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FEASIBILITY OF METAKAOLIN AND LIME MATERIAL AS A BINDER MIXTURE FOR CONCRETE THE CASE OF BALENGOU CLAY IN WEST CAMEROON AND THE LIME OF FIGUIL NORTHERN CAMEROON

Pr. GOUAFO Casimir
University of Dschang (Cameroon)
Pr. DEFO Célestin
University of d'Ebolowa (Cameroon)
Pr. NDONGO Barthélémy
University of Dschang (Cameroon)
Dr. KOUGOUM GOUAFO Géraldine
University of Dschang (Cameroon)
M. ZOYEM GOUAFO MATHURIN
University of Dschang (Cameroon)

Abstract: Diverse complexities and challenges are faced during cement production processes especially the Portland cement. The process requires huge amounts of energy and these fossils fuels when burnt at up to 1450°C released excessive greenhouse gases such as CO_2 which contributes to the calamity called global warming. All these facts, in addition to the low economic growth of countrys, and the expensive price of construction materials, reduce the availability of a liveable, ecological and sustainable shelter house for the middle classes. It is in this line that we thought of valorising the calcined clay from Balengou by studying the feasibility of making a binder by mixing it with lime from Figuil as this could help to partially cover some domains done by Portland cement, to improve the lime mortar properties, to reduce the construction cost, and to reduce the degradation of the ozone layer. In this work, Hallosysite clay from Balengou was calcined at 750°C, mixed with lime and the effects of it at metakaolin percentage substitution to lime (10 %, 15 %, 20 %, 25% to 90 %.) were examined on their physico-mechanical properties such as Consistency, water absorption, pozzolanicity etc. In the lime-metakaolin mixture with metakaolin from Balengou's clay, as we increase the percentage of metakaolinin the paste, the setting duration reduces. The proportion of pure lime improves the plasticity of the paste while the metakaolin at its optimum percentage (20%) increases the strength of the paste to its maximum value at about 28 days. The low energy consumption during the manufacturing process and the CO_2 absorption during the carbonation process reduce the environmental impact and cost of lime-metakaolin mixture. Also, the aesthetic appearance of that paste makes it adequate for internal pointing and plastering. At 20% of metakaolin in the paste, the pozzolanicity is at the highest point and the paste gains good plasticity and perspiration with optimum resistance. Lime-metakaolin mixture is good for works where the need of breathability and lower strength is of great importance. Then, we do not recommend it for works with high strength structural elements.

Key words: metakaolin, pozzolanicity, Balengou clay, Figuil, lime-metakaolin, binder
Mots clés : métakaolin, pouzzolanicité, argile de Balengou, Figuil, chaux-métakaolin, liant

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1. INTRODUCTION

For centuries now, Lime was used in both mortars and concrete until it was replacing by Portland cement, due to faster hardening, higher strength and apparent longevity. However, for the production of cement, massive extraction of limestone is done in quarries so much so that it gradually become scarce and consequently expensive. Also, cement has a considerable environment impact due the fact that its manufacturing contributes to greenhouse gases both directly through production of CO_2 when calcium carbonate is thermally (900°C) decomposed, producing lime and Carbone dioxide, and also through the use of energy, particularly from the use of fossil fuels (from 900°C to 1450°C for the combination of lime, alumina and Silica).

Looking at that environmental impact of cement (creating up to 5% of worldwide emission of CO_2) and also it non breathable characteristic for walls and slabs, many researchers nowadays, are coming back to the use of lime for Ecological construction. Indeed, Lime is the most sustainable binder due to lower production energy needed, lower CO_2 during emission and CO_2 absorption by carbonation. Lime mortar is softer than cement mortar, allowing brickwork a certain degree of flexibility. It is considered breathable in that it will allow moisture to freely move through and evaporate from the surface; allows moisture to escape through evaporation and keeps the wall dry. The only problem with lime mortar is its strength which is not high enough.

However, the addition of fly ash, calcined clays or any material with natural or artificial pozzalanic properties, has the ability to improve the strength and lifelong of binders made up of lime **(Mertens et *al.*, 2009),** particularly the addition in binder of calcined clay (with a high content in SiO_2 and Al_2O_3) at 750°C with an appropriate percentage has an upswing in the construction field. Hallosite clay from balengou is calcined at 750°C (Métakaolin). The feasibility of a binder made up of lime and metakaolin at different percentage were examined on the pozzolanicity, consistency, setting time, compressive strength, water absorption, flexibility, aesthetic and ecological properties such as the fight against humidity and its breathable conditions for civil engineering structures.

2. STATEMENT OF THE PROBLEM

As the consumption of cement undergoes an annual increase of 7.9% in Africa in year 2016 (Global cement report) with 2.8 million tons of cement per year in Cameroon **(Nimpa, 2016),** the growing population will be always in need for shelter constructions. Looking at the damages caused by global warming such as floods, climate change, skin cancers and heart illness, there is an urgent need to come out with ecological materials for construction. People are now called to choose construction materials not only focused on the resistance (for the project owner) and the cost (for the middle class); but also on their ecological aspect in order to respect our ecosystems. The main problem that we are facing is to come out with construction materials which are ecologic, resistant and economic.

3. PURPOSES OF THE STUDY

The main objective of this work is to study the feasibility of a binder made up lime and metakaolin. Specifically, this will consist of evaluating the physico-mechanical properties of lime mortar by addition of metakaolin from Balengou hallosyte at different percentages in the lime from Figuil, studying the ecological behavior of that new binder within the house and its effect on the environment, making some recommendation in a bid to delimitated the scope that new binder can cover in Civil Engineering projects for sustainable, ecologic and economic constructions.

4. SIGNIFICANCE OF THE STUDY

Pozzalanic materials such as fly ash, calcined clay are known for their strength and long life term improvement (Pandey, 2003). And for centuries, lime has been always known for its good ecological effect on the environment through carbonation. Thus, studying the possible combination of lime and clay, could be helpful in valorizing local materials as the kaolinite clay from Balengou and Lime from Figuil. This will also help us reducing the effect of global warming caused by Portland cement manufacturing. To reduction the amount of cement produced due to its partial replacement with lime-metakaolin mixture. This obviously lead to the reduction of construction cost and to low impact on the environment.

5. VARIABLES

- The calcination temperature of hallosyte clays from Balengou; 750°C
- The mass percentage of S_{750} in the blended mixture 10%,15%, 20%, 25%, 35%,50%, 65%, 75%, 80%, 85%
- CO_2 emission, CO_2 absorption, heat effects (dependent variables)

6. Lime as Building Construction Materials

Lime used in building materials is broadly classified as "pure", "hydraulic", and "poor" lime; can be natural or artificial; and may be further identified by its magnesium content such as dolomitic or magnesium lime. Uses include lime mortar, lime plaster, lime render, lime-ash floors, tabby concrete, whitewash, silicate mineral paint, and limestone blocks which may be of many types. The qualities of the many types of processed lime affect how they are used. The Romans used two types of lime mortar to make Roman concrete, which allowed them to revolutionize architecture, sometimes called the Concrete revolution.

7. Clay Definition

Clays are defined according to the state of knowledge at the time and their uses. As universal materials they cannot be limited to only one definition. The Word "clay", a generic universal name is that of

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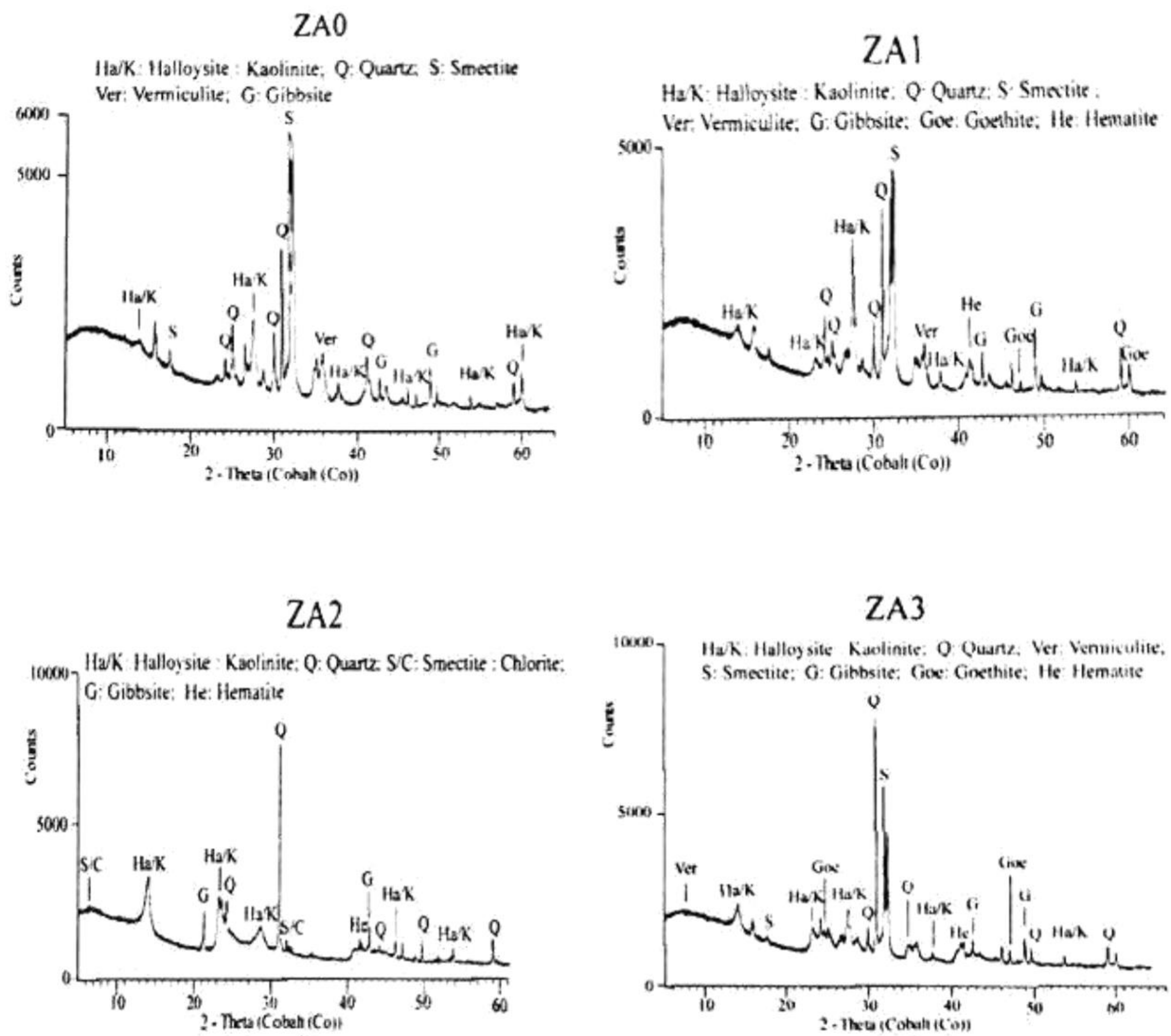

