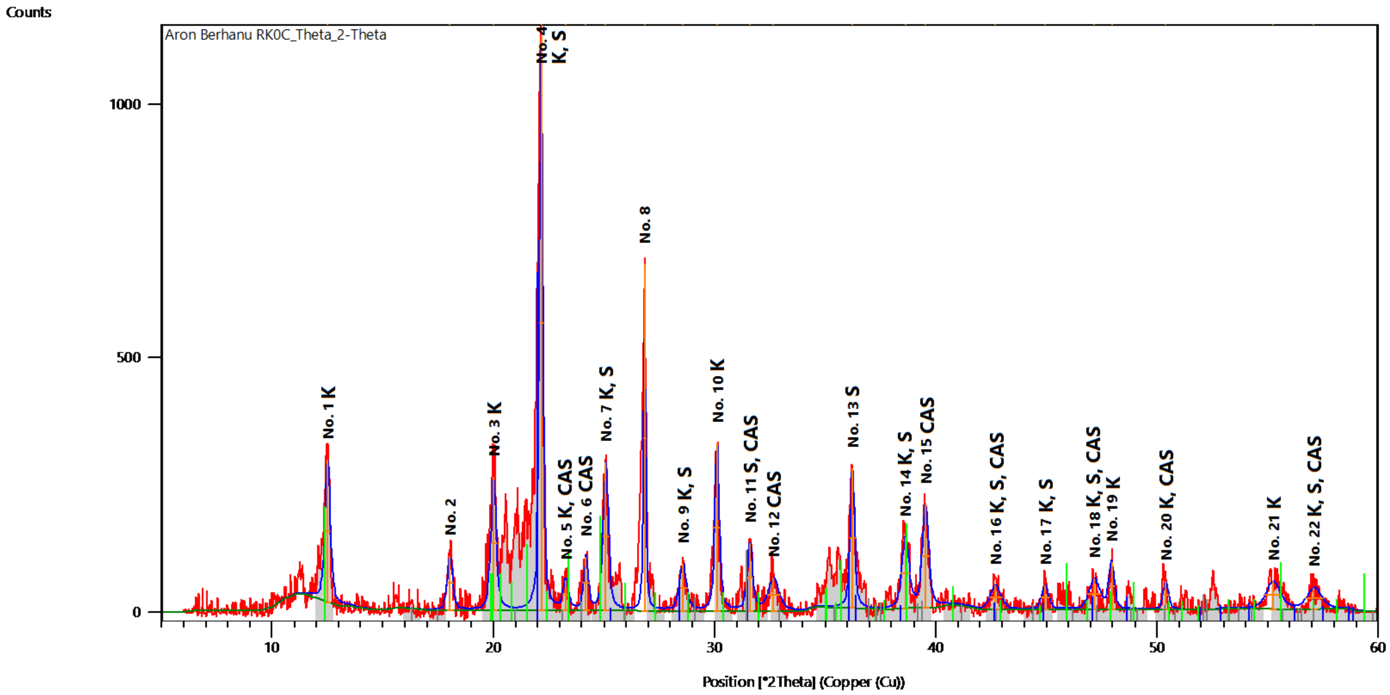

Figure 5-19: XRD pattern of red kaolin without thermal treatment

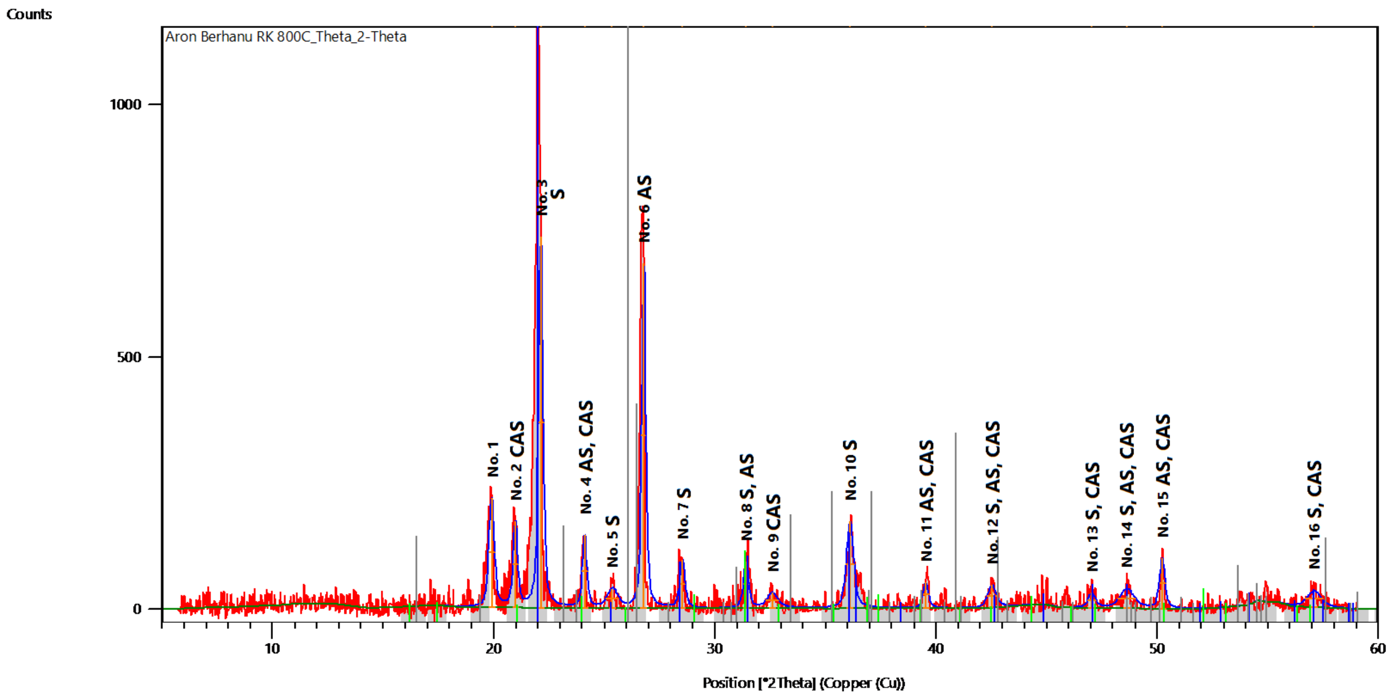

Figure 5-20: XRD pattern of red kaolin thermally treated at 800 ^{0}C for 135min

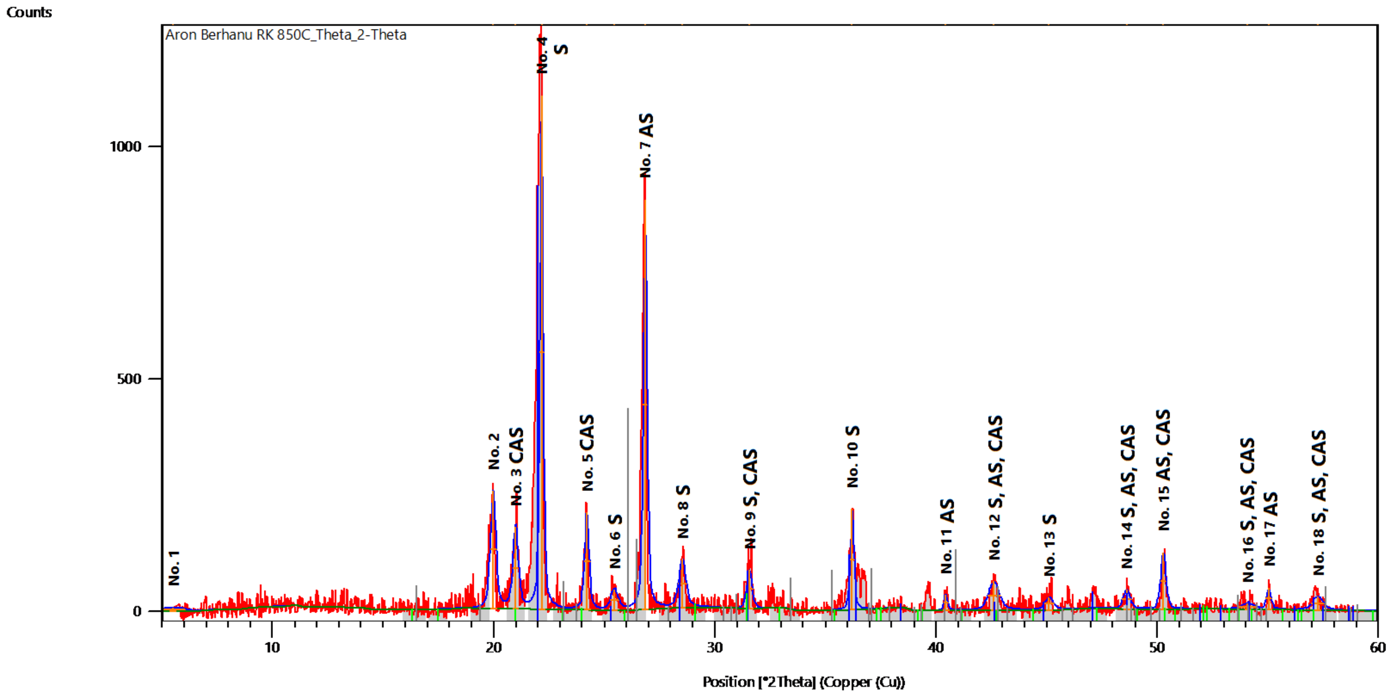

Figure 5-21: XRD pattern of red kaolin thermally treated at 850 ^{0}C for 135min

Table 5-17: The compound names and chemical formulas of the abbreviations used in the XRD analysis

Number	Abbreviation	Compound Name	Chemical Formula
1	K	Kaolinite	Al2 Si2 O5 (O H)4
2	S	Silicon Oxide	Si O2
3	AS	Aluminum Silicate	Al2 Si O5
4	CAS	Calcium Aluminum Silicate	Ca2 Al2 Si O7

The main points noted in this experiment are:

- Metakaolin can be produced from kaolin by heating at temperatures ranging from 600^0c - 850^0c. This point is mentioned in previous literature (Narmatha and Felixkala 2016).

- Metakaolin can be produced from white kaolin with a heating temperature of 700^0c and a heating time of 90 min or with a heating temperature of 750^0c and a heating time of 45 min while it is produced from red kaolin with a heating temperature of 800^0c and a heating time of 135 min.

CHAPTER 6 SOFTWARE SIMULATION

6.1 About the software

Analytical simulations for parts of the research were done using Virtual Cement and Concrete Testing Laboratory (VCCTL) software version 9.5. The software was developed at the National Institute of Standards and Technology (NIST) in the USA. This allows users to easily measure hydration and quantify a range of engineering characteristics of cement paste, mortar, and concrete.

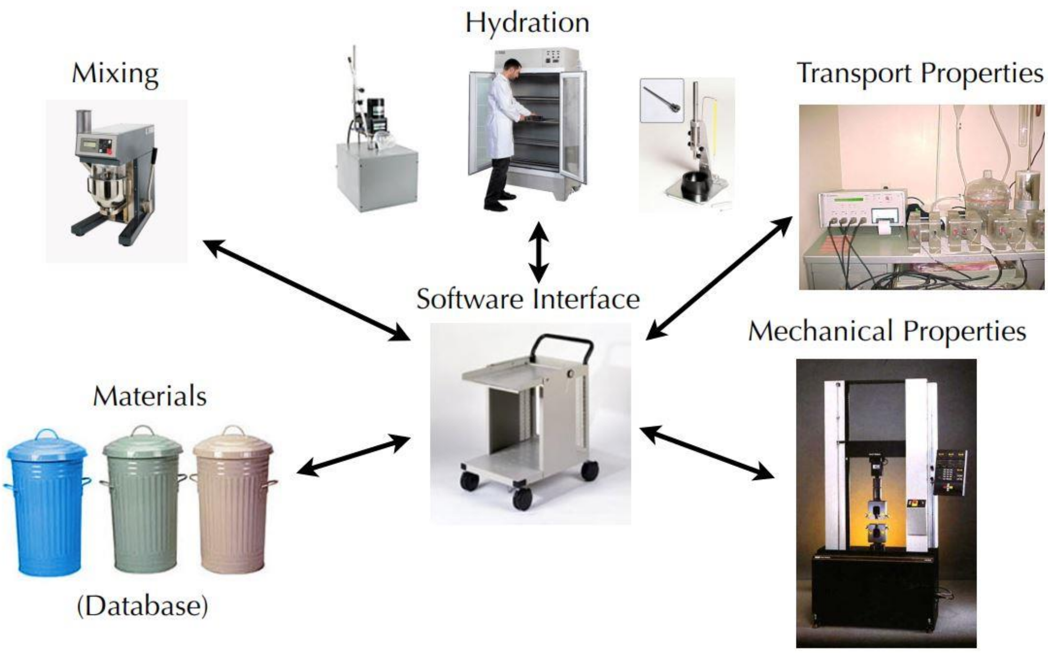

Figure 6-1: VCCTL software design (Bullard et al. 2013)

VCCTL models have been designed on the basis of chemistry, physics and material science. "It simulates microstructure development of cement hydration at a micrometer scale and an integrated finite element program is used to calculate the effective linear elastic moduli of the hydrated cement paste, and these computed data are then used in a second C program, based on differential effective medium theory, to compute the elastic properties and to estimate the compressive strength of the mortar or concrete" (Bullard et al. 2009). The basic workflow of VCCTL is presented in **Figure 6-1.**

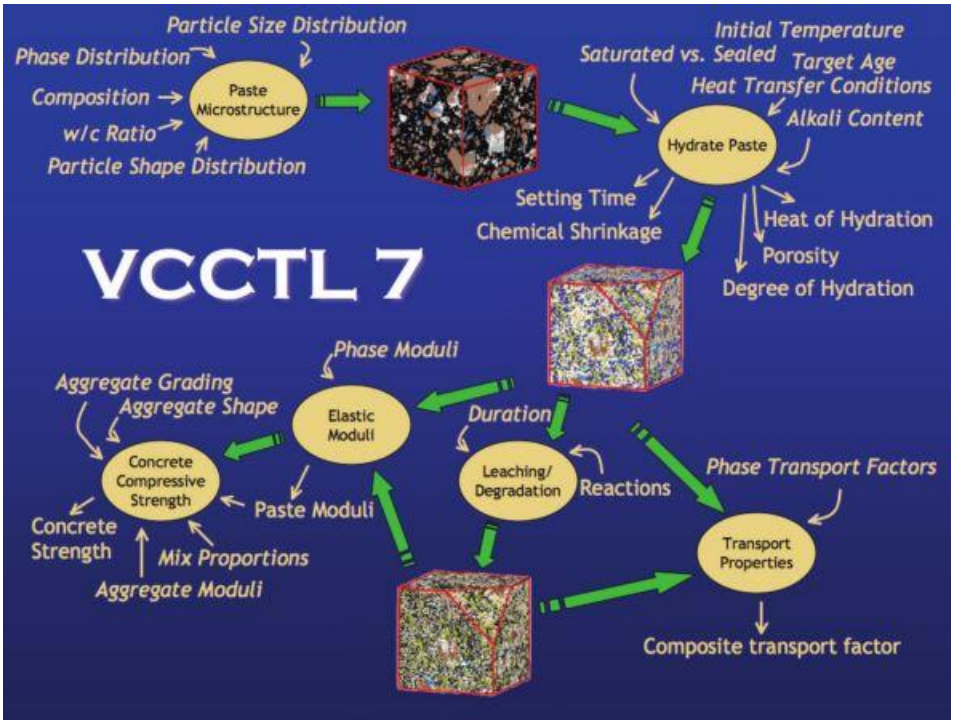

Figure 6-2: VCCTL software workflow, showing the various steps of virtual testing and the inputs (in italics) and outputs (Bullard et al. 2009)**.**

6.2 Simulated compressive strength

The compressive strength of the parent concrete in order to produce RCA was simulated. Additionally, the compressive strengths of NCA concrete with and without metakaolin and RCA concrete with different percentages of RCA were simulated.