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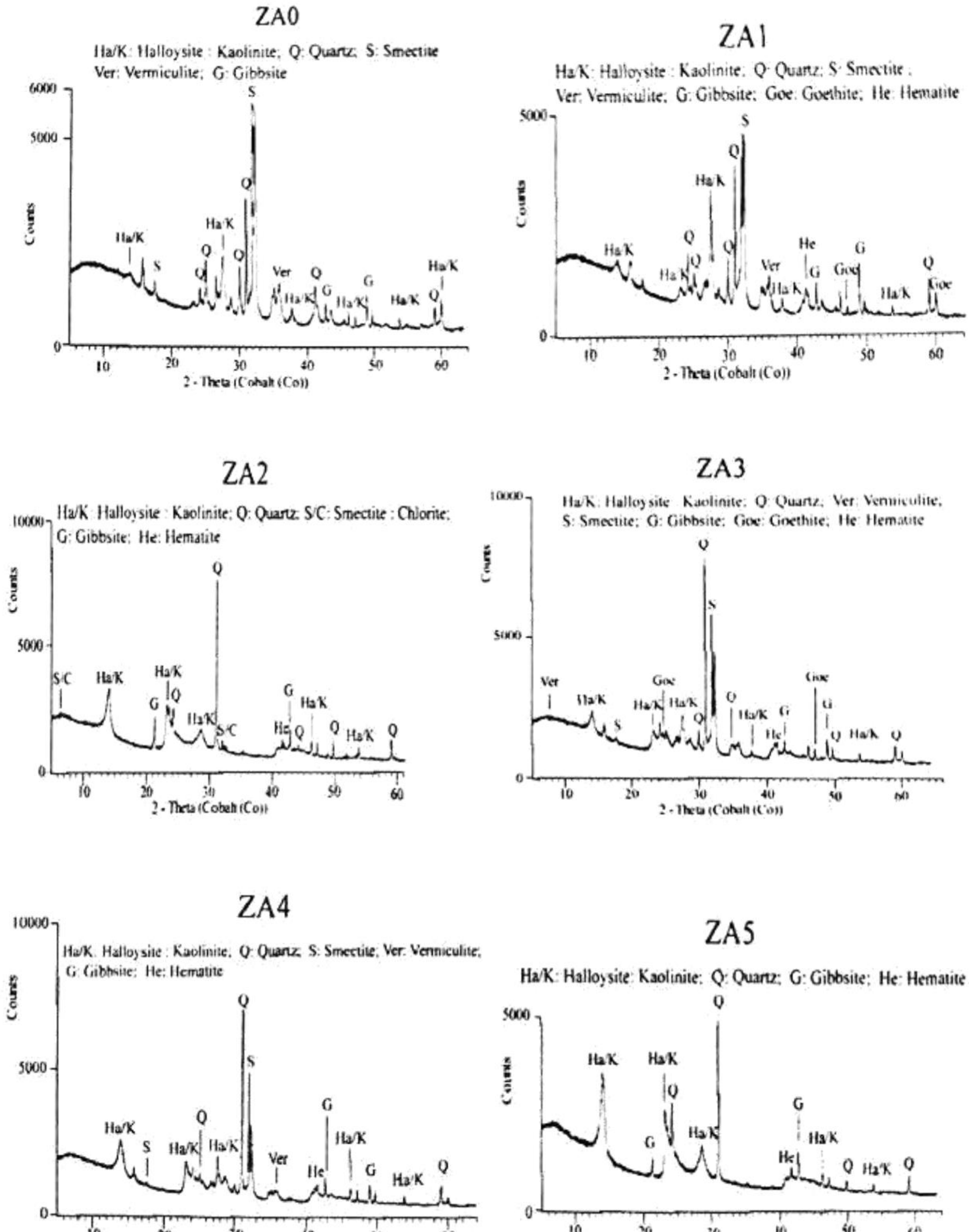

Figure 1: X-ray diffraction results for the Hallosite clay from Balengou (Zangue, 2016)

b) Chemical composition

Hallosite is a naturally occurring clay with kaolinite being the main component having a chemical composition of $Al_2Si_2O_5(OH)_4$. Halloysite is when heated at temperatures between 100 and 200 there is loss of interlayer water to produce metahallosite. This loss of mass explains the procedure of pre-dehydration and the formation of metahalloysite **(Minato and Aoki, 1979)** according to the equation.

$$\mathbf{Al_2Si_2O_5(OH)_42H_2O \rightarrow Al_2Si_2O_5(OH)_4 + 2H_2O}$$

Halloysite metahalloysite + water

It has a layered silicate structure, where oxygen atoms links in an octahedral sheet of alumina with a tetrahedral sheet of silica. When metahalloysite is calcined at elevated temperatures between 450°C and 750°C, dehydroxylation occurs which collapses the crystal structure to produce amorphous metakaolin ($Al_2Si_2O_7$), according the folloxing proposed reaction scheme:

$$\mathbf{Al_2Si_2O_5(OH)_4 \rightarrow Al_2Si_2O_7 + 2H_2O\uparrow}$$

Metahalloysite metakaolin+ water

The thermal transformation of metakaolin above 1000°C first produces either a Al-Si spinel phase or a aluminosilicate phase which transform into mullite and above 1150°C cristobalite (c-SiO_2) is formed. The thermal transformation of kaolin has been the subject of numerous studies, which reported on how the different heating conditions influence the dehydroxylation process. At temperature below 400°C, a dehydroxylation process takes place, upon cooling the hydroxle peaks reappear in the infrared spectra. Above temperature of 400°C irreversible moss of hydroxyls groups occur. The temperature at which the highest rate of dehydroxylation occurs is dependant on the original structural state of the kaolin, the particle size, density of packing, the water pressure.

- **DTA curves for kaolinic clays**

Differential thermal Analysis for halloysite type clay minerals shows that they are characterized by a sharp endothermic reaction between 500°C and 600°C and by a very abrupt exorthermic reaction at about 950°C. hydratedhalloysite shows an additional sharp endothermic reaction about 100°c and 200°C. the following diagram emanates from the works of **(Tchamo, 2015),** representing the DTA curves of kaolin and halloysite.

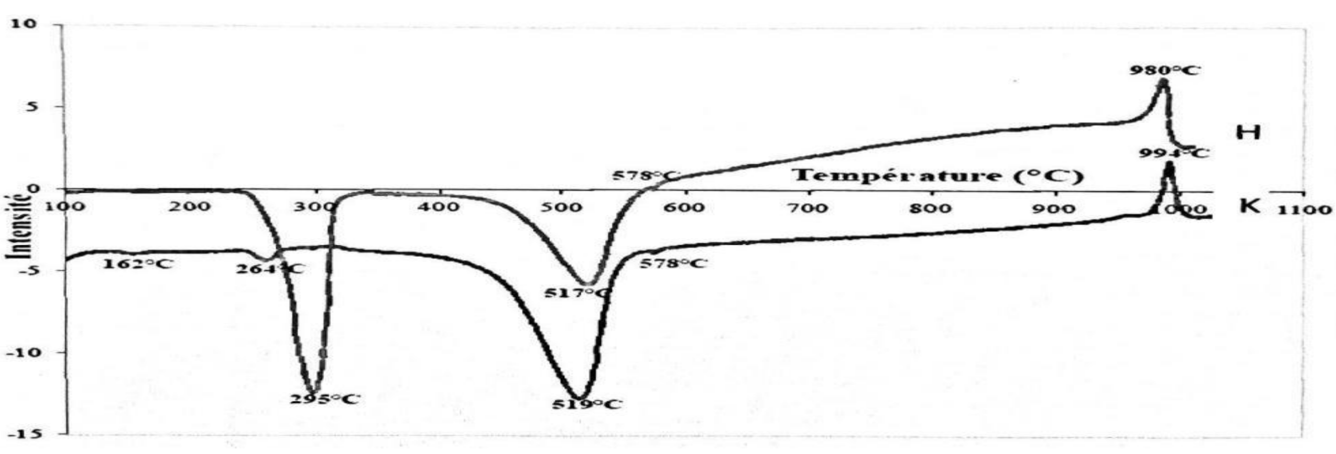

Figure 2: DTA curves of K and H (Kaolin and Hallosite respectively) (Tchamo, 2015)

- **Clay Activation and Optimum Calcinations Temperature**

Clay needs to be activated by modifying its original crystallinestructure so as to achieve good pozzalanic properties. This is achieved either by chemical, mechanical or the thermal activation methods, among which thermal activation is the best **(Elert et *al.,* 2008).** An endothermic reaction occurs during the conversion of hallosite to metakaolin due to the large amount of energy require to remove the structural bound water whereby the clay structure is modified and a less ordered phase is created. This phenomenon is referred to us as the dehydroxylation of clay minerals. The optimal calcination temperature is influenced by the mineralogy of chemical composition of the clay. In order to produce a pozzolan, nearly complete dihydroxylation must be attained without the clay being burnt because overheating or high calcination temperature above 900°C can lead to recrystallization of high temperature phases and additional sintering resulting in a decrease in the pozzolanic reactivity of the material. Several researchers have studied the pozzolanic activity and the optimal calcination temperature for pure clays.The optimal activation temperature depends mainly on the type and structure of the clay mineral. An overview of these studies and the most important conclusions drawn are given in the table below.

Table 2: Calcination temperatures for the different clay types (Taylor-Langr et *al.*, 2015)

Author	Ye	Clay type	Calcination T (°C)	Optimum act. (°C)	Pozzolanic activity
Ambroise	1984	Kaolinite	750	750, 5h	High
		Montmorillonite			Low
		Illite			Low
		Lateritic soils			Medium
Martin-calle	1989	Kaolinite	600, 650, 700, 750,	700, 24h	High
		Brick clay (illite/kaolinite	800, 850		Medium
He	1995	Kaolinite	550, 650, 800, 950	650	High
		Na-montmorillonite	740, 830, 920	830	Low
		Ca-montmorillonite	730, 830, 920	830	Medium
		Illite	650, 790, 930	930	Low
		Sepiolite	370, 570, 830	830	Low
		Mixed layer mica-smectile	560, 760, 960	960	Low
Chakchouk	20[illegible]	Kaolinite	500, 600, 700	700-800	High
		Illite/montmorillonite clay	500, 700, 800	-	Low
Fernandez	2013	Kaolinite	600, 800	600	High
		Illite		800	Low/Inert
		Montmorillonite		800	Medium
Taylor-Lange	2015	Kaolinite	650, 830, 930	650	High
		Kaolinite – Ca-montmorillonite		930	Medium
		Kaolinite – Na-montmorillonite		830	Medium to high

The optimum activation temperature of clay is the Temperature at which that clay starts to modify its original crystalline structure so as to achieve good pozzolanic properties.

Mejía de Gutiérrez et al. (2008a), carried out a test to show the influence of the calcination temperature of kaolin and they conclude that the optimum eating temperature for the kaolin in between 700 and 800°C. Summarily, most reported optimal activation temperature for kaolinites and hallosyte cluster between 650 and 750°C, we can therefore conclude that the optimum calcination temperature for hallosyte clay is very close to 750°c, where by the metakaolin produced has a very high pozzolanicity.

c) The Metakaolin from Balengou Halloysite Clay

Metakaolin is produced when Halloysite or kaolinite clays are calcined. It is used as supplement in cementitious material and calcination conditions is one of the most important factors influencing the pozzolanic property. It is produced by the calcination of kaolin clay within a define temperature range. The temperature range depends on th kaolin characteristics such as degree of crystallinity and particle size. The heating parameter such as temperature, heating rate and time, as well as cooling parameters (cooling rate and ambient conditions), significantly influence the heating process, the heating temperature range from 550°C to 850°C, but can still change depending on the nature of the kaolin. The appropriate temperature to obtain metakaolin from the Balengouhalloysite clay is 750°C **(SADO NJIKE, 2017).** The heating process drives off water from the mineral kaolin cly and collapses the material structure, resulting in an amorphous alumino silicate metakaolin. Below is a simple representation of metakaolin.

Figure 3: Metakaolin from Balengou (Sado, 2017**)**

Metakaolin is usually a white mineral material of aluminosilicate **(Kimberly, 2011).** The formula of metakaolin chemistry is $Al_2O_3Si_2O_2$ (Lilla et *al.*, 2013). The grain size of metakaolin is between cement and microsilica and the specific surface is about 15000 m²/ Kg. it‟s a supplementary cementing materials, these are materials used to replace part of the clinker in a concrete. Since it contains pozzolana which contributes to the amelioration of both fresh and hardened concrete properties.Metakaolin is neither the by-product of an industrial process nor is it entirely natural. It is derived from naturally occurring mineral and is manufactured specially for cementing applications. The following table elaborates the physical and chemical composition of metakaolin.

Table 3: Physical and chemical composition of metakaolin (Sanjay et *al*., 2013).

Chemical compsition (% by mass)				Physical properties	
Si_2O_2	54-53%	CaO	<0.2%	specific density	2.40 to 2.60
Al_2O_3	42-44%	MgO	<0.10%	Physical form	powder
Fe_2O_3	<2.20%		<0.050%	Color	White, Buff Gray
TiO_2	<3.0%	K_2O	0.4%	brightness	80-82
SO_4	<0.5%	L.O.I	<0.50%	Specific surface	8-15 m²/g

8 METHOD OF TESTING POZZOLANICITY.

a) **Direct methods.**

- **The Chapelle Test**

The principal is based on mixing a pozzolanic material in the presence of lime in water at a temperature of (85+-5°c) for 16 hours. The residunis filtered and reacted with hydrochloric acid to determine the quantity of lime that reacted with the material (Melissa and Ndigui., 2005)

- **The Frattini Test**

The procedure is specified by **EN 196-5 norm : 2011.** The pastes are prepared with 80% cement and 20% of the pozzolanic material. After preparation, the samples are conserved in scraped plastics for 8 days at 40°c. The pastes obtained are filtered at room temperature. The filtrate is then reacted either with hydrochloric acid or with E.D.T.A. (Ethylene DammineTetraacetic acid**) (Bich, et *al.*,2009).** The EN 196-5 norm evaluates the reduction in the amount of Ca^{2+} and OH- in a satured solution of lime at a temperature of 40 °C and in contact with the pozzolanic material. This solution of lime at a temperature of 40°C and in contact with the pozzolanic material. This solution is then titrated with E.D.T.A. solution or hydrochloric acid. **(Ndigui et *al.,* 2008).**

b) **Indirect methods.**

- **Different Thermal Analysis.**

This method is based on the measurement of residual pick of DTA of Calcium hydroxide on the powders of pastes done with a mixture of percentages of cement and pozzolanic material. The pastes are demolded after 24 hours, ground and sieved to obtain particles less than 100 micro meter. The powder is submitted to the ATD and the pozzolanic activity is determined **(Bich et *al*., 2009).**

- **Mechanical Measurements**

It is focused in the determination of the material reactivity it is calculated by the ratio between resistances to compression of mortars made with pozzolans those made with pozzolan after 28 days **(Cyr et *al*., 2012).** For various decades these materials were mixed with limestone to produce binders with high durabilities at room temperature. Nowadays, these materials are used in the elaboration of cement like Portland cements, dairy cements, lime cements and also geopolymer cements **(Melo and Ndigui, 2005).**

9. Thermogravimetric Analsis:

Thermo-gravimetric analysis allows to determine to which extent the calcined clay reacts with the lime by calculating the weight loss of the unreacted lime. Research has shown that this method gives an accurate determination of the pozzolanic reactivity **(Silva et *al.*, 2014).** In addition, it has been proven that also the evolution of the activity in time and the formed hydration phases, specific for the calcined clay-lime system, and be monitored efficiently (Tironi et *al.*, 2014)

10. MATERIALS AND EXPERIMENTAL METHODS

Any scientific domain or field of study requires a well-established and defined object of study as well as appropriate methodologies to develop knowledge about that object. However, with time, the concept or definition of the object of study can undergo modifications and adaptations due to scientific and technological advances, both in theoretical and experimental aspects.

10.1 Materials

Three principal materials are used in this study: clay, lime and distilled water.

10.1.1 Nature and origin of materials.

- **Clay material:**

The type of clay material use is the hallosite clay from Balengou, located at about 10km from the North-West of Bazou town, NDE Division, West Region of Cameroon and has as geographical coordinates 05°05'' and 05°10'' North, 10°25'' and 10°30'' East. The locality of Balengou is made up of granites partially covered by basalts (Déruelle et *al.*, 1991).