6.3 VCCTL inputs

6.3.1 Materials

6.3.1.1 Cement

The main data needed to characterize the cement used in this research are PSD and phase fractions of clinker composition. The PSD of cement done with sieve and hydrometer analysis is attached in the APPENDIX A. However, finer particle sizes of cement are extrapolated from the hydrometer test which is shown in **Table 6-2**. The clinker composition of cement, Dangote brand, was taken from a previous research paper done in Addis Ababa Institute of Technology (Siraj 2015).

Table 6-1: Phase fractions of clinker composition of Dangote cement

Clinker composition	C_3S	C_2S	C_3A	C_4AF
Phase fractions	57.29	22.22	5.45	10.25

Table 6-2: PSD of Dangote cement

Diameter, μm	Percent Finer, %	Method of PSD determination
150.0000	99.500	Sieve analysis
125.0000	99.100	
75.0000	94.900	
63.0000	90.200	
44.7603	85.417	Hydrometer analysis
31.6503	85.417	
22.8908	82.977	
14.6888	80.536	
8.5709	79.316	
6.1238	78.096	
4.5316	71.260	
3.2816	66.864	
2.3852	59.776	
1.6998	58.556	
1.2495	53.684	
1.0586	48.810	
0.9009	47.577	Extrapolation of hydrometer analysis
0.8359	42.703	
0.7902	37.828	
0.7176	36.598	
0.6950	31.722	
0.6774	26.845	
0.6294	25.618	
0.6205	20.742	
0.6134	15.863	
0.5764	14.639	
0.5736	9.761	
0.5715	4.881	
0.5406	3.660	
0.5411	0	

6.3.1.2 Metakaolin

The main data needed to characterize the metakaolin used in this research are PSD and phase distribution components. The PSD of metakaolin done with sieve and hydrometer analysis is attached in APPENDIX A.

Table 6-3: PSD of Metakaolin

Diameter, μm	Percent Finer, %	Method of PSD determination
150.000	100.000	Sieve analysis
125.000	99.710	
75.000	99.350	
63.000	98.250	
40.035	97.620	Hydrometer analysis
28.848	95.179	
20.958	91.519	
13.824	86.638	
8.359	83.587	
5.910	81.757	
4.302	77.361	
3.125	74.185	
2.263	68.926	
1.601	67.096	
1.193	62.835	
1.002	58.572	
0.846	57.946	Extrapolation of hydrometer analysis
0.778	53.684	
0.731	49.420	
0.659	48.797	
0.634	44.534	
0.614	40.268	
0.567	39.648	
0.556	35.383	
0.547	31.116	
0.511	30.498	
0.506	26.232	
0.502	21.964	
0.473	21.349	
0.471	17.081	
0.470	12.813	
0.444	12.199	
0.445	7.931	
0.445	3.661	
0.423	3.050	
0.424	0	

Finer particle sizes of metakaolin are also extrapolated from the hydrometer test as it was done for cement. The phase distribution components of the metakaolin are obtained from XRD results presented in APPENDIX E plus chemical analysis using limiting reagent.

Table 6-4: Phases of metakaolin

Phases	AS	CAS2	Silica
Phase fractions	53.74	0.64	42.36

6.3.1.3 Aggregates

The main data needed to characterize aggregates are PSD, specific gravity, bulk modulus, and shear modulus.

6.3.1.3.1 Fine aggregates

The specific gravity and PSD of this aggregate are presented in **Table 3-2** and **Table 3-3** respectively, in the materials and properties section of this paper. Additionally, the bulk and shear modulus of this aggregate is initially inputted in the software as aggregates that meet ASTM C-33 requirements.

6.3.1.3.2 Natural coarse aggregate

To determine the bulk modulus and shear modulus of this aggregate first, it is necessary to determine the modulus of elasticity and Poisson's ratio of this aggregate. Accordingly, the parent material from which this aggregate is produced from (basaltic rock) has a modulus of elasticity ranging from 20-80Kg/m^2 and Poisson's ratio ranging from 0.1- 0.25 (Suryakanta 2015). Thus, the modulus of elasticity and Poisson's ratio are found to be 53GPa and 0.25, respectively, by simulation of the test concretes.

The next step is the derivation of bulk and shear modulus using elasticity equations provided in **Table 6-5**.

Table 6-5: Elastic constants and their relationship

Input constants	Output relations			
	E (Modulus of elasticity) =	ν (Poisson's ratio) =	K (Bulk modulus) =	G (Shear modulus) =
E, ν	-	-	$\frac{E}{3(1-2\nu)}$	$\frac{E}{2(1+\nu)}$
E, G	-	$\frac{E-2G}{2G}$	$\frac{EG}{3(3G-E)}$	-
E, K	-	$\frac{3K-E}{6K}$	-	$\frac{3KE}{9K-E}$

Table 6-6: Elasticity properties of NCA

Parent rock type	Modulus of elasticity (E)	Poisson's ratio (v)	Bulk modulus (K)	Shear modulus (G)
Basalt Rock	53GPa	0.25	35.33GPa	21.2GPa

6.3.1.3.2.1 Well-graded NCA type 1

The specific gravity and PSD of this aggregate are presented in **Table 3-4** and **Table 3-5** respectively, in the materials and properties section of this paper.

6.3.1.3.2.2 Well-graded NCA type 2

The specific gravity and PSD of this aggregate are presented in **Table 3-9** and **Table 3-6** respectively, in the materials and properties section of this paper.

6.3.1.3.3 Recycled coarse aggregate

The specific gravity and PSD of this aggregate are presented in **Table 3-13** and **Table 3-6** respectively, in the materials and properties section of this paper.

The modulus of elasticity of this aggregate is estimated from the modulus of elasticity of NCA. The modulus of elasticity reduction range for fully replacing NCA with RCA is 20-25 % (Kheder and Al-Windawi 2005**.** Accordingly, 22% reduction is taken by simulation of test concretes and, the bulk and shear modulus are determined using equations depicted in **Table 6-7.**

Table 6-7: Elasticity properties of RCA

Aggregate composition	Modulus of elasticity (E)	Poisson's ratio (v)	Bulk modulus (K)	Shear modulus (G)
100% RCA	43.44GPa	0.25	28.96GPa	17.38GPa

The properties of different replacement percentages of RCA are derived from NCA and 100% RCA properties. The computations are presented below.

A. 25% RCA and 75% NCA

   Modulus of elasticity (E) = $0.25 \times 43.44 + 0.75 \times 53 = 50.61$GPa

   Specific gravity = $0.25 \times 2.39 + 0.75 \times 2.72 = 2.64$

B. 50% RCA and 50% NCA

Modulus of elasticity (E) = $0.5 \times 43.44 + 0.5 \times 53$ = 48.22GPa

Specific gravity = $0.5 \times 2.39 + 0.5 \times 2.72$ = 2.56

C. 75% RCA and 25% NCA

Modulus of elasticity (E) = $0.75 \times 43.44 + 0.25 \times 53$ = 45.83GPa

Specific gravity = $0.75 \times 2.39 + 0.25 \times 2.72$ = 2.47

Finally, the bulk and shear modulus of 25% RCA, 50% RCA and 75% RCA are similarly derived from **Table 6-8.**

Table 6-8: Elasticity properties of different percentages of RCA

Aggregate composition	Modulus of elasticity (E)	Poisson's ratio (v)	Bulk modulus (K)	Shear modulus (G)
25% RCA and 75% NCA	50.61GPa	0.25	33.74GPa	20.24GPa
50% RCA and 50% NCA	48.22GPa	0.25	32.15GPa	19.29GPa
75% RCA and 25% NCA	45.83GPa	0.25	30.55GPa	18.33GPa

6.3.2 Mix design

The main mix designs that are used for simulation are provided in **Table 4-1,** in the experimental program chapter of this paper.

6.3.3 Other inputs

- Cement hydration reaction activation energy = 40KJ/Mole (ASTM C 1074 1999)
- Initial temperature for well-graded NCA type 1 = 23^0c
- Initial temperature for well-graded NCA type 2 and RCA = 20^0C

6.4 VCCTL outputs

6.4.1 28th-day compressive strength results

Table 6-9: Comparison of laboratory compressive strength results of RCA concrete with VCCTL simulations

Mix notations	Laboratory compressive strength (MPa)	VCCTL compressive strength (MPa)	Deviation of VCCTL result from Laboratory result (%)
1N	33.88	32.55	3.93
2N	32.04	31.77	0.84
2R25	31.13	30.36	2.47
2R50	28.29	27.98	1.10
2R75	26.73	26.52	0.79
2R100	25.02	24.95	0.28

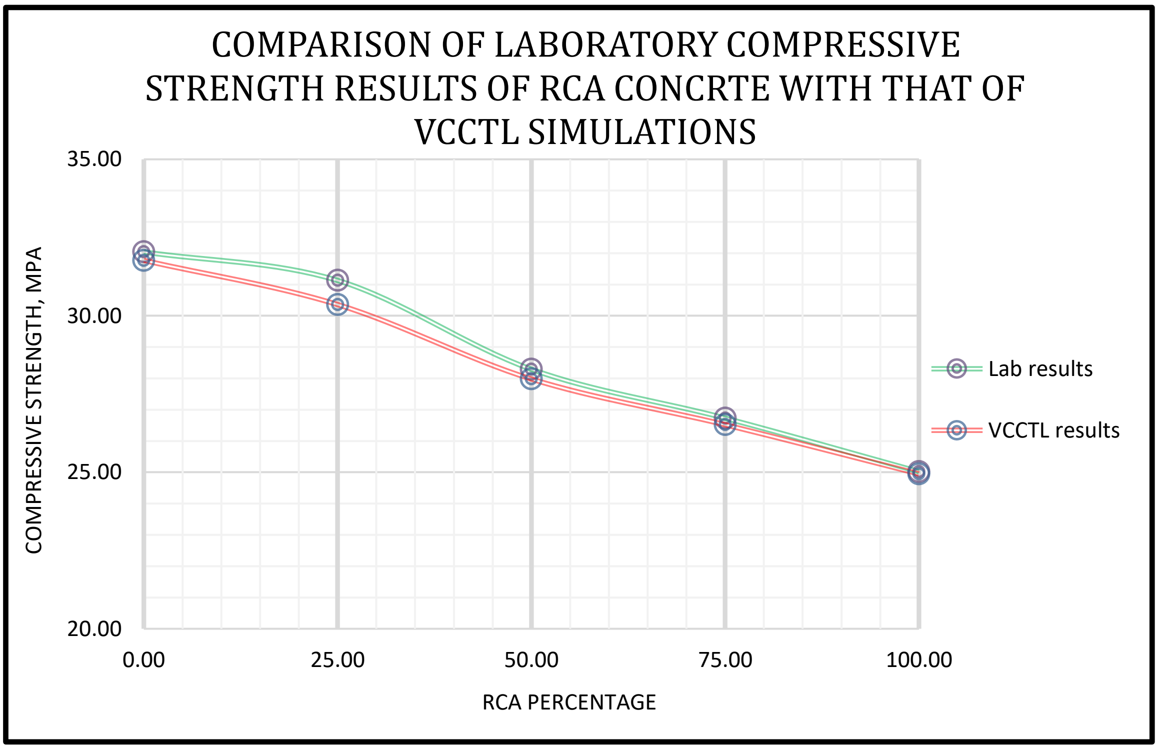

Figure 6-3: Comparison of laboratory compressive strength results of RCA concrete with VCCTL simulations

Table 6-10: Comparison of laboratory compressive strength results of metakaolin concrete with VCCTL simulations

Mix notations	Laboratory compressive strength (MPa)	VCCTL compressive strength (MPa)	Deviation of VCCTL result from Laboratory result (%)
1N	33.88	32.55	3.93
2N	31.69	31.77	0.25
2NM6	31.86	31.88	0.05
2NM12	33.55	33.92	1.11
2NM18	30.78	33.15	7.71
2NM24	29.83	31.90	6.93

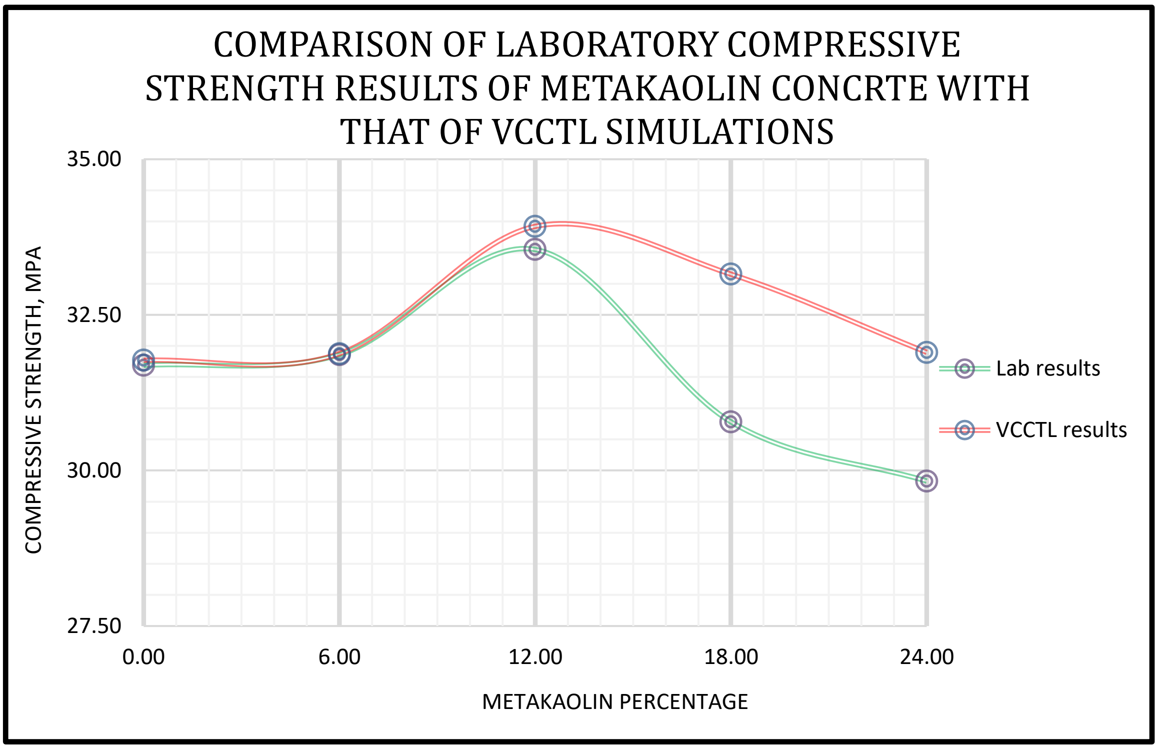

Figure 6-4: Comparison of laboratory compressive strength results of metakaolin concrete with VCCTL simulations

It can be seen from the results that VCCTL software accurately simulated the laboratory compressive strength results. Additionally, VCCTL simulations captured the laboratory results with less than 8.0% error.

CHAPTER 7 CONCLUSIONS AND RECOMMENDATIONS

7.1 Conclusions

In this paper, the effects of RCA and metakaolin on the compressive strength as well as the physical properties of concrete have been investigated. It should be noted that the RCA used in this experiment was obtained from the crushing of laboratory concrete cubes, which were first-hand cast and had an average compressive strength of 33.88 MPa.

On the basis of the results obtained, the following conclusions and suggestions are drawn:

1. The workability of RCA concrete increases with an increase in RCA replacement while the workability of concrete containing metakaolin as cement replacement decreases with an increase in metakaolin content
2. The compressive strength of both RCA and NCA concrete increases with incorporation of optimal amount of metakaolin as cement replacement.
3. The optimum content for replacing cement with metakaolin in NCA concrete and RCA concrete is 12% and 6% respectively.
4. The 7th-day and 28th-day compressive strength of RCA concrete with metakaolin as cement replacement exceeded the compressive strengths of NCA concrete but the 56th-day compressive strength of RCA concrete with metakaolin as cement replacement decreased when compared to NCA concrete and to a maximum difference of 5%.
5. The strength enhancement effect of metakaolin is more pronounced in the higher percent replacements of RCA concrete than the lower percent replacements.
6. The compressive strength enhancement rate of metakaolin as cement replacement is more during early ages (7 and 28 days) than later age (56 days) for both NCA and RCA concrete.
7. RCA concretes have lower weights than NCA concrete and could be minimized to a maximum of 6.9% by fully replacing RCA in place of NCA.
8. Metakaolin can be produced from locally found white kaolin with a heating temperature of 700^{0}c and a heating time of 90 min or with a heating temperature of 750^{0}c and a heating time of 45 min while it can be produced from locally found red kaolin with a heating temperature of 800^{0}c and a heating time of 135 min.

7.2 Recommendations

- In order to reduce the high workability of RCA concrete, the RCA must be pre-soaked to a level where the difference in moisture content and absorption capacity is less than 1%.
- In both NCA concrete and RCA concrete, metakaolin can be used to partially replace OPC with the added benefit of increase in strength.
- Metakaolin can be produced in Ethiopia from locally occurring white and red kaolin but in order to enhance the engineering characteristics of metakaolin, the original kaolin whereby metakaolin is produced must be further purified and grounded.

On the basis of this research and other similar studies, which have been carried out in this particular area, the work can be extended to include:

- Use of RCA concrete with metakaolin as cement replacement in high strength concrete.
- Thermal behavior of RCA concrete with metakaolin as cement replacement.
- Effect of particle size distribution in RCA concrete or a better optimization technique in order to quantify the amount of mortar paste adhered to the aggregate.
- Permeability characteristics of RCA concrete with metakaolin as cement replacement.
- Durability, shrinkage and creep properties of RCA concrete with metakaolin as cement replacement.
- Microstructural studies including SEM and XRD in order to characterize metakaolin extracted from other sources in Ethiopia as well as a detailed investigation of the ITZ of RCA containing metakaolin as cement replacement.
- A thorough environmental and economical analysis of RCA concrete with metakaolin as cement replacement.

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APPENDIX

APPENDIX A

A1. HYDROMETER AND SIEVE ANALYSIS TEST RESULTS

Cement		Metakaolin		Method of PSD determination
Diameter, mm	Percent Finer, %	Diameter, mm	Percent Finer, %	
0.1500000	99.500	0.150000	100.000	Sieve analysis
0.1250000	99.100	0.125000	99.710	
0.0750000	94.900	0.075000	99.350	
0.0630000	90.200	0.063000	98.250	
0.0447603	85.417	0.040035	97.620	Hydrometer analysis
0.0316503	85.417	0.028848	95.179	
0.0228908	82.977	0.020958	91.519	
0.0146888	80.536	0.013824	86.638	
0.0085709	79.316	0.008359	83.587	
0.0061238	78.096	0.005910	81.757	
0.0045316	71.260	0.004302	77.361	
0.0032816	66.864	0.003125	74.185	
0.0023852	59.776	0.002263	68.926	
0.0016998	58.556	0.001601	67.096	
0.0012495	53.684	0.001193	62.835	
0.0010586	48.810	0.001002	58.572	

APPENDIX B

B1. 28TH-DAY COMPRESSIVE STRENGTH RESULTS OF EXPERIMENT 1

Mix (1N)	Number	Mass	Load (KN)	Compressive strength (MPa)
Batch 1	1	8.095	790.0	35.11
	2	8.030	798.8	35.50
	3	8.025	810.0	36.00
	4	8.025	815.4	36.24
	5	8.120	811.4	36.06
	6	8.090	792.0	35.20
	7	8.115	817.2	36.32
	8	8.140	804.6	35.76
	9	7.935	793.8	35.28
	10	-	-	7th-day trial
Batch 2	1	7.885	739.8	32.88
	2	8.080	817.2	36.32
	3	8.010	772.9	34.35
	4	7.950	775.7	34.47
	5	8.060	808.0	35.91
	6	8.155	763.5	33.93
	7	8.050	799.7	35.54
	8	8.030	803.4	35.71
	9	8.040	759.1	33.74
	10	8.105	832.0	36.98
Batch 3	1	8.010	740.5	32.91
	2	8.030	788.8	35.06
	3	7.980	754.6	33.53
	4	8.050	785.6	34.92
	5	8.060	790.9	35.14
	6	8.025	712.3	31.66
	7	8.015	702.8	31.23
	8	8.025	690.4	30.68
	9	7.910	743.8	33.06
	10	8.020	741.9	32.97
Batch 4	1	8.070	817.1	36.31
	2	7.985	812.4	36.10
	3	8.055	807.4	35.88
	4	7.975	827.2	36.76
	5	7.970	814.8	36.21
	6	8.060	802.5	35.66
	7	8.095	771.2	34.27
	8	8.020	768.9	34.17
	9	8.080	770.0	34.22
	10	-	-	7th-day trial

B1. (CONTINUED)

Mix (1N)	Number	Mass	Load (KN)	Compressive strength (MPa)
Batch 5	1	8.260	781.2	34.72
	2	7.915	717.1	31.87
	3	7.960	806.6	35.85
	4	7.875	758.9	33.73
	5	7.955	809.5	35.98
	6	7.920	743.2	33.03
	7	8.030	815.7	36.25
	8	8.035	724.2	32.19
	9	8.150	798.5	35.49
	10	-	-	7^{th}- day trial
Batch 6	1	7.925	709.2	31.52
	2	7.905	732.7	32.57
	3	7.950	728.3	32.37
	4	8.300	771.0	34.27
	5	8.055	774.5	34.42
	6	7.900	775.8	34.48
	7	8.030	794.1	35.29
	8	8.100	746.9	33.19
	9	8.000	752.9	33.46
	10	8.040	757.5	33.66
Batch 7	1	8.210	803.9	35.73
	2	8.100	728.1	32.36
	3	8.080	698.7	31.05
	4	8.050	759.5	33.76
	5	8.050	734.7	32.65
	6	8.100	734.8	32.66
	7	8.075	759.6	33.76
	8	8.090	749.4	33.30
	9	7.990	709.3	31.52
	10	-	-	7^{th}-day trial
Batch 8	1	7.870	754.8	33.55
	2	7.975	781.4	34.72
	3	7.980	746.3	33.17
	4	7.875	714.5	31.75
	5	8.225	722.4	32.11
	6	8.205	810.4	36.02
	7	8.325	773.8	34.39
	8	7.900	790.5	35.13
	9	7.900	772.4	34.33
	10	8.445	774.5	34.42

B1. (CONTINUED)

Mix (1N)	Number	Mass	Load (KN)	Compressive strength (MPa)
Batch 9	1	7.995	740.8	32.92
	2	8.060	759.9	33.77
	3	8.105	721.9	32.08
	4	8.025	734.0	32.62
	5	7.970	768.2	34.14
	6	8.130	731.5	32.51
	7	8.010	733.8	32.61
	8	7.915	702.5	31.22
	9	8.180	726.1	32.27
	10	8.030	714.4	31.75
Batch 10	1	7.975	720.5	32.02
	2	7.990	799.9	35.55
	3	8.085	750.9	33.37
	4	8.060	735.6	32.69
	5	8.200	709.3	31.52
	6	7.955	701.6	31.18
	7	8.005	755.6	33.58
	8	8.030	745.3	33.12
	9	8.055	753.8	33.50
	10	-	-	7^{th}-day trial
Batch 11	1	7.920	717.2	31.88
	2	8.160	731.6	32.52
	3	8.000	729.9	32.44
	4	8.055	692.0	30.75
	5	7.975	763.2	33.92
	6	8.115	778.9	34.62
	7	8.090	847.1	37.65
	8	8.030	704.2	31.29
	9	8.110	755.7	33.58
	10	-	-	7^{th}-day trial

B2. 7TH-DAY COMPRESSIVE STRENGTH RESULTS OF EXPERIMENT 2

Mix notation	Number	Mass	Load (KN)	Compressive strength (MPa)
2N	1	8.163	485.5	21.58
	2	8.013	465.5	20.69
	3	8.103	467.1	20.76
2R25	1	8.047	444.6	19.76
	2	7.960	434.9	19.33
	3	7.883	422.7	18.79
2R50	1	7.778	407.7	18.12
	2	7.773	394.4	17.53
	3	7.861	418.5	18.60
2R75	1	7.533	362.7	16.12
	2	7.736	374.8	16.66
	3	7.571	347.4	15.44
2R100	1	7.494	370.3	16.46
	2	7.480	345.8	15.37
	3	7.548	340.9	15.15

B3. 28TH-DAY COMPRESSIVE STRENGTH RESULTS OF EXPERIMENT 2

Mix notation	Number	Mass	Load (KN)	Compressive strength (MPa)
2N	1	8.082	735.1	32.67
	2	8.202	708.5	31.49
	3	8.184	719.1	31.96
2R25	1	8.060	702.7	31.23
	2	7.970	715.3	31.79
	3	8.042	683.3	30.37
2R50	1	7.717	631.8	28.08
	2	7.664	649.3	28.86
	3	7.759	628.6	27.94
2R75	1	7.753	649.4	28.86
	2	7.712	574.9	25.55
	3	7.671	579.6	25.76
2R100	1	7.628	567.2	25.21
	2	7.670	544.3	24.19
	3	7.494	577.1	25.65

B4. 7TH-DAY COMPRESSIVE STRENGTH RESULTS OF EXPERIMENT 3

Mix notation	Number	Mass	Load (KN)	Compressive strength (MPa)
2N	1	8.077	486.9	21.64
	2	8.343	463.1	20.58
	3	8.016	458.8	20.39
2NM6	1	7.996	513.1	22.80
	2	8.037	462.1	20.54
	3	8.293	449.5	19.98
2NM12	1	7.808	566.5	25.18
	2	7.841	507.9	22.57
	3	7.979	558.4	24.82
2NM18	1	8.078	488.1	21.69
	2	8.098	489.3	21.75
	3	8.169	483.9	21.51
2NM24	1	8.039	470.7	20.92
	2	7.972	458.6	20.38
	3	8.009	459.9	20.44

B5. 28TH-DAY COMPRESSIVE STRENGTH RESULTS OF EXPERIMENT 3

Mix notation	Number	Mass	Load (KN)	Compressive strength (MPa)
2N	1	8.123	697.3	30.99
	2	8.179	715.7	31.81
	3	8.193	726.3	32.28
2NM6	1	8.165	713.6	31.72
	2	8.137	717.0	31.87
	3	8.118	719.7	31.99
2NM12	1	8.019	744.5	33.09
	2	8.082	765.3	34.01
	3	8.049	754.9	33.55
2NM18	1	8.110	692.8	30.79
	2	8.022	680.9	30.26
	3	8.168	703.9	31.28
2NM24	1	8.049	683.1	30.36
	2	8.067	671.2	29.83
	3	8.064	659.3	29.30

B6. 56TH-DAY COMPRESSIVE STRENGTH RESULTS OF EXPERIMENT 3

Mix notation	Number	Mass	Load (KN)	Compressive strength (MPa)
2N	1	8.162	842.0	37.42
	2	8.117	868.7	38.61
	3	8.186	853.9	37.95
2NM6	1	8.212	872.7	38.79
	2	8.023	844.7	37.54
	3	8.145	851.0	37.82
2NM12	1	7.903	884.6	39.31
	2	8.076	861.1	38.27
	3	8.025	839.5	37.31
2NM18	1	8.168	837.0	37.20
	2	7.954	854.1	37.96
	3	7.898	828.4	36.82
2NM24	1	8.207	862.0	38.31
	2	8.008	840.3	37.35
	3	8.146	807.8	35.90

B7. 7TH-DAY COMPRESSIVE STRENGTH RESULTS OF EXPERIMENT 4

Mix notation	Number	Mass	Load (KN)	Compressive strength (MPa)
2R25M6	1	7.773	501.4	22.29
	2	7.857	478.7	21.27
	3	7.892	508.9	22.62
2R25M12	1	7.811	466.3	20.72
	2	7.763	449.2	19.97
	3	7.840	519.8	23.10
2R25M18	1	7.808	432.9	19.24
	2	7.735	444.3	19.75
	3	7.754	438.1	19.47
2R50M6	1	7.674	474.5	21.09
	2	7.739	476.9	21.20
	3	7.791	518.8	23.06
2R50M12	1	7.730	528.4	23.49
	2	7.810	470.0	20.89
	3	7.708	497.2	22.10
2R50M18	1	7.719	469.5	20.87
	2	7.707	464.3	20.64
	3	7.777	493.0	21.91
2R75M6	1	7.689	544.7	24.21
	2	7.768	477.9	21.24
	3	7.709	495.5	22.02
2R75M12	1	7.580	469.3	20.86
	2	7.663	487.6	21.67
	3	7.632	451.9	20.09
2R75M18	1	7.693	400.7	17.81
	2	7.694	416.0	18.49
	3	7.624	447.2	19.88
2R100M6	1	7.646	484.1	21.52
	2	7.613	527.7	23.45
	3	7.618	536.8	23.86
2R100M12	1	7.595	495.7	22.03
	2	7.487	472.5	21.00
	3	7.560	477.9	21.24
2R100M18	1	7.712	450.9	20.04
	2	7.579	486.1	21.60
	3	7.620	476.9	21.20