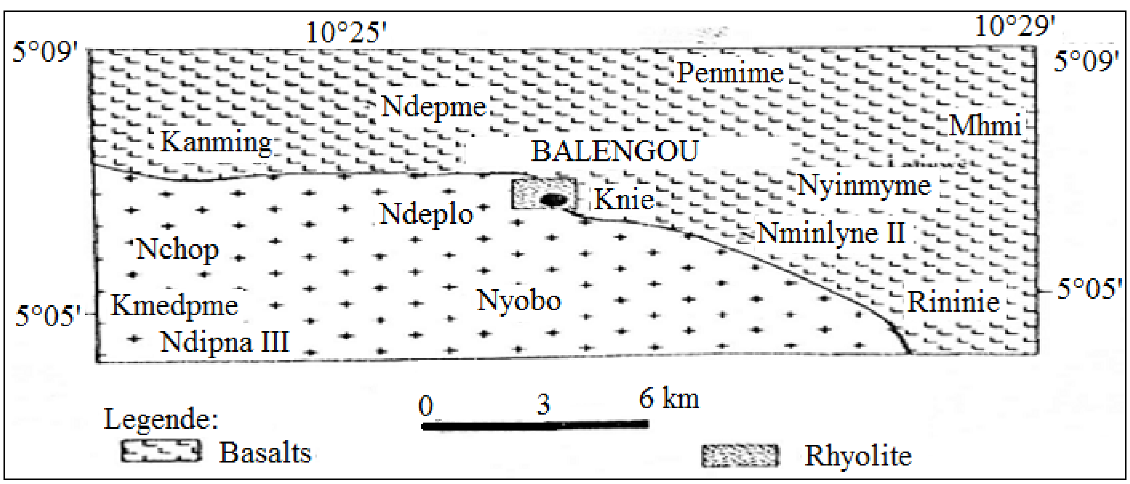

Figure 4: Representation of the mineral composition of the Balengou soil.

- **Lime :**

The lime is the pure lime CaO. We first of all mixed it with water, crushed it and then we dry it for about 24 hours.After that we pass it through the sieve in order to have a fine powder. Its apparent density is 430 kg/m^3

Figure 5: Lime at its solid state and at its powder state.

- **Distilled water :**

It is simply water without impurities. Water from the CDE is distilled in the IUT-BANDJOUN laboratory using a water distillator. It is done electrically in a condenser.

10.2 **Experimental Methods**

10.2.1 Préparations of the Materials

a) **Crushing and Sieving**

After extraction of the Balengou clay quarry, the halloysite is brought to the laboratory. It is allowed to air dry and split in small pieces with the help of a harmer. It is then crushed manually using a mortarand pestle and sieved with a 500 µm sieve. An amount of halloysite clay is left uncalcined and the second part is calcined at 750°C. Placed in a ball mill, the samples are crushed once more and allowed to pass through 100 µm sieve.

b) **Calcination**

The halloysite clay is calcined at 750°C using in an oven with 2hours soaking. These arrangements have been chosen in accordance with the works of Tchamo 2015, which showed that when the kaolinitic clays (like the Halloysite of Balengou) are treated as such, they produce metakaolin which are amorphous with high properties.

c) **Ball millgrining.**

The samples were therefore introduced in a rotating ball mill for a period of 2hours to obtain a maximum of fine particles. The mixture was then collected and sieved on a sieve of 100 µm with the help of brush. The refusal of this sieving are putted away. The passing is collected and kept on a close and dry bucket.

d) **Mix design**

The clays particles during the calcinations at 750°C are partially replaced in LIME cement. The percentage of substitution are 10%, 15%, 20%, 25%, 35%, 50%,65%, 75%, 80%, 85%. The substitutions are denoted S_{10}, S_{15}, S_{20}, S_{25}, S_{35}, S_{50}, S_{65}, S_{75}, S_{80}, S_{85}The control experiment is done with 100% Lime mortar for comparison purposes. It is denoted L.

Table 4: Percentage of Constituents in 1000 g of mixes.

Samples	Lime (Pure lime) about 95% of $Ca(OH)_2$, lime ROCA from Figuil		Metakaolin (clay at 750°C) from Balengouhalloysite	
	Mass (g)	**Percentage (%)**	**Mass (g)**	**Percentage(%)**
S10	**900**	**90**	**100**	**10**
S15	**850**	**85**	**150**	**15**
S20	**800**	**80**	**200**	**20**
S25	**750**	**75**	**250**	**25**
S35	**650**	**65**	**350**	**35**
S50	**500**	**50**	**500**	**50**
S65	**350**	**35**	**650**	**65**
S75	**250**	**25**	**750**	**75**
S80	**200**	**20**	**800**	**80**
S85	**150**	**15**	**850**	**85**
Control sample for comparison	**LIME**			
	Mass (g)		**Percentage (%)**	
	1000		**100**	

10.3 **Test on the Binder And Blended Mixtures**

10.3.1 Apparent Density

This test is done to evaluate the weight that is occupied by a sample in a given volume. Collect a dry granulated cylinder and weigh (Mc) fill the cylinder with the material until the edge used a trowel or ruler to level the surface of the cylinder weigh the cylinder + the material (Mc+p). Empty the cylinder and fill it with water (Mc+w), the apparent density is determined by the formula: $d = ((Mc+P) - Mc)/((Mc+W) - Mc))$

Figure 6: Apparent density Test process

10.3.2 Consistency

Consistency refers to the relative mobility of a freshly mixed cement paste or mortar or its ability to flow (flow ability). Standard consistency of a cement paste is defined as that consistency which will permit a Vicat plunger having a diameter of 10mm and 50 mm length to penetrate to a depth of 33-35 mm top of the mold. This is achieved using a Vicat apparatus, a 1kg capacity scale balance of accuracy 1g and a measuring cylinder. Environmental conditions are assured by a temperature of 27 ± 2°C and humidity 65 ± 5%. Generally, the normal consistency for OPC ranges from 26 to 33%. (IS, 1988), 300 g of either cement or the blended mixture is placed in the enameled tray. It is mixed with about 25% water by weigh of dry cement thoroughly to get a cement paste. Total time taken to obtain thoroughly mixed water paste i.e. « Gauging time » should not be more than 3 to 5minutes. The Vicat - mold is field, resting upon a glass plate, with this cement paste. After filling the mold completely, the surface of the Paste, is softened making a level with top of the mold. The whole assembly (i.e. mold + cement paste + glass) is then place under the rod bearing plunge. The latter is lowered gently so as to touch the surface of the test block and is quickly released allowing it to sink into the paste. The depth of penetration is measured and recorded. Trial pastes with varying percentages of water content are also prepared and the procedure is repeated as described above, until the depth of penetration becomes 33 to 35 mm.

The percentage of water (P) by weight pf dry cement required to prepare cement paste of standard consistency is determined by the following formula, and expressed to one decimal place. **P = W/C**

— Where W= quantity of water added

C= quantity of cement or blended mixture used

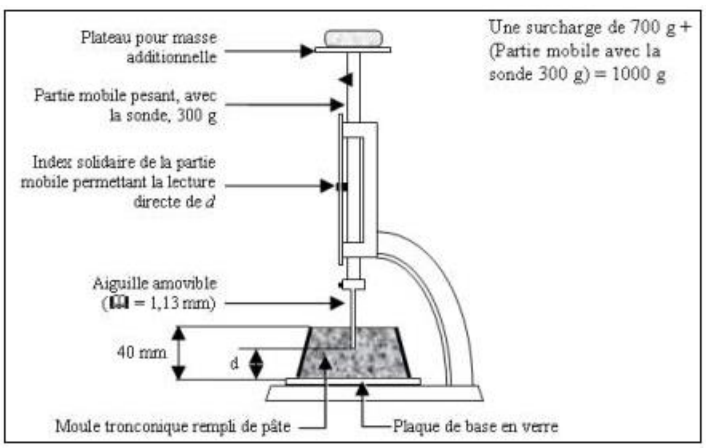

Figure 7 : Vicat content

- **Initial setting time**

The test block with the non-porous resting plate, placed immediately is under the rod bearing the initial setting needle. The needle is lowered and quickly released allowing it to penetrate into the mold. In the beginning the needle will completely pierce the mold. The procedure is repeated until the needle fails to pierce the mold for 5 + 0.5mm. The period elapsed between the time of adding water to the cement (t1) and the time when needle fails to pierce the mold by 5 + 0.5mm (t2) is recorded as the initial setting time.

Initial setting time = t2 – t1

- **Final setting time**

Replace the needle of the Vicat apparatus by the needle with an annular ring lower the needle and quickly release. Repeat the process until the annular ring makes an impression on the mold. Record the period elapsed between the time of adding water to the cement (t1) and the time when the annular ring fails to make the impression on the mould (t3) as the final setting time.

Final setting time = t3 – t1

The illustration below unveils the mechanism of needle penetration during the initial and final setting of the blended cements.