B8. 28TH-DAY COMPRESSIVE STRENGTH RESULTS OF EXPERIMENT 4

Mix notation	Number	Mass	Load (KN)	Compressive strength (MPa)
2R25M6	1	7.856	708.6	31.49
	2	7.923	721.2	32.05
	3	7.752	715.5	31.80
2R25M12	1	7.838	702.2	31.21
	2	7.739	706.4	31.39
	3	7.802	705.1	31.34
2R25M18	1	7.821	681.4	30.28
	2	7.803	685.8	30.48
	3	7.815	682.4	30.33
2R50M6	1	7.891	708.6	31.49
	2	7.789	694.7	30.88
	3	7.893	682.1	30.32
2R50M12	1	7.761	724.2	32.19
	2	7.724	716.5	31.85
	3	7.856	714.5	31.76
2R50M18	1	7.978	706.6	31.41
	2	7.851	698.9	31.06
	3	7.894	697.0	30.98
2R75M6	1	7.871	733.6	32.60
	2	7.767	735.3	32.68
	3	7.782	739.0	32.85
2R75M12	1	7.713	702.7	31.23
	2	7.701	707.1	31.43
	3	7.728	690.0	30.67
2R75M18	1	7.674	685.8	30.48
	2	7.683	693.7	30.83
	3	7.699	696.0	30.93
2R100M6	1	7.647	762.8	33.90
	2	7.606	793.0	35.24
	3	7.612	771.5	34.29
2R100M12	1	7.815	726.2	32.27
	2	7.703	720.0	32.00
	3	7.620	703.1	31.25
2R100M18	1	7.733	717.3	31.88
	2	7.810	697.7	31.01
	3	7.628	699.7	31.10

B9. 56TH-DAY COMPRESSIVE STRENGTH RESULTS OF EXPERIMENT 4

Mix notation	Number	Mass	Load (KN)	Compressive strength (MPa)
2R25M6	1	7.908	820.5	36.47
	2	7.790	833.8	37.06
	3	7.807	822.7	36.56
2R25M12	1	7.894	768.7	34.17
	2	7.858	816.8	36.30
	3	7.739	792.5	35.22
2R25M18	1	7.822	725.4	32.24
	2	7.901	688.8	30.61
	3	7.835	701.2	31.16
2R50M6	1	7.674	837.0	37.20
	2	7.786	828.6	36.83
	3	7.746	815.3	36.23
2R50M12	1	7.725	813.8	36.17
	2	7.789	854.1	37.96
	3	7.775	826.7	36.74
2R50M18	1	7.811	765.0	34.00
	2	7.726	699.2	31.08
	3	7.819	725.9	32.26
2R75M6	1	7.798	792.2	35.21
	2	7.757	820.0	36.44
	3	7.692	846.5	37.62
2R75M12	1	7.674	779.4	34.64
	2	7.635	783.3	34.82
	3	7.637	764.8	33.99
2R75M18	1	7.672	691.0	30.71
	2	7.621	786.8	34.97
	3	7.597	714.5	31.76
2R100M6	1	7.616	776.2	34.50
	2	7.736	853.1	37.92
	3	7.683	807.3	35.88
2R100M12	1	7.651	806.4	35.84
	2	7.616	803.4	35.71
	3	7.724	788.8	35.06
2R100M18	1	7.596	712.6	31.67
	2	7.683	761.1	33.83
	3	7.631	728.6	32.38

APPENDIX C

C1. CHEMICAL PROPERTIES OF CEMENT

	GEOLOGICAL SURVEY OF ETHIOPIA	Doc.Number: GLD/F5.10.2	Version No: 1
	GEOCHEMICAL LABORATORY DIRECTORATE		Page 1 of 1
Document Title:	Complete Silicate Analysis Report	Effective date:	May, 2017

Issue Date: -22/07/2019

Customer Name:- Aron Berhanu

Request No:- GLD/RN/448/19

Report No:- GLD/TR/424/19

Sample type:-Powder

Sample Preparation: - 200 Mesh

Date Submitted: - 16/06/2019

Number of Sample:- One(1)

Analytical Result: In percent (%) Element to be determined Major Oxides & Minor Oxides

Analytical Method: $LiBO_2$ FUSION, HF attack, GRAVIMETERIC, COLORIMETRIC and AAS

Collector's code	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	TiO_2	H_2O	LOI
DC-02	22.28	6.88	3.26	60.66	1.16	<0.01	<0.01	0.04	0.11	0.22	0.73	3.26

Note: - This result represent only for the sample submitted to the laboratory.

Analysts	Checked By	Approved By	Quality Control
Yirgalem Abriham Tihitna Beletkachew Tizita Zemene Yohanis Getachew Bethelhem Tefera	Dessie Abebe	Gosa Haile	Negash Worku

Geological Survey of Ethiopia Central Geological Laboratory

C2. CHEMICAL PROPERTIES OF METAKAOLIN

	GEOLOGICAL SURVEY OF ETHIOPIA	Doc.Number: GLD/F5.10.2	Version No: 1
	GEOCHEMICAL LABORATORY DIRECTORATE		Page 1 of 1
Document Title:	Complete Silicate Analysis Report	Effective date:	May, 2017

Issue Date: -26/06/2019

Customer Name:- Aron Berhanu

Request No:- GLD/RN/391/19

Report No:- GLD/TR/353/19

Sample type:- Powder

Sample Preparation: - 200 Mesh

Date Submitted: - 05/06/2019

Number of Sample:- One (1)

Analytical Result: In percent (%) Element to be determined Major Oxides & Minor Oxides

Analytical Method: $LiBO_2$ FUSION, HF attack, GRAVIMETERIC, COLORIMETRIC and AAS

Collector's code	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	TiO_2	H_2O	LOI
MK-01	62.36	34.12	1.90	0.26	0.10	<0.01	0.28	<0.01	0.09	0.64	0.36	0.65

Note: - This result represent only for the sample submitted to the laboratory.

Analysts	Checked By	Approved By	Quality Control
Yirgalem Abriham Tihitna Beletkachew Tizita Zemene Yohanis Getachew Bethelhem Tefera	Dessie Abebe	Gosa Haile	Negash Worku

C3. MATERIAL SAFETY DATA SHEET OF METAKAOLIN

ZIGMA INTERNATIONAL
Manufacturers of Functional Fillers
E-mail:zigmacolors@vsnl.net
zigmatalc@gmail.com
Http://www.functionalfillers.com

Material Safety Data Sheet
POZZOFILZ "METAKAOLIN"

Revision date: 03/08/2017 Date Printed: 17/08/2018

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Chemical Name:	METAKAOLIN
Product Use:	Filler & Extender
Supplier:	**ZIGMA INTERNAIONAL** 18, Adoshi Road Village: Honad, Taluka: Khalapur Dist. Raigad, Maharashtra, India Tel: 22-27579004

2. COMPOSITION / INFORMATION ON INGREDIENTS

Appearance: Off White to Ivory
Boiling Point: N/A deg C
Melting Point: 2000 deg C
Vapors Pressure: N/A
Specific Gravity: 2.54
Flash Point: N/A
pH: N/A
Solubility in water: Insol g/l (25 deg C)
Flammability Limits (as percentage volume in air)
Lower Explosion Limit: N/A

3. HAZARDS IDENTIFICATION

Emergency Overview:
Color: Off White to Ivory
Form: Powder
Odor: Odorless
Flash Point, °C: Not Applicable
Most Important Hazards: Prolonged or repeated exposure may cause lung damage.
Potential Health Effects:

Inhalation:	Nuisance dust. May cause irritation of the respiratory tract.
Ingestion:	No adverse health effects are expected from swallowing.
Skin Contact:	May cause mechanical irritation.
Eye Contact:	May cause mechanical irritation.

ZIGMA INTERNATIONAL

Address : A- 411 / 412, Shiv Chambers, Sector 11, Plot No. 21, C.B.D. Belapur, Navi Mumbai 400 614.
Tel. : 022 27579020 Telefax: 022 27579004

C3. (CONTINUED)

ZIGMA INTERNATIONAL

Manufacturers of Functional Fillers

E-mail: zigmacolors@vsnl.net

zigmatalc@gmail.com

Http://www.functionalfillers.com

Carcinogenicity:

Ingredient

CAS NO #	CHEMICAL NAME
1332-58-7	KAOLIN CALCINED
14808-60-7	TOTAL QUARTZ (SiO_2)

4. FIRST AID MEASURES

Inhalation: Move person to fresh air. Aid in breathing, if necessary, and get immediate medical attention.

Ingestion: Procedures normally not needed. If large quantities are ingested, seek medical advice.

Skin Contact: Flush skin with large amounts of water. If irritation persists, get medical attention.

Eye Contact: In case of contact, immediately flush eyes with plenty of water for at least 15 minutes and get medical attention if irritation persists.

5. FIRE FIGHTING MEASURES

Flash Point, °C:	Not Applicable
Auto ignition Temperature, °C:	Not Applicable
Lower Explosive Limit, %:	Not Applicable
Upper Explosive Limit, %:	Not Applicable

Extinguishing Media: None - does not burn.
Use extinguishing media appropriate for surrounding fire.
Fire Fighting Procedures: None required; however, when fighting chemical fires, self-contained breathing apparatus and protective clothing is recommended.
Unusual Fire and Explosion Not a fire or explosion hazard.

Spill Procedures:

• Store in cool dry place.
• Material may be slippery when wet. • Avoid generating or breathing dust. • Avoid contact with eyes.
• Wash thoroughly after handling. • Use with adequate ventilation. • For industrial use only.

6. ACCIDENTAL RELEASE MEASURES

Scoop up or vacuum into a container for reclamation or disposal. Avoid dusting.

7. HANDLING AND STORAGE

Do not breathe dust. Keep away from acid & alkalis. Keep container tightly closed.

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ZIGMA

Http://www.functionalfillers.com

ZIGMA INTERNATIONAL

Manufacturers of Functional Fillers
E-mail:zigmacolors@vsnl.net
zigmatalc@gmail.com

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

SECTION V: HEALTH HAZARD INFORMATION

HAZARD BY ROUTES OF EXPOSURE:

INHALATION: WARNING: Metakaolin is not listed as a carcinogen by the International Agency for Research on Cancer (IARC), the National toxicology Program (NTP) or the Occupational Safety and Health Administration (OSHA). In October, 1996, IARC working group re-assessing crystalline silica, a component of this product, designated crystalline silica as a Group 1 carcinogenic. The NTP indicates that crystalline silica is reasonably anticipated to be a Group 2 carcinogen. These classifications are based on sufficient evidence of carcinogenicity in certain experimental animals and on selected epidemiological studies of workers exposed to crystalline silica. These studies only rarely, however, include data on smoking, potential confounding exposures, and assurance of the comparability of referent population.

INGESTION: EYE: SKIN CONTACT/ABSORPTION:
Nausea may result Inflammation of eye Inflammation from contract with open from accidental ingestion.

Overexposure

SIGNS AND SYMPTOMS OF EXPOSURE:
Short term: Shortness of breath, coughing associated with inhalation of dust.
EMERGENCY/FIRST AID PROCEDURES;
INHALATION/INGESTION: Consult physician and/or obtain competent medical assistance.
EYE CONTACT: Flush with water, consult physician and/or obtain competent medical assistance.

9. PHYSICAL AND CHEMICAL PROPERTIES

Form: Powder
Color: Off White to Ivory
Odor: Odorless
Specific Gravity: 2.54
Solubility (in water): Insoluble
Melting Point: 1710C
Boiling Point: 2230C

10. STABILITY AND REACTIVITY

Stability Data:	Stable
Conditions/Hazards to Avoid:	None anticipated
Incompatibility (Materials to Avoid):	None known.

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Address : A- 411 / 412, Shiv Chambers, Sector 11, Plot No. 21, C.B.D. Belapur, Navi Mumbai 400 614.
Tel. : 022 27579020 Telefax: 022 27579004

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Manufacturers of Functional Fillers

E-mail:zigmacolors@vsnl.net
zigmatalc@gmail.com

Http://www.functionalfillers.com

Hazardous Decomposition Products: None anticipated
Polymerization:None anticipated.

11. TOXICOLOGICAL INFORMATION

Acute toxicity:

LD/LC50 values that are relevant for classification:		
1332-58-7 natural aluminosilicate (Kaolin)		
Oral	LD50	>5000 mg/kg (rat)
Dermal	LD50	>5000 mg/kg (rat)
14808-60-7 quartz (SiO2)		
Oral	LD50	500 mg/kg (rat) IUCLID Dataset 18-Feb-2000

Primary irritant effect:
* **on the skin**: May cause irritation with dryness and abrasion.
* **on the eye**: Irritating effect.
Sensitization: No sensitizing effects known.
Additional toxicologycal information:
* The product is not subject to classification according to internally approved calculation methods for preparations. When used and handled according to specifications, the product does not have any harmful effects according to our experience and the information provided to us.

12. ECOLOGICAL INFORMATION

Information on Product:
Environmental Fate:Not hazardous to the environment.
Ecotoxicological Information: Not hazardous to the environment.

13. DISPOSAL CONSIDERATIONS

US EPA Waste Number: Not Regulated
Disposal of Waste Method: This product, if disposed as received, is a non-hazardous waste. Local disposal laws and regulations will determine the proper waste disposal/recycling/reclamation procedure. Disposal requirements are dependent on the hazard classification and will vary by location and the type of disposal selected.

14. TRANSPORT INFORMATION

International Transport Regulations:
ICAO Class: Not Regulated
IMO Class: Not Regulated

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Address : A- 411 / 412, Shiv Chambers, Sector 11, Plot No. 21, C.B.D. Belapur, Navi Mumbai 400 614.
Tel. : 022 27579020 Telefax: 022 27579004

C3. (CONTINUED)

ZIGMA
Http://www.functionalfillers.com

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Manufacturers of Functional Fillers
E-mail:zigmacolors@vsnl.net
zigmatalc@gmail.com

15. REGULATORY INFORMATION

International Inventories:

United States:	This product or its ingredients are listed on or compliant with the TSCA Inventory.
Canada:	This product or its ingredients are listed on or compliant with the DSL.
Europe:	This product or its ingredients are listed on or compliant with EINECS.
Japan:	Not Determined
Australia:	Not Determined
Korea:	This product or its ingredients are listed on or compliant with the ECL.
Philippines:	This product or its ingredients are listed on or compliant with PICCS.
China:	This product or its ingredients are listed on or compliant with the IECSC.

16. OTHER INFORMATION

Prepared By: ZIGMA INTERNATIONAL

The information in this Material Safety Data Sheet should be provided to all who will use, handle, store, transport, or otherwise be exposed to this product. This information has been prepared for the guidance of plant engineering, operations, management and for persons working with or handling this product. The information presented in the MSDS is premised upon proper handling and anticipated uses, and is for the material without chemical additions/alterations. We believe this information to be reliable and up-to-date as of the date of publication, but make no warranty of merchantability or any other warranty, express or implied, with respect to such information, and we assume no liability resulting from its use.

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Tel. : 022 27579020 Telefax: 022 27579004

C4. CHEMICAL PROPERTIES OF WHITE KAOLIN AND RED KAOLIN

	GEOLOGICAL SURVEY OF ETHIOPIA	**Doc.Number: GLD/F5.10.2**	**Version No: 1**
	GEOCHEMICAL LABORATORY DIRECTORATE		Page 1 of **1**
Document Title:	**Complete Silicate Analysis Report**	**Effective date:**	**May, 2017**

Issue Date: -26/09/2019

Customer Name:-Aron Berhanu

Request No:- GLD/RN/622/19

Report No:- GLD/TR/544/19

Sample type:- Powder

Sample Preparation: - 200 Mesh

Date Submitted: - 10/09/2019

Number of Sample:- Two(2)

Analytical Result: In percent (%) Element to be determined Major Oxides & Minor Oxides

Analytical Method: $LiBO_2$ FUSION, HF attack, GRAVIMETERIC, COLORIMETRIC and AAS

Collector's code	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	TiO_2	H_2O	LOI
RK	57.82	20.28	1.46	<0.01	<0.01	<0.01	0.96	<0.01	0.05	0.39	5.86	11.87
Wk	60.68	23.10	0.66	1.34	<0.01	<0.01	0.28	<0.01	0.07	0.51	4.55	10.22

Note: - This result represent only for the sample submitted to the laboratory.

Analysts	Checked By	Approved By	Quality Control
Yirgalem Abriham			
Tihitna Beletkachew			
Tizita Zemene	for Dessie Abebe	Gosa Haile	Negash Worku
Bethelhem Tefera			
Lidet Endeshaw			
Yohannis Getachew			

Geological Survey of Ethiopia Central Geological Laboratory

C5. SPECIFIC GRAVITY OF WHITE KAOLIN AND RED KAOLIN

Geological Survey of Ethiopia
Mineralogy & Geotechnical Laboratory Directorate
Result Form

Directorate: - Mineralogy and Geotechnical Laboratory | **Lab section: - Mineralogy** ☐ | **Physical** ☒

Client /Originator Name: - Aron Berhanu

Client Category: - Survey ☐ **Gov.** ☐ **Pvt.** ☒

File name: - 8031/19 PVT | **Area Ref: -** | **No of Samples: - 2** | **Sample No.**

Sample Type: - Kaolin | **Lab No: -**

Type of Analysis: - Specific gravity | **Preparation required: -** | **Date Submitted: - 10/09/19**

Coll.No.	Lab. No.	Pycnometer No.	m_2 Mass of picnometer in g	m_3 Mass of test solution in the picnometer without test sample in g	Q_2 Density of test solution in g/cm³	m_4 Mass of picnom.plus test sample in g	m_4-m_2 mass of test sample in g	m_5 mass of picnom. test sample and test solution in g	m_3+m_4 - m_5 volume of test sample in g/cm³	Specific Gravity in g/cm³	Average
RK	8031/19	51/51	28.578	78.4488	1 g/cm³	36.8787	8.3007	83.5303	3.2192	2.57	2.51
		20/20	27.7091	77.6683	1 g/cm³	36.6396	8.9305	82.9552	3.6436	2.45	
WK	8032/19	60/60	29.246	78.9482	1 g/cm³	38.5856	9.3396	84.6215	3.6663	2.54	2.51
		56/56	27.649	77.7618	1 g/cm³	35.3477	7.6987	82.3609	3.0996	2.48	

Described By / Analysts: -
Aregahagn Kefelegn

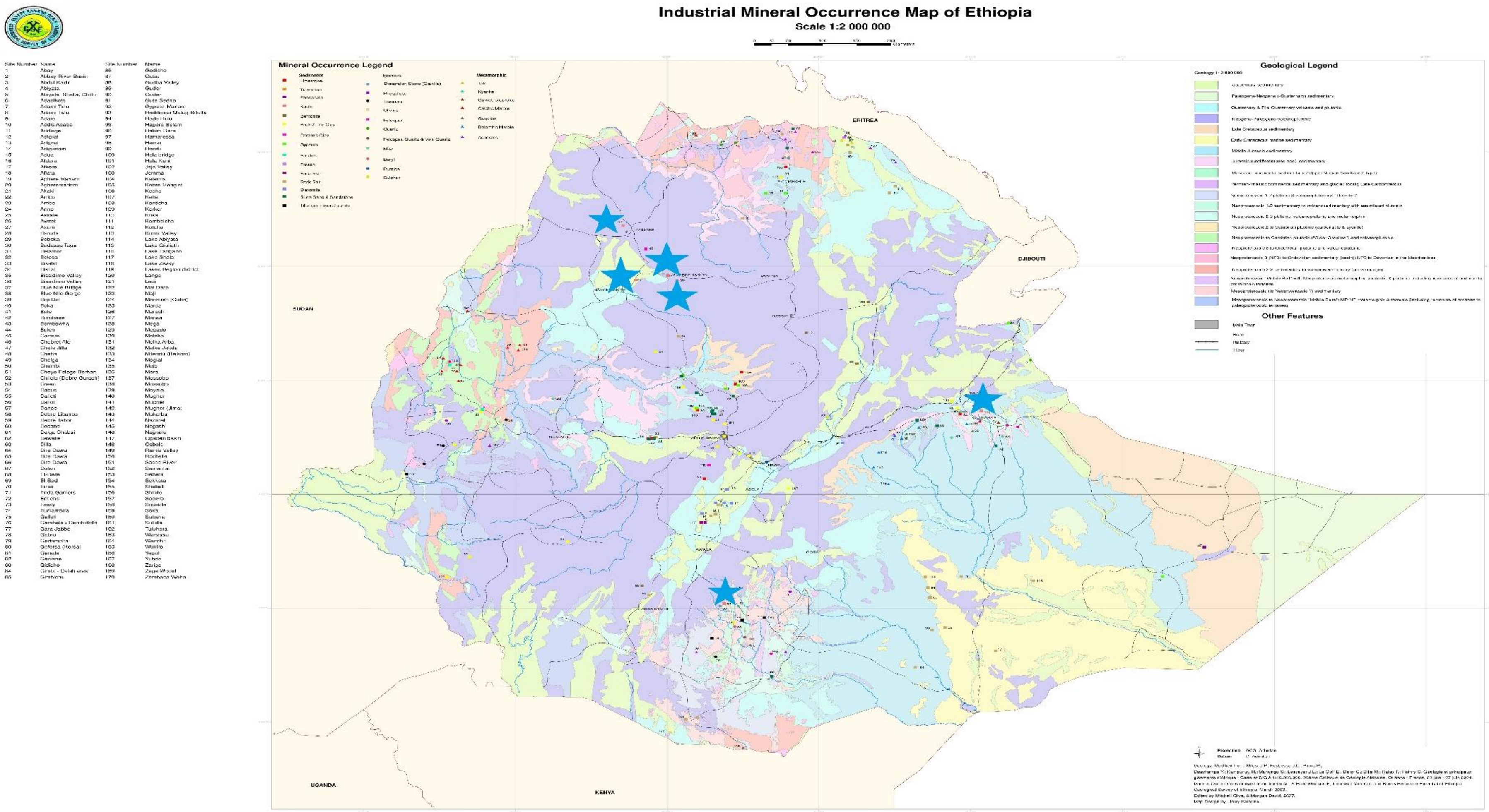
Checked by: -
Girma Asemu

Date Completed: - 26/09/19

Geological Survey of Ethiopia
Central Geological Laboratory

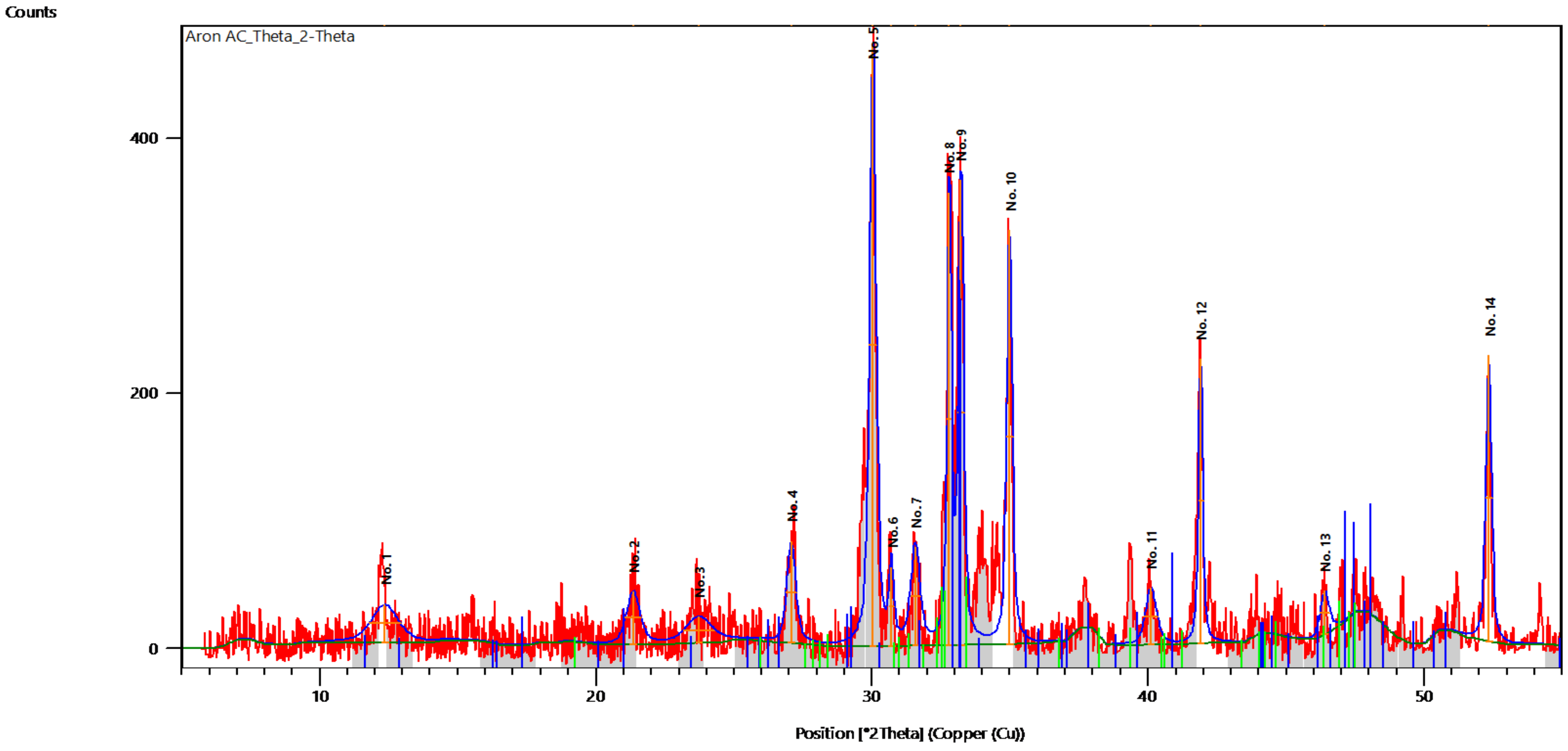
APPENDIX D

D1. INDUSTRIAL MINERAL OCCURRENCE MAP OF ETHIOPIA



APPENDIX E

E1. XRD ANALYSIS RESULT OF CEMENT



E1. (CONTINUED)

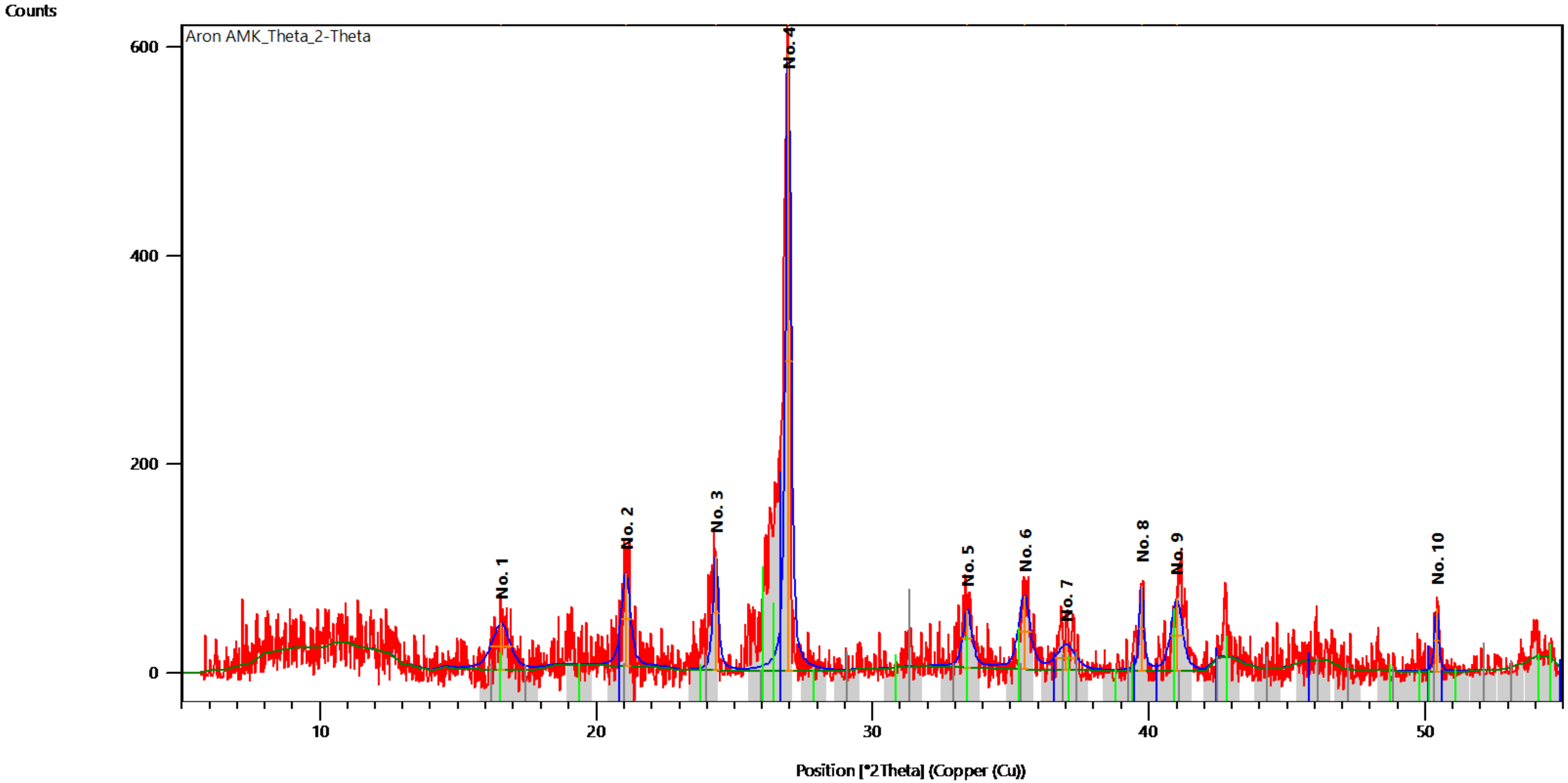
PATTERN LIST

Number	Ref. Code	Score	Compound Name	Scale Factor	Chemical Formula
1	00-032-0148	22	Calcium Aluminum Oxide	0.585	Ca3 Al2 O6
2	00-033-0303	8	Calcium Silicate	0.109	Ca2 Si O4