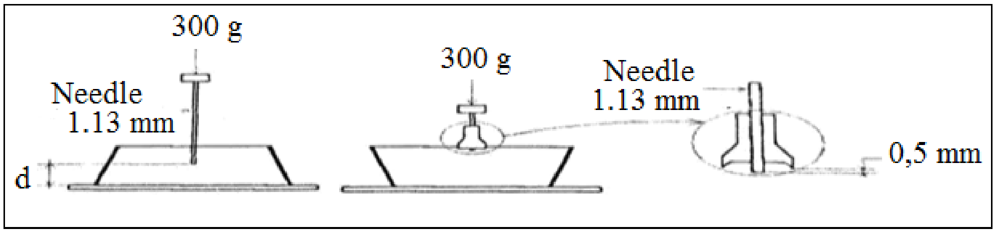

Figure 8: a) Initial setting **b) Final setting**

10.3.4 Compressive Strength of the Paste

Compressive strength of this research work has been carried out in the Geotechnical laboratory of the IUT Bandjoun It is defined as the ration of the load per unit area. The objective of this test is to determine the extent to which a sample + can resist to a compressive force. The mold used for this test are cylindrical mold obtained from the cutting of water pipes. The mold has a diameter of 22 mm and a height that is twice the diameter which is 44mm.

A vibrating machine, a poking rod, a gauging trowel having steel blade are used in this experiment. 300 g of cement is added to a quantity of water corresponding to (P/4 + 3) %, where P is the water content of the normal consistency (IS, 1988). It is mixed thoroughly to ensure a homogenous paste. The mold is filled with the paste in 3 layers, each layer submitted to vibrations by exerting vertical shocks in order to eliminate voids. The surface of the mold is levelled with a spatula. The mold is then removed after 24hours of the starting of the mixing and the samples kept in a humid condition system until the days of the crushing, done at 7, 14 and 28 days. The compressive force is determined by crushing the sample with an electric hydraulic crusher. The pictures below were taken during curing of the test specimens.

Figure 9: Curing test of the specimens at different percentage of lime and metakaolin

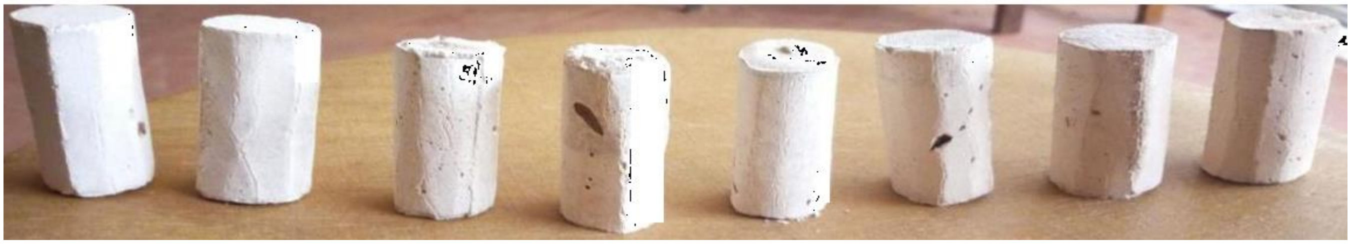

Figure 10: Curing test of the specimens at different percentage of lime and metakaolin

The specimens are centered vertically on the electric hydraulic crusher as shown below.

Figure 11: Compression of the specimens with the electric crusher

The speed of content is regulated at 2400 N/s to ensure a reasonable reading time. When the speed is regulated manually, the decrease is compensated closer to rupture. The resistance to compression is obtained by the ratio of the force and the surface area of the test specimens.

10.3.5 Water absorption

This test aimed at determining the quantity of water absorbed by a dry sample after immersion in water. To determine the water absorption of our paste, the samples are dried in an oven for 24 hours, the dry masses are recorded and they are placed in water for 7, 14, 28 days.

The samples are removed from water at the stated dates and water on the surface is cleaned gently afterwards the masses are recorded once more and the absorption determined by the following formula:

10.3.6 Study of the pozzalanicity of the materials: The Chappelle Test

The pozzalanicity of materials is assessed using two principal methods, either by the Chappelle Test or indirectly the activity index of the pozzolan. The test gives usefulindications on fixation of Calcium HydroxideCa$(OH)_2$ as well as the quantity fixed after 16 hours necessary for pozzalanic reaction. This test is done on blended mixture of lime and calcined clay at different percentage. That is 10%, 15%, 20%, 25%, 35% and 50% of calcined clay. The method aimed at determining the amount of Calcium hydroxyde fixed by the material in well stated conditions. The material is placed in excess calcium hydroxide for 16 hours at a temperature of 90°C. After filtration, the residue constituted of Calcium hydroxide which has not been fixed is made to react with an aqueous solution of 0.1 M of hydrochloric acid. The figures below demonstrate the Chappelle test execution.

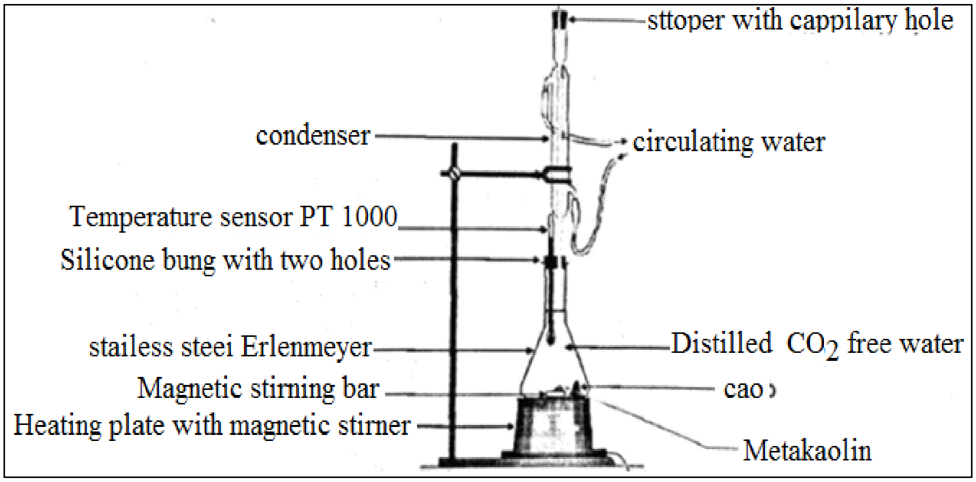

Figure 12: Assembly of the modified Chappelle test equipment

11. PRESENTATION AND INTERPRETATION OF RESULTS

Here, we display the results we obtained from the various laboratory test carried out. These results are based on the influence of the partial substitution of metakaolin to the lime at different percentage on the texture, consistency, initial and final setting time, apparent density of the solid mixture paste, water absorption, compressive strength and the Chappelle test of the calcined clays samples.

11.1 The Texture

The texture of the clay under study is evaluated by itscolor change as the firing temperature increases from 0° to 750° C.

Figure 13: HallosyteBalengou clay at it raw state and at 750°C after crushing.

The color change is cleary noticed with metakaolin fired at 750°C. It moves from the White to the Light-Orange. This phenomenon is explained by the high concentration of the hallosite clay in Iron II oxide which gradually gets oxidized to Iron III as the temperature increase.

On the blended cement mixture (lime+metakaoline), as the percentage of metakaolin increases, the blended mixture"s color changed. The color moves from the extremely white to the brown. This is due to the color of the calcined clays.

11.2 **Apparent Density**

Table 5 : APPARENT DENSITY

Sample	Mass of can	Mass of can + sample	Mass of can + water	Apparent Density
S10	31,37	145,46	303,62	0,419
S15	31,37	146,46	303,62	0,423
S20	31,37	150,64	303,62	0,438
S25	31,37	151,71	303,62	0,442
S35	31,37	158,04	303,62	0,465
S50	31,37	158,44	303,62	0,467
S65	31,37	169,36	303,62	0,507
S75	31,37	176,66	303,62	0,534
S80	31,37	177,01	303,62	0,535
S85	31,37	178,36	303,62	0,540

Lime and metakaolinmixures at different percentage (10%, 15%, 20%, 25%, 35% and 50%, 65%, 75%, 80%

and 85%) were passed through a 100µm sieve. And the absolute densities of those pozzolanic mixtures are given by the figure below;