PEAK LIST

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
12.3507	29.59	1.1520	7.16080	6.27
21.3618	41.55	0.5760	4.15617	8.80
23.7224	20.81	1.1520	3.74765	4.40
27.0843	79.78	0.3840	3.28962	16.89
30.0388	472.34	0.2880	2.97245	100.00
30.7099	61.75	0.1920	2.90901	13.07
31.5772	76.65	0.3840	2.83106	16.23
32.8043	354.61	0.1920	2.72791	75.08
33.2281	364.48	0.2400	2.69408	77.17
34.9846	324.55	0.2400	2.56273	68.71
40.1137	44.83	0.4800	2.24608	9.49
41.9071	222.71	0.1920	2.15401	47.15
46.4086	33.89	0.3840	1.95502	7.17
52.3381	223.89	0.2400	1.74663	47.40

E2. XRD ANALYSIS RESULT OF METAKAOLIN



E2. (CONTINUED)

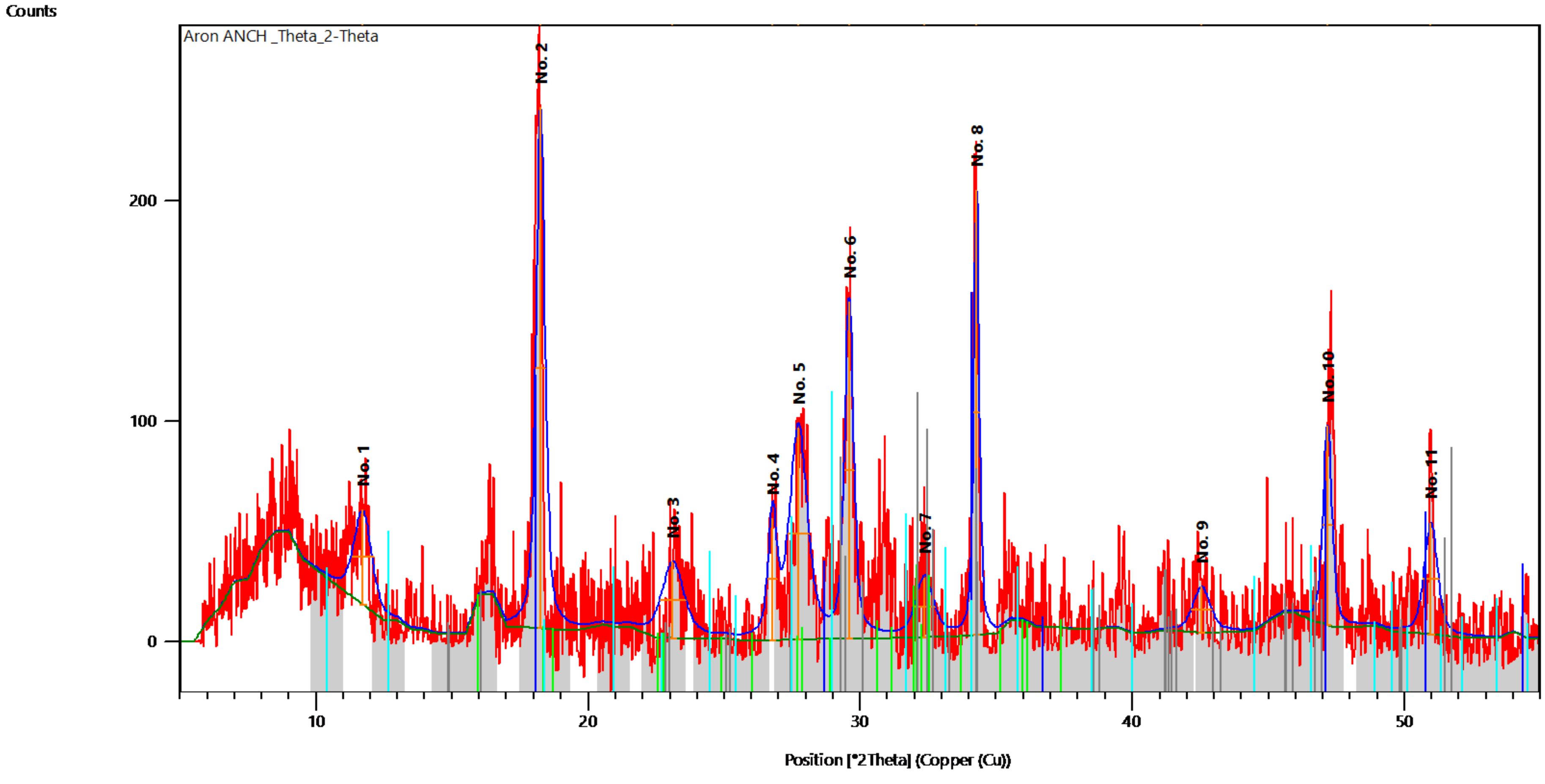
PATTERN LIST

Number	Ref. Code	Score	Compound Name	Scale Factor	Chemical Formula
1	00-046-1045	26	Silicon Oxide	0.307	Si O2
2	00-022-0018	19	Aluminum Silicate	0.160	Al2 Si O5
3	00-020-0199	2	Calcium Aluminum Silicate	0.119	Ca2 Al2 Si O7

PEAK LIST

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
16.5473	44.65	0.7680	5.35299	7.51
21.0744	89.40	0.3840	4.21218	15.03
24.3229	109.52	0.2400	3.65647	18.42
26.9510	594.72	0.2400	3.30559	100.00
33.4396	55.01	0.3840	2.67752	9.25
35.4926	70.38	0.4800	2.52720	11.83
37.0054	23.90	0.7680	2.42729	4.02
39.7341	81.11	0.1920	2.26666	13.64
41.0099	69.42	0.4800	2.19904	11.67
50.4169	59.86	0.1920	1.80858	10.06

E3. XRD ANALYSIS RESULT OF CONTROL NATURAL AGGREGATE CONCRETE (2N)



E3. (CONTINUED)

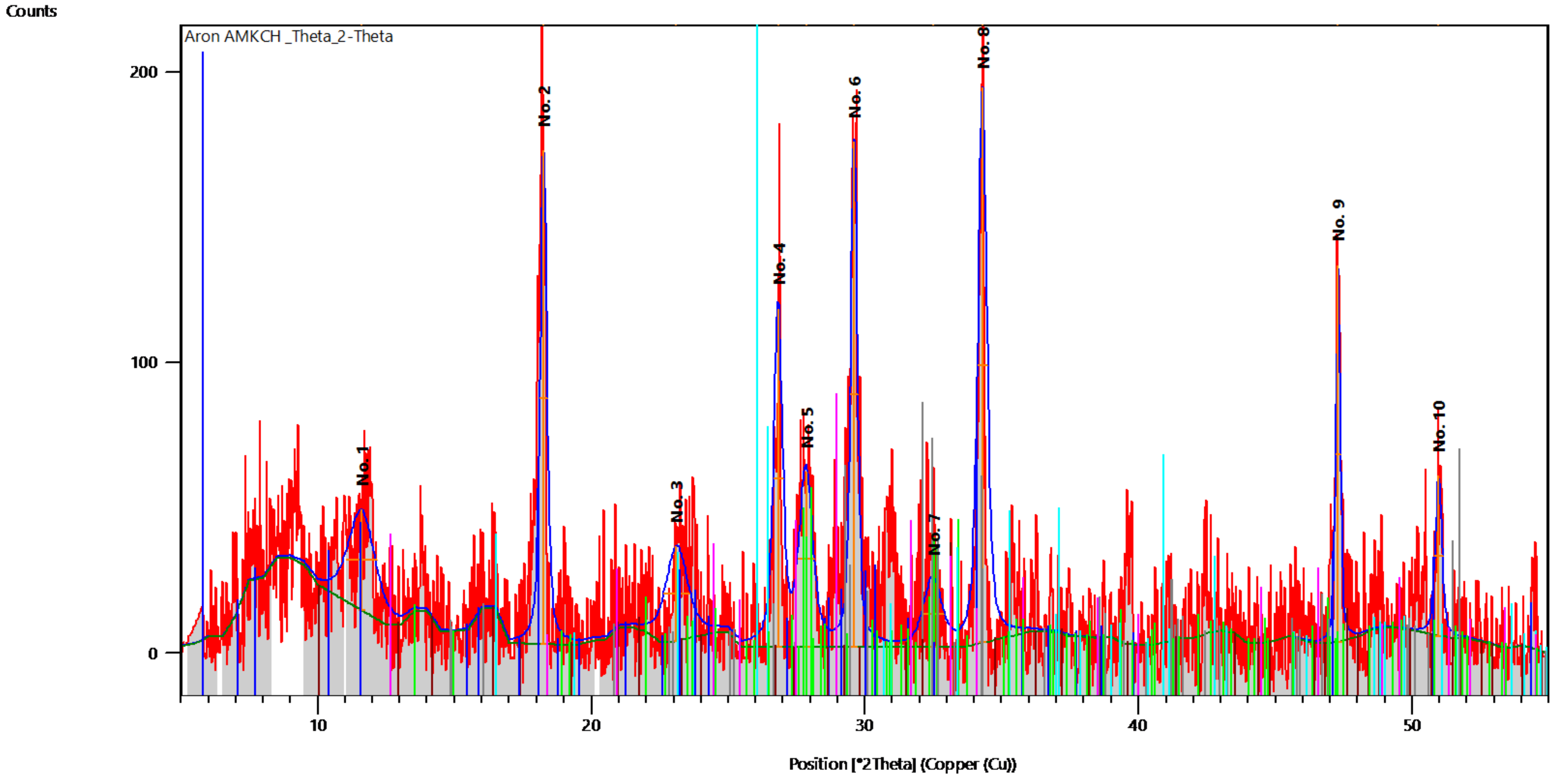
PATTERN LIST

Number	Ref. Code	Score	Compound Name	Scale Factor	Chemical Formula
1	00-004-0733	33	Calcium Hydroxide	0.554	Ca (OH)2
2	00-029-0369	12	Calcium Silicate	0.118	Ca2 Si O4
3	00-049-0442	12	Calcium Silicate	0.396	Ca3 Si O5
4	00-023-0125	2	Calcium Silicate Hydrate	0.400	Ca6 Si6 O17 (OH)2

PEAK LIST

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
11.7245	43.12	0.7680	7.54184	18.23
18.2531	236.60	0.3840	4.85638	100.00
23.1110	34.33	0.9600	3.84539	14.51
26.7809	55.16	0.3840	3.32620	23.31
27.7364	96.35	0.7680	3.21374	40.72
29.5900	152.74	0.3840	3.01650	64.56
32.3659	26.91	0.7680	2.76385	11.38
34.2772	201.88	0.2400	2.61398	85.32
42.5469	20.85	0.7680	2.12309	8.81
47.1799	90.46	0.3840	1.92484	38.23
50.9802	50.90	0.5760	1.78991	21.51

E4. XRD ANALYSIS RESULT OF NATURAL AGGREGATE CONCRETE WITH 12% METAKAOLIN (2NM12)



E4. (CONTINUED)

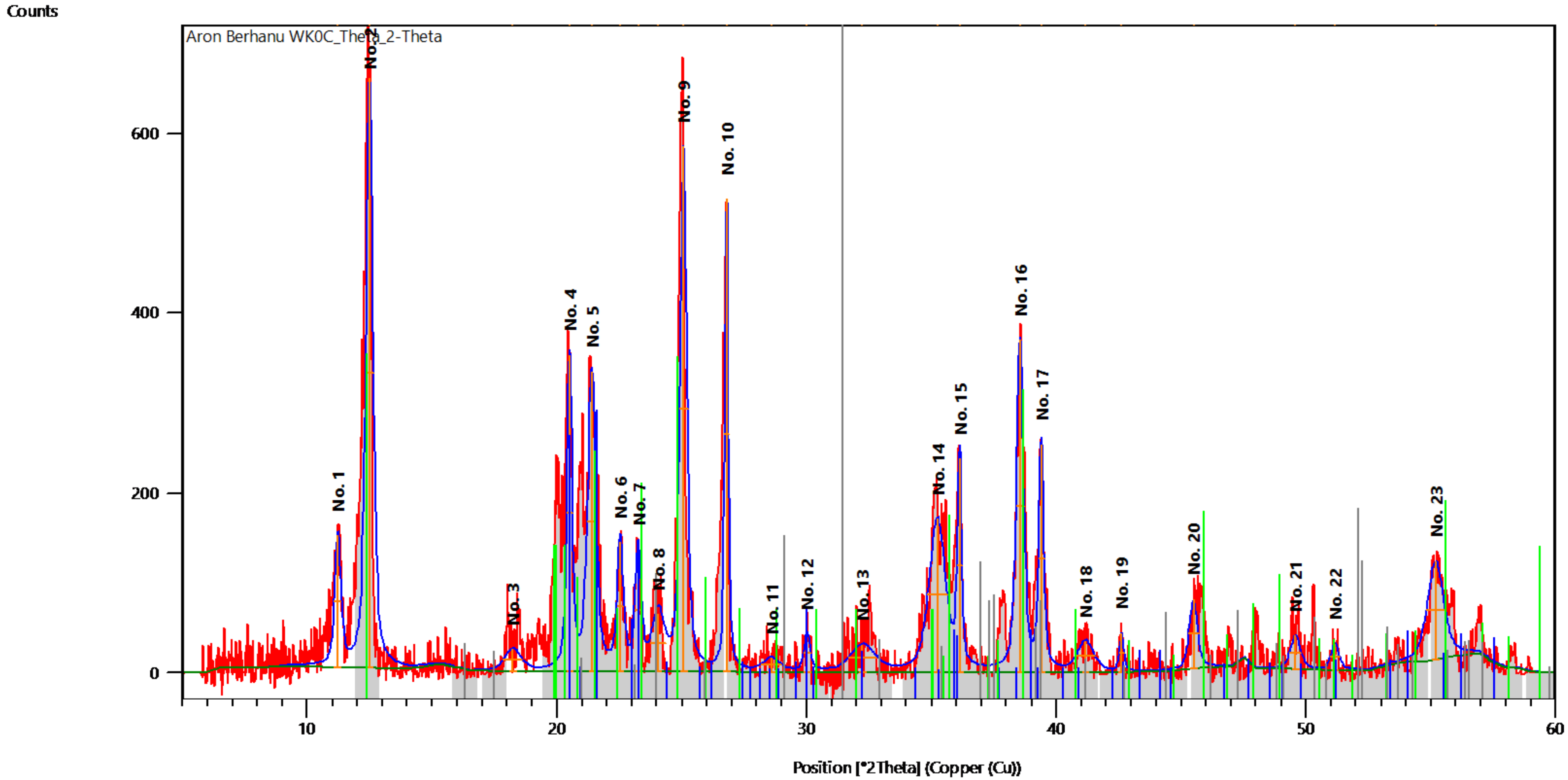
PATTERN LIST

Number	Ref. Code	Score	Compound Name	Scale Factor	Chemical Formula
1	00-004-0733	26	Calcium Hydroxide	0.345	Ca (OH)2
2	00-033-0303	6	Calcium Silicate	0.205	Ca2 Si O4
3	00-049-0442	13	Calcium Silicate	0.390	Ca3 Si O5
4	00-038-1429	2	Calcium Aluminum Oxide	0.168	Ca3 Al2 O6
5	00-038-0471	1	Aluminum Silicate	2.041	Al2 Si O5
6	00-023-0125	1	Calcium Silicate Hydrate	0.402	Ca6 Si6 O17 (OH)2
7	00-046-1405	2	Calcium Aluminum Silicate Hydrate	0.932	Ca Al2 Si3 O10 !6 H2O
8	00-041-1486	19	Calcium Aluminum Silicate	0.253	Ca Al2 Si2 O8
9	00-046-1045	12	Silicon Oxide	0.071	Si O2