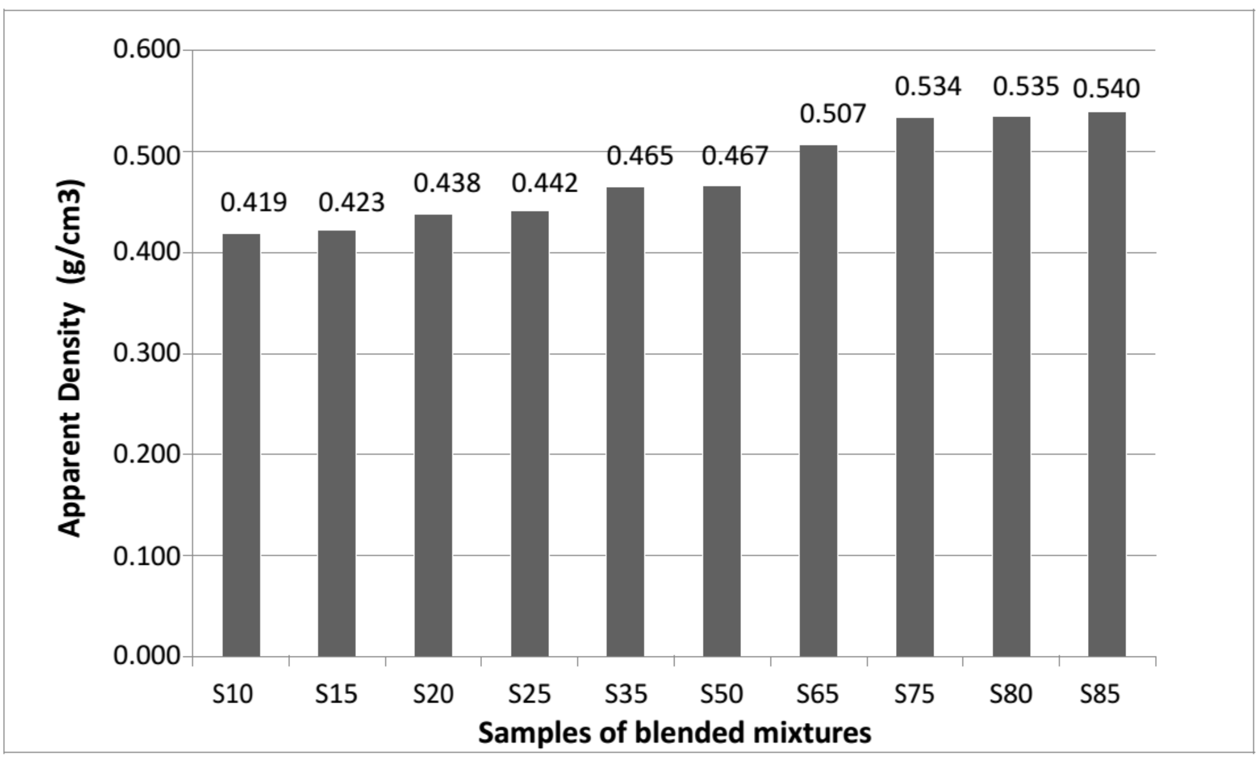

Figure 14: Apparent density of lime-metakaolin mixture at different percentage

The general observation of the graph shows that, as the percentage of metakaolin increases in the mixture, the apparent density of the blended cement also increases. This is due to the fineness of the particles contained in the metakaolin; finesness caused by its calcination. And also, that calcination increases the apparent density of hallosyte clay from 0,53at 0°C to 0,638 g/cm^3 (638 kg/m^3) at 750°C.

11.3 **The Consistency and the Workability**

Table 6 : CONSISTENCY TEST RESULTS

Sample	Added water	Consistency (%)
L	0.00	100
S10	2.00	90
S15	7.00	90
S20	11.00	87.5
S25	20.00	85
S35	2.00	82.5
S50	8.00	82.5
S65	12.00	80
S75	18.00	75
S80	7.50	75
S85	68.00	72.5

An increase in the amount of metakaolin, limits the penetration of the Vicat Needle. Water is then added again into the mixtures until the normal penetration of the Vicat plunger is reached (5mm ± 1mm) and the total amount of water added is noted as a ratio to the original mass of pozzolanic mixture as shown below.

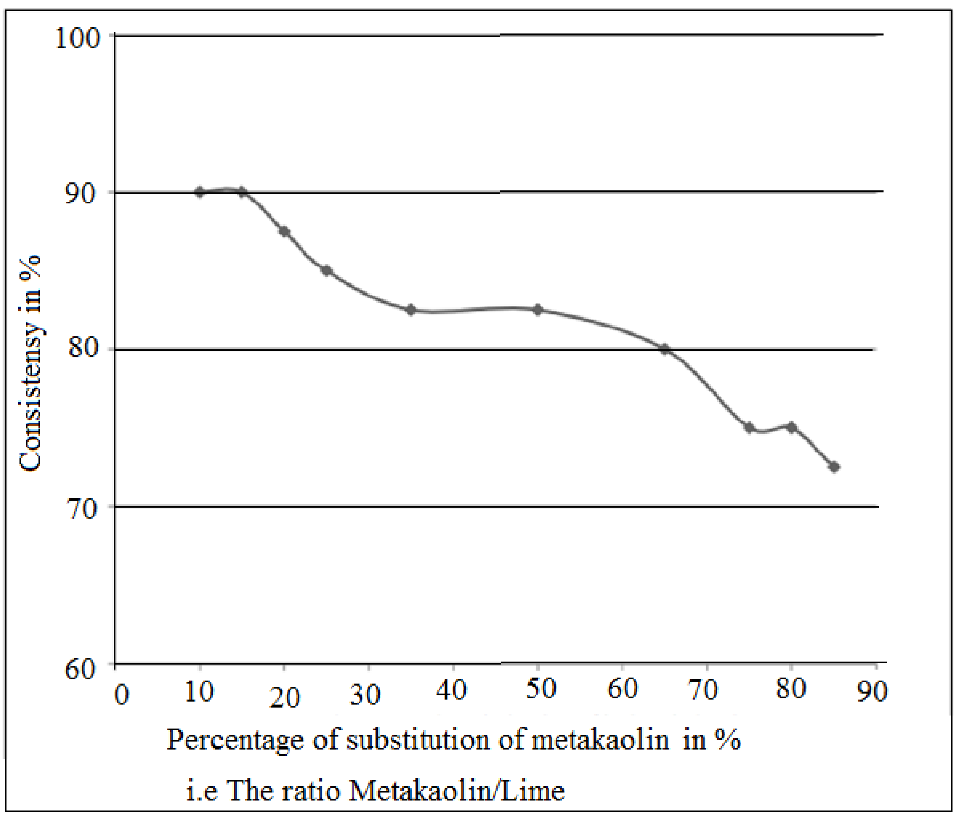

Figure 15 : Partial substitution by metakaolin in %

Looking at this diagram, the consistency of mortar reduces with quantity of metakaolin, this is link to the high demand of metakaolin in water which reduce the fluidity and plasticity of the mortar. As the demand in water for optimum plasticity reduces with the quantity of metakaolin, it affects negatively the workability. Increases in metakaolin to the mixture leads then to the loose of its workability.Whereas at low percentage of metakaolin substitution, the longer the final mixing time the more workable the mortar will be.

11.4 Setting – Time

Setting time is one of the most important property of a binder. The results obtained during the setting time experiment are presented in the following table.

Table 7: Initial and Final setting time

Samples	Initial setting / min	Final setting / min	Setting duration
Lime	237	357	178
S10	150	220	160
S15	145	210	172
S20	123	203	175
S25	133	158	171
S35	133	158	140
S50	97	124	90
S65	95	121	65
S75	58	103	50
S80	55	99	37
S85	50	85	20

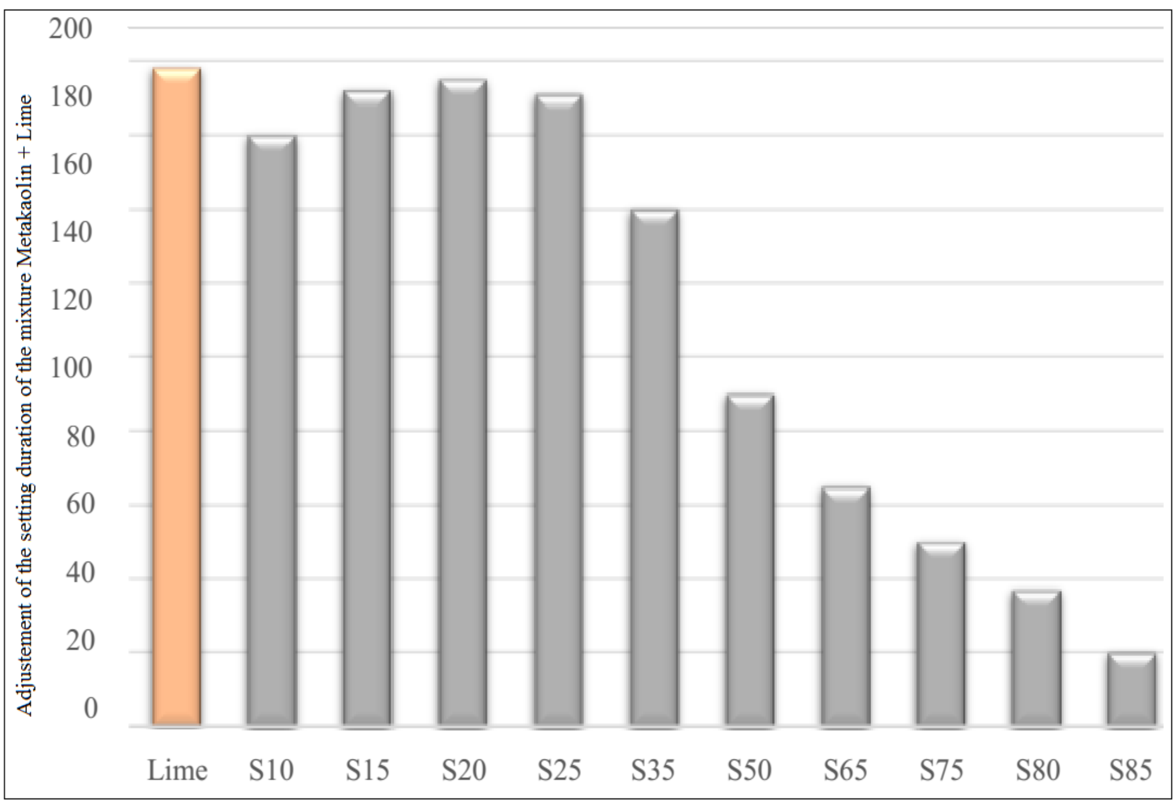

Figure 16 : Adjustment of the setting duration of the mixture metakaolin +Lime

It can be seen that initial and final intake are delayed by all samples. Samples containing metakaolin increase the duration of socket of the dough. A more stable structure is found with the formation of metakaolin at 750 °C

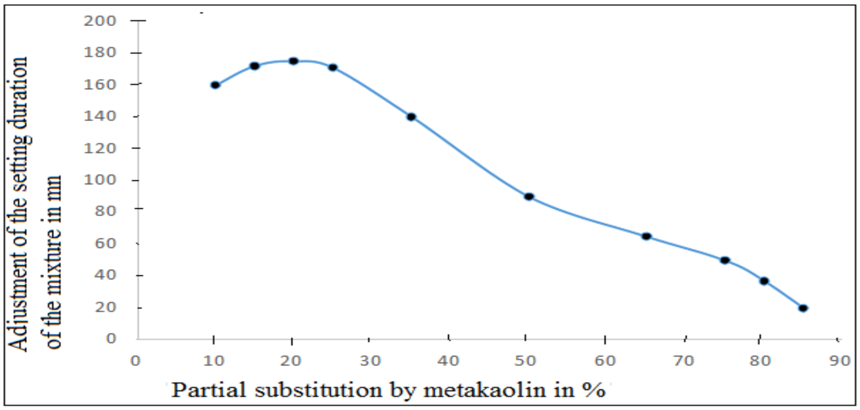

Figure 17: Definition of setting time curves for blended mixtures

Setting duration of the mixture paste up to 20% after which it drops. This increase is due to the increasing level of pozzolanic reactions taking place, residual lime originally liberated by the mixture which consequently reduces the degree of pozzolanic reactions and setting duration. Increase in the setting duration provides a greater time for the manipulation of the binder mortar paste during construction before hardening.

11.5 **Water Absorption**

The water contents of the cylindrical solid of our various binders were done and the results are presented in the table 9 to 11. The influence of the pastes when immersed in water for 24 hours after curingand drying in the oven for 24 hours are presented in the figure below.

The metakaolin reacts with the calcium hydroxide liberated by lime hydratation to form strong calcium aluminium silicates. Due to the formation of calcium alumino silicate hydrate precipitates (CASH), the ports of metakaolin become closed and absorb less water. Less water absorption is observed at 20% substitution. At this percentage, the structures build with lime + metakaolin mixture will maintain its shape and strength when exposed to water.

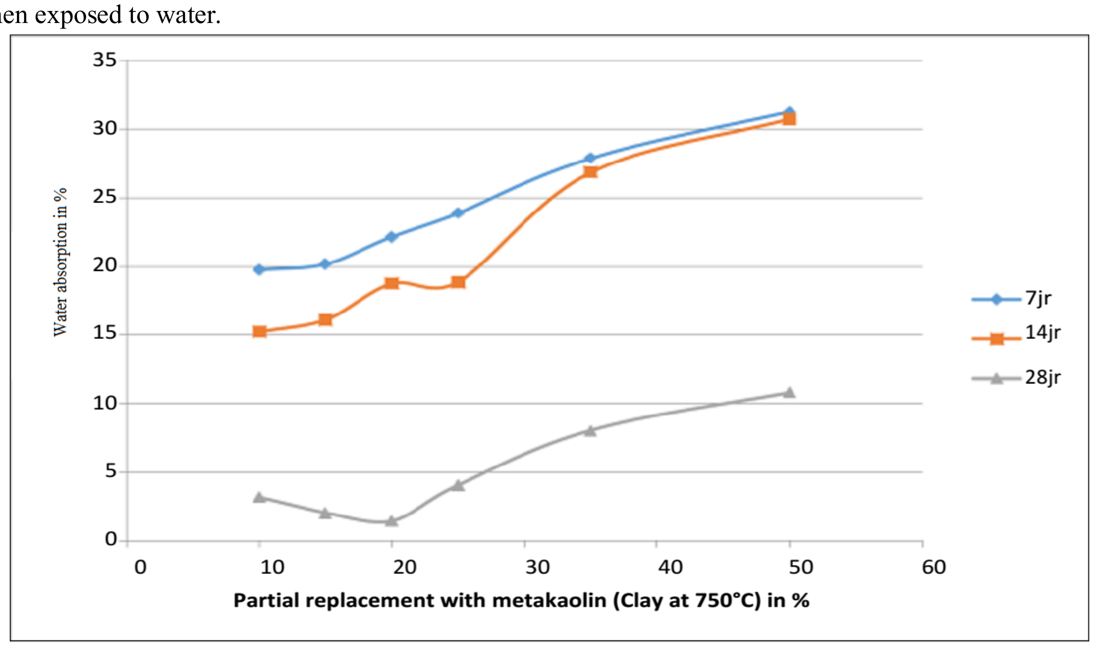

Figure 18: Water absorption

11.6 **Pozzolanic Activity in the Solution**

An acid-base reaction occured between 0.1 M of HCl and $Ca(OH)_2$. Assuming that all the OH^- jobs are obtained from the dissociation of $Ca(OH)_2$ which constitute the main constituent of lime, the following table is obtained.

Table 8: Summary of the results obtained from the Chappelle test.

Samples	Concentration of Ca(OH)2 in filtrate (mol/l)	Concentration of Ca(OH)2 in filtrate (g/l)	Quantity if Ca(OH)2 consumed (g/l)	Percentage of Ca(OH)2 consumed (%)
2.5 g metakaolin	0.0023275	0.17575	0.78625	81.73
10 g metakaolin	0.001	0.074	0.888	92.31
15 gmetakaolin	0.000625	0.04625	0.91575	95.19
20 gmetakaolin	0	0	0.962	100
Lime 0.013	0.926	0	0.00	

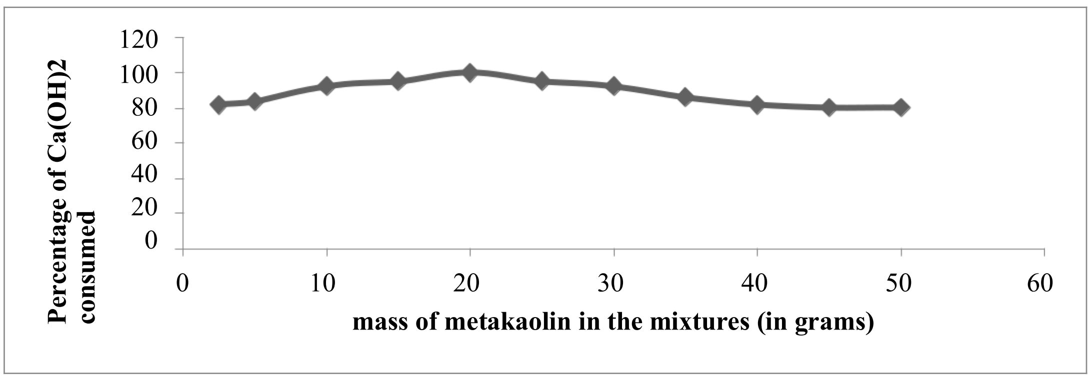

Figure 19: Percentage of $Ca(OH)_2$ consumed with as metakaolin is added.

The test indicate that thermally activated clay at 750°c absorb $Ca(OH)_2$ in solution at 20g. This is corresponding to the blended lime + metakaolin mixture $\mathbf{S_{20}}$. This is explained by the fineness of clay at 750°c and its greater specific surface which absorb a lot. The specific surface is therefore decisive for the pozzalanic activity, observable for materials comprising of phases in which silica and alumina are amorphous or mobilized. Crystalized forms such as quartz, cristobalite, trydimite and corridon do not participate in pozzalanic reactions.

11.7 **Compressive Strength**

The crushing of the samples was done after 7, 14 and 28days in University Technology Institute Bandjoun by using an electric compressive machine. For each type of binders, 3 samples of cylindrical paste were crushed and the average determine. The diagram below shows the compressive strength of various binders at different percentages of substitution

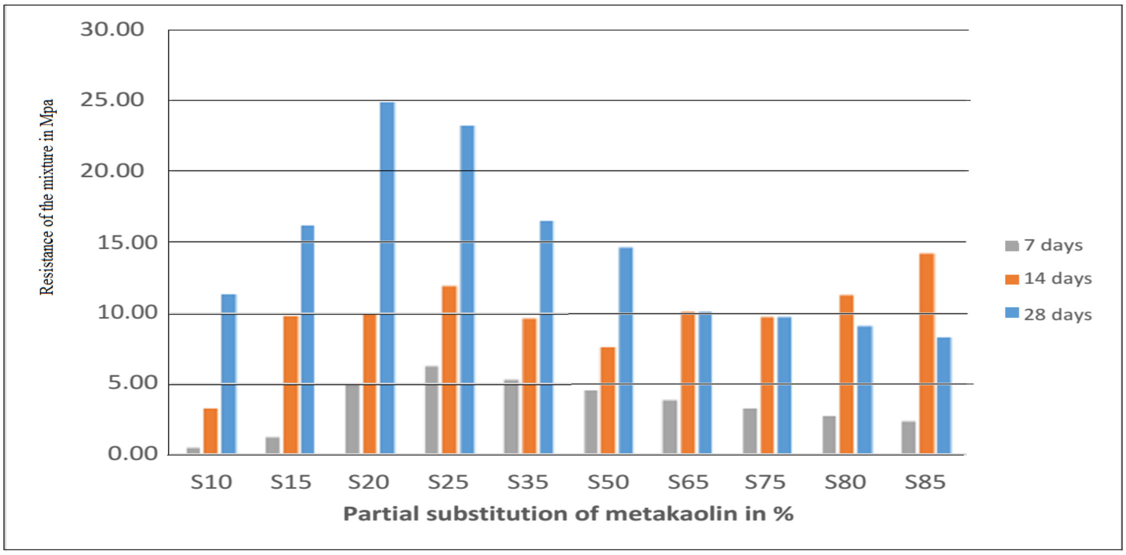

Figure 20: Compressive Strength chart

Table 9 : WATER ABSORPTION AND COMPRESSIVE STRENGTH IN MPA AT 7 DAYS

Sample	Dry mass	Wet mass %	H_2O absorption	Effort (N)	Compression (MPa)
S10	16,8	20,12	19,74	224	0,50
S15	14,34	17,23	20,13	556	1,23
S20	11,65	14,23	22,13	2233	4,94
S25	9,18	11,37	23,86	2816	6,23
S35	10,85	13,87	27,86	2395	5,30
S50	14,72	19,32	31,25	2036	4,50
S65	15,87	21,01	32,4	1730	3,83
S75	17,02	22,73	33,55	1473	3,26
S80	18,17	24,47	34,7	1249	2,76
S85	19,32	26,25	35,85	1065	2,36

Table 10 : WATER ABSORPTION AND COMPRESSIVE STRENGTH IN MPA AT 14 DAYS

Sample	Dry mass	Wet mass %	H_2O absorption	Effort (N)	Compression (MPa)
S10	15,23	17,55	15,23	1474	3,26
S15	16,41	19,05	16,07	4404	9,74
S20	10,36	12,30	18,72	4487	9,92
S25	11,78	14,00	18,83	5389	11,91
S35	11,20	14,20	26,83	4338	9,59
S50	18,84	24,62	30,69	3427	7,58
S65	19,99	26,35	31,84	4545	10,05
S75	21,14	28,11	32,99	4396	9,72
S80	22,29	29,90	34,14	5104	11,28
S85	23,44	31,71	35,29	6423	14,20

Table 11 : WATER ABSORPTION AND COMPRESSIVE STRENGTH IN MPA AT 28 DAYS

Sample	Dry mass	Wet mass %	H_2O absorption	Effort (N)	Compression (MPa)
S10	14.00	14,44	3,17	5111	11,30
S15	14,53	14,82	2,01	7302	16,14
S20	10,57	10,72	1,45	11251	24,87
S25	10,39	10,81	4,03	10483	23,17
S35	11,26	12,16	8,03	7458	16,49
S50	16,30	18,06	10,82	6605	14,60
S65	17,45	19,54	11,97	4545	10,05
S75	18,60	21,04	13,12	4396	9,72
S80	19,75	22,57	14,27	4098	9,06
S85	20,9	24,12	15,42	3740	8,27

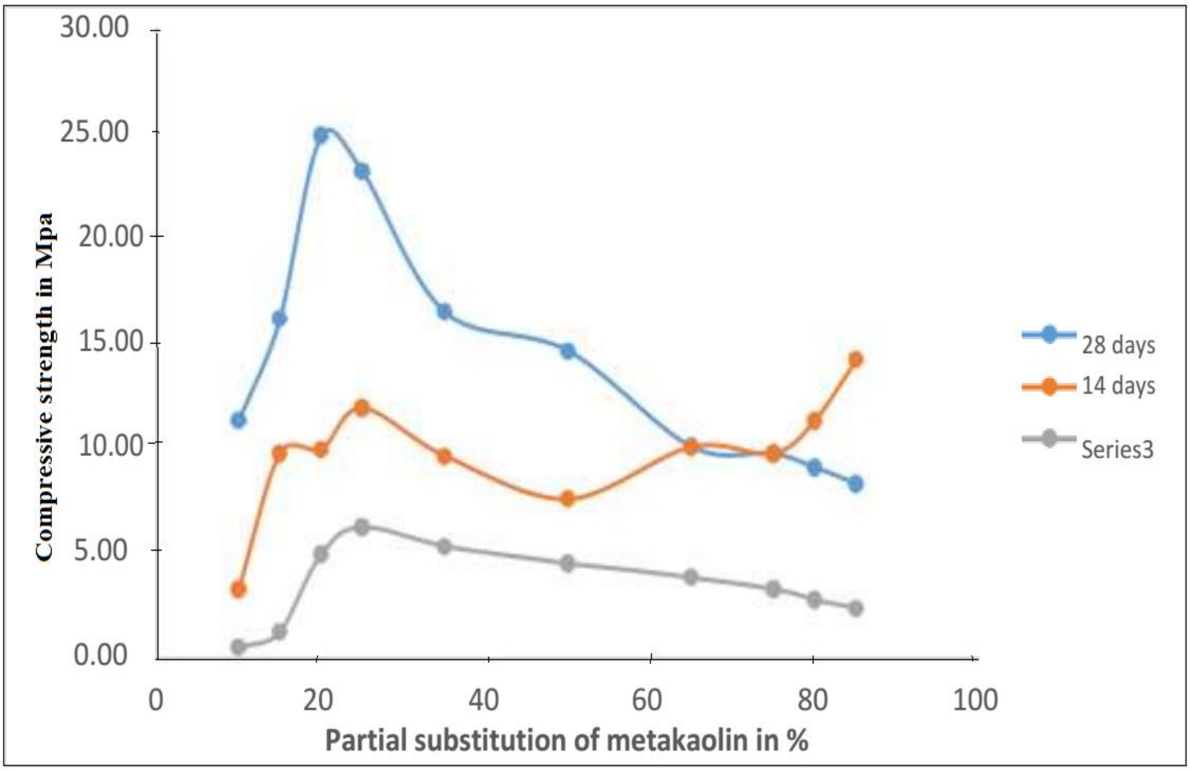

Figure 21: Compressive strength curves at different days.

The development of strength of the blended binder (lime-metakalon) depends on the pozzolanic activity of the calcined clays added at different percentage. At 7 days all the samples have a slightly lower compressive strength, not more than 7 MPa. This early low strength is probably due to the early reaction of pozzolanic materials. The mechanical resistances gradually increase at 14 days; it is observed that metakaolin, represented in red in the **Figure 21,** increases the mechanical strength of lime-metakaolin mixture. But finally at 28 days, when the strength starts to stabilize, the compressive strength highly increases till its maximal value at 20% of metakaolin substitution. This is due to the precipitation of C-S-H phases as a result of the reaction between the silica and the Ca2+ ions and the amount mainly depends on the Si/Al ration of the starting material. High alumina concentration will enhance the uptake of alumina in the C-S-H structure and C-A-S-H will additionally precipitate. The most common C-A-S-H for metakaolin is stratlingite.

Although the pozzolanic (metakaolin) addition increases the mechanical strength of the mixtures from the 14 days of curing as seen on Figure 21, this did not occur anymore when on the 28 days of curing; the metakaolin percentage passes 20%. The compressive strength drastically decreases. A possible explanation for this is that the surplus of metakaolin is not partaking in the lime-metakaolin reaction. In fact, taking into account the Volumic mass of lime (430 kg/m^3), of metakaolin (638 kg/m^3) and the fact that some of the lime will be used by carbonation and some by pozzolanic reaction, some of the pozzolan will not be involved in pozzolanic reaction due to lack of lime. It will directly lead to the cracking and crumbling of the surplus metakaolin, which reduices the mechanical strength of the paste after some days.

12. Conclusion, Scientific Relevance and Recommendations

12.1 Conclusion

We have studied the feasibility of metakaolin and lime as a binder mixture in Civil Engineering construction projects: case of Balengou clay. The hypothesis of this study are that Lime-metakaolin mixture as a binder in Civil Engineering is feasible, that Lime plus metakaolin binder affects the environment lower than normal cement" and that it is cheaper than the normal cement and can cover some domains in Civil Engineering projects." The literature review presented the generalities on hydraulic lime and calcined clays as pozzolans, the research methodology presented the materials and the various test to be done and the methodology displayed the presentation and discussion of results. From the investigation, the following conclusion can be drawn:

12.1.1 On the feasibility of lime-metakaolin as a binder mixture

- The calcination of clay at 750°C activates its reactivity with lime in a reaction called: pozzalanic reaction. At 20% of metakaolin substitution, that reaction is at its optimum state.
- Addition of Hydrated Lime improves the mix plastic properties but reducethe mortar strength designation. Whereas addition of metakaolin reduce the plasticity, Permeability, capillary, porosity and increases the strength of the paste.
- at 20% of metakaolin substitution, we obtain a maximum compressive strength of 24.87 MPa at 28 days, a good consistency at 90 % of water content.
- The setting duration reduce as we increase the metakaolin.

12.1.2 On the low impact of lime-metakaolin