PEAK LIST

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
11.6109	34.71	1.1520	7.61533	18.18
18.2561	169.73	0.2880	4.85560	88.90
23.1172	32.52	0.9600	3.84439	17.04
26.8260	116.47	0.3840	3.32071	61.00
27.8481	60.44	0.5760	3.20110	31.66
29.6021	173.88	0.2880	3.01530	91.08
32.4775	23.50	0.7680	2.75460	12.31
34.2995	190.92	0.3840	2.61233	100.00
47.2961	129.52	0.1920	1.92038	67.84
50.9699	54.87	0.2880	1.79025	28.74

E5. XRD ANALYSIS RESULT OF WHITE KAOLIN WITHOUT THERMAL TREATMENT



E5. (CONTINUED)

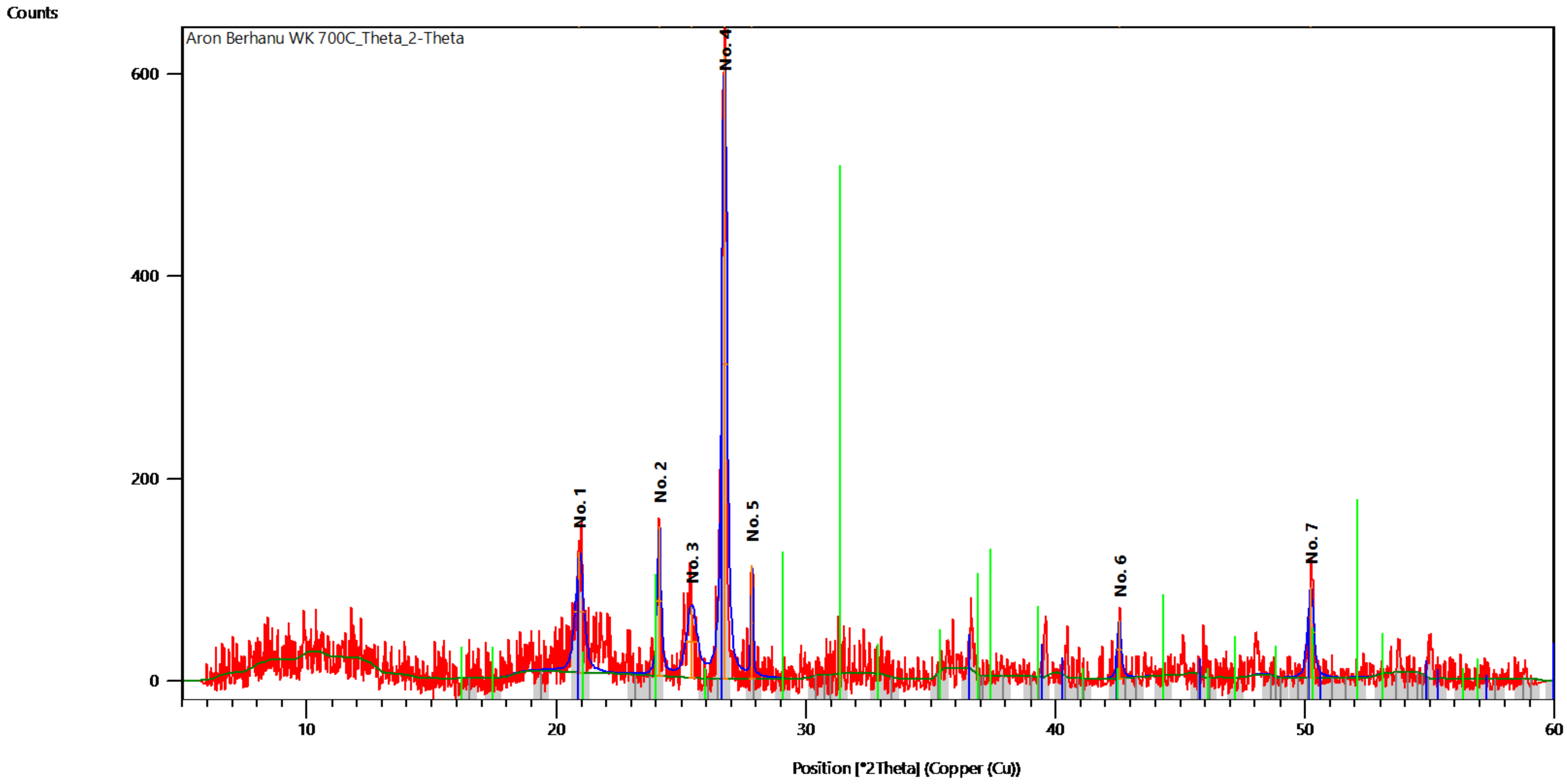
PATTERN LIST

Number	Ref. Code	Score	Compound Name	Scale Factor	Chemical Formula
1	00-018-1170	37	Silicon Oxide	0.403	Si O2
2	00-010-0446	31	Aluminum Silicate Hydroxide	0.485	Al2 Si2 O5 (OH)4
3	00-035-0755	4	Calcium Aluminum Silicate	1.175	Ca2 Al2 Si O7

PEAK LIST

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
11.2269	146.10	0.2880	7.87493	22.32
12.4911	654.68	0.3360	7.08065	100.00
18.2174	24.78	0.7680	4.86583	3.79
20.5056	351.27	0.2400	4.32773	53.66
21.3958	332.46	0.3840	4.14963	50.78
22.5352	143.68	0.2880	3.94233	21.95
23.2548	136.12	0.2880	3.82195	20.79
24.0596	63.73	0.5760	3.69589	9.73
25.0675	583.45	0.3120	3.54953	89.12
26.7940	525.72	0.1680	3.32460	80.30
28.5809	15.19	0.7680	3.12067	2.32
30.0214	42.42	0.2880	2.97413	6.48
32.2371	30.24	1.1520	2.77459	4.62
35.2368	169.81	0.7680	2.54496	25.94
36.1219	236.86	0.1920	2.48461	36.18
38.5433	368.83	0.3360	2.33391	56.34
39.3990	252.59	0.2400	2.28516	38.58
41.1694	34.01	0.7680	2.19089	5.19
42.6171	42.65	0.1920	2.11976	6.51
45.4896	77.71	0.3840	1.99236	11.87
49.5787	37.51	0.4800	1.83718	5.73
51.1628	29.54	0.3840	1.78395	4.51
55.1921	109.49	0.7680	1.66287	16.72

E6. XRD ANALYSIS RESULT OF WHITE KAOLIN THERMALLY TREATED AT 700 ^{0}C FOR 90 MINUTES



E6. (CONTINUED)

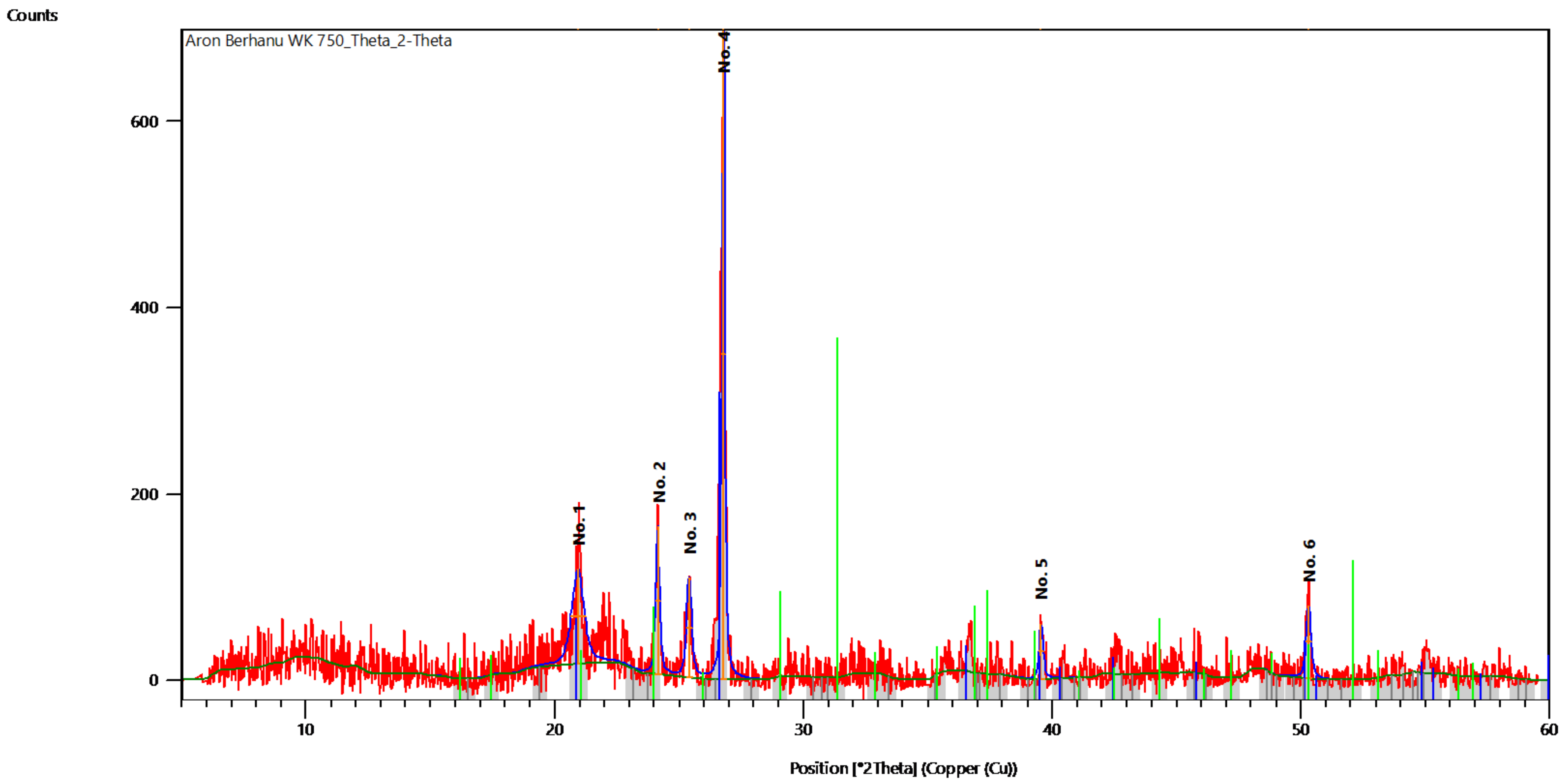
PATTERN LIST

Number	Ref. Code	Score	Compound Name	Scale Factor	Chemical Formula
1	00-033-1161	43	Silicon Oxide	0.639	Si O2
2	00-020-0199	1	Calcium Aluminum Silicate	0.776	Ca2 Al2 Si O7
3	00-038-0471	-	Aluminum Silicate	0.000	Al2 Si O5

PEAK LIST

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
20.9255	118.79	0.3840	4.24183	19.14
24.1147	147.94	0.1920	3.68757	23.83
25.4183	69.31	0.4800	3.50133	11.17
26.7256	620.72	0.2400	3.33295	100.00
27.8330	111.74	0.0960	3.20281	18.00
42.5534	57.10	0.1920	2.12278	9.20
50.2232	89.47	0.2880	1.81510	14.41

E7. XRD ANALYSIS RESULT OF WHITE KAOLIN THERMALLY TREATED AT 750 ^{0}C FOR 45 MINUTES



E7. (CONTINUED)

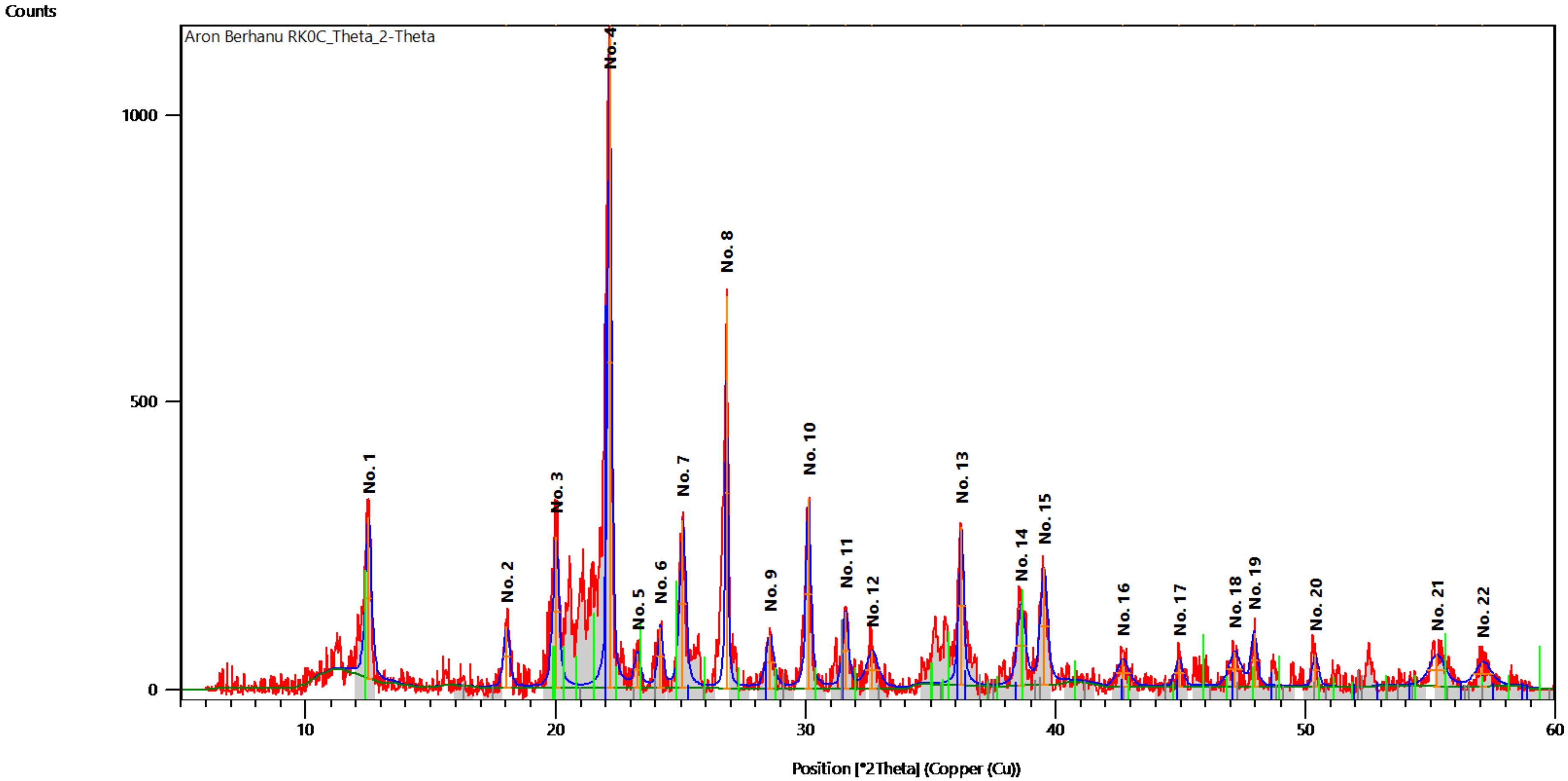
PATTERN LIST

Number	Ref. Code	Score	Compound Name	Scale Factor	Chemical Formula
1	00-046-1045	30	Silicon Oxide	0.441	Si O2
2	00-020-0199	1	Calcium Aluminum Silicate	0.519	Ca2 Al2 Si O7
3	00-038-0471	-	Aluminum Silicate	0.000	Al2 Si O5

PEAK LIST

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
20.9306	101.79	0.5760	4.24080	14.57
24.1385	158.86	0.1920	3.68399	22.74
25.3961	106.70	0.2400	3.50434	15.28
26.7810	698.44	0.1440	3.32619	100.00
39.5578	59.28	0.1920	2.27635	8.49
50.3076	77.71	0.2400	1.81226	11.13

E8. XRD ANALYSIS RESULT OF RED KAOLIN WITHOUT THERMAL TREATMENT



E8. (CONTINUED)

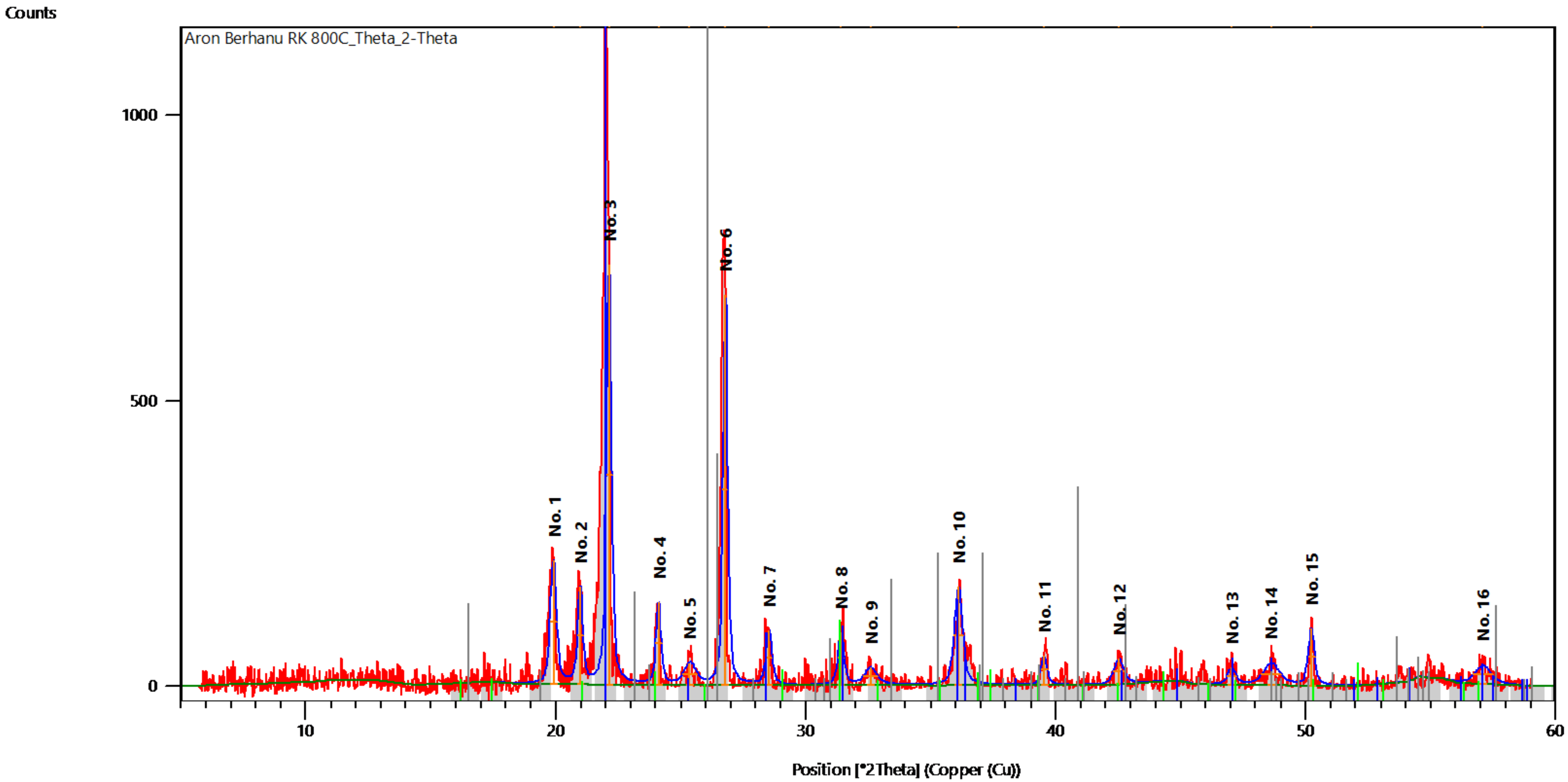
PATTERN LIST

Number	Ref. Code	Score	Compound Name	Scale Factor	Chemical Formula
1	00-039-1425	42	Silicon Oxide	0.576	Si O2
2	00-010-0446	25	Aluminum Silicate Hydroxide	0.161	Al2 Si2 O5 (OH)4
3	00-035-0755	7	Calcium Aluminum Silicate	0.102	Ca2 Al2 Si O7

PEAK LIST

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
12.5087	280.11	0.2880	7.07072	24.72
18.0307	107.08	0.2880	4.91579	9.45
20.0027	262.75	0.2880	4.43537	23.19
22.1539	1132.91	0.1920	4.00932	100.00
23.2674	59.16	0.2880	3.81991	5.22
24.1858	106.21	0.2400	3.67689	9.38
25.0803	292.90	0.2880	3.54774	25.85
26.8331	683.15	0.1200	3.31984	60.30
28.5630	91.50	0.3840	3.12259	8.08
30.1081	330.61	0.2400	2.96577	29.18
31.5953	132.41	0.2880	2.82948	11.69
32.6628	64.21	0.5760	2.73940	5.67
36.2222	276.59	0.2400	2.47795	24.41
38.6236	140.58	0.3840	2.32924	12.41
39.5300	202.63	0.3360	2.27789	17.89
42.6935	48.72	0.5760	2.11614	4.30
44.9336	48.17	0.3840	2.01571	4.25
47.1682	61.27	0.5760	1.92529	5.41
47.9414	94.85	0.2880	1.89603	8.37
50.3914	57.38	0.2880	1.80944	5.06
55.2449	55.46	0.7680	1.66141	4.90
57.0988	42.75	0.7680	1.61179	3.77

E9. XRD ANALYSIS RESULT OF RED KAOLIN THERMALLY TREATED AT 800 ^{0}C FOR 135 MINUTES



E9. (CONTINUED)

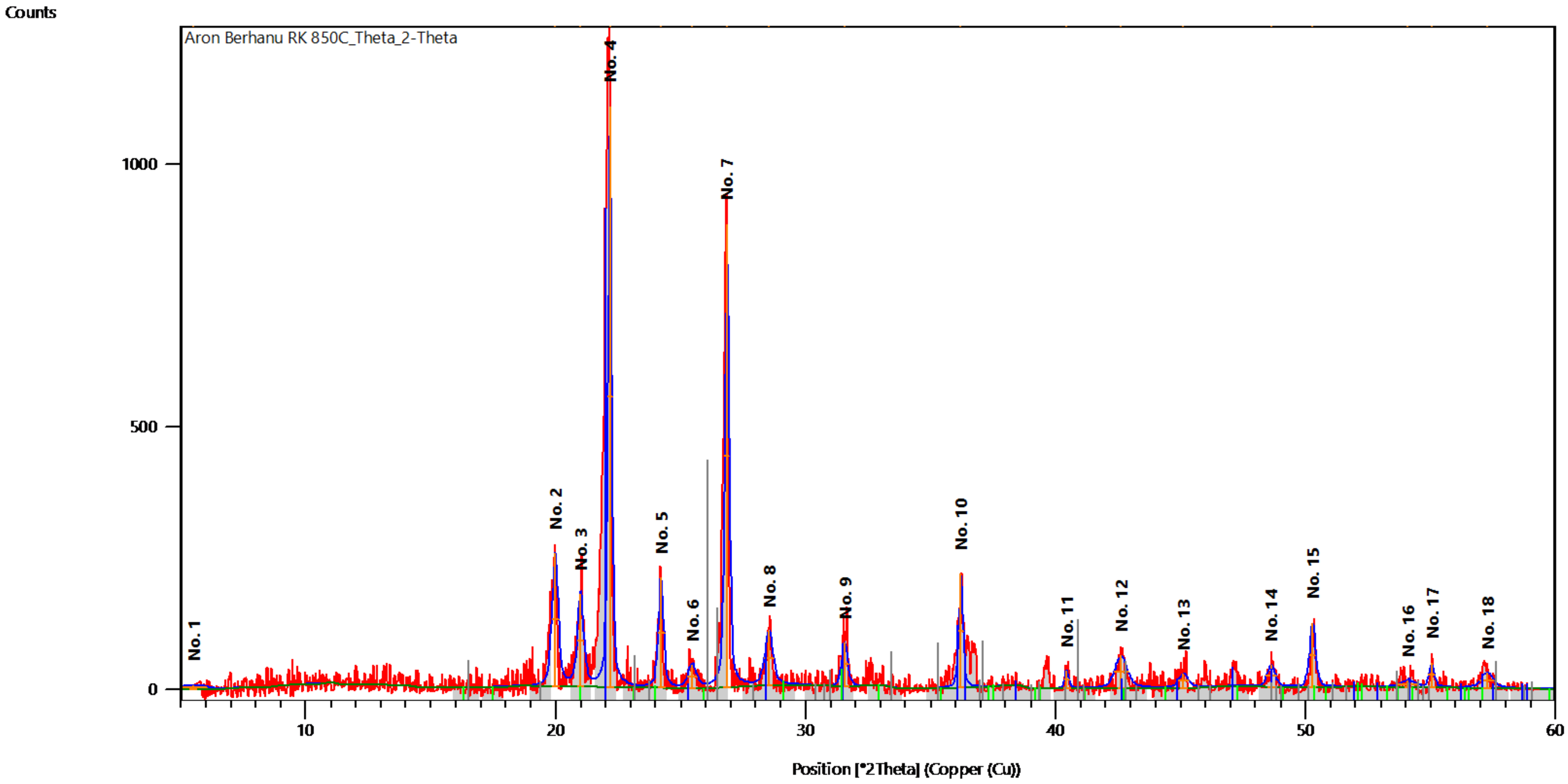
PATTERN LIST

Number	Ref. Code	Score	Compound Name	Scale Factor	Chemical Formula
1	00-039-1425	57	Silicon Oxide	0.997	Si O2
2	00-020-0199	10	Calcium Aluminum Silicate	0.097	Ca2 Al2 Si O7
3	00-038-0471	1	Aluminum Silicate	1.402	Al2 Si O5

PEAK LIST

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
19.9001	216.16	0.2880	4.45801	29.42
20.9765	169.94	0.2400	4.23163	23.13
22.1215	734.84	0.2400	4.01511	100.00
24.1103	144.30	0.2400	3.68823	19.64
25.3582	38.40	0.5760	3.50949	5.23
26.7829	683.24	0.2160	3.32595	92.98
28.5282	94.44	0.2880	3.12631	12.85
31.4083	91.78	0.2880	2.84590	12.49
32.5850	30.42	0.5760	2.74576	4.14
36.1140	171.52	0.3840	2.48513	23.34
39.5296	50.69	0.2880	2.27792	6.90
42.5248	44.29	0.4800	2.12414	6.03
47.0408	29.87	0.3840	1.93021	4.07
48.6262	38.61	0.7680	1.87092	5.25
50.2230	100.11	0.2880	1.81511	13.62
57.0827	32.79	0.7680	1.61220	4.46

E10. XRD ANALYSIS RESULT OF RED KAOLIN THERMALLY TREATED AT 850 ^{0}C FOR 135 MINUTES



E10. (CONTINUED)

PATTERN LIST

Number	Ref. Code	Score	Compound Name	Scale Factor	Chemical Formula
1	00-039-1425	49	Silicon Oxide	0.722	Si O2
2	00-035-0755	4	Calcium Aluminum Silicate	0.027	Ca2 Al2 Si O7
3	00-038-0471	1	Aluminum Silicate	0.344	Al2 Si O5

PEAK LIST

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
5.4935	8.33	1.1520	16.07411	0.75
19.9726	254.02	0.2880	4.44201	22.97
20.9916	177.41	0.2880	4.22862	16.04
22.1612	1105.73	0.1680	4.00802	100.00
24.2044	209.71	0.2400	3.67411	18.97
25.4524	44.76	0.3840	3.49672	4.05
26.8497	881.76	0.1920	3.31782	79.75
28.5304	103.34	0.3840	3.12608	9.35
31.5598	83.64	0.2880	2.83258	7.56
36.2065	216.54	0.1920	2.47900	19.58
40.4420	34.32	0.1920	2.22860	3.10
42.6146	62.45	0.5760	2.11988	5.65
45.1031	26.92	0.4800	2.00852	2.44
48.6212	42.65	0.3840	1.87110	3.86
50.2770	123.35	0.2880	1.81329	11.16
54.0611	12.77	0.7680	1.69496	1.15
55.0438	46.92	0.2400	1.66700	4.24
57.2514	30.46	0.5760	1.60785	2.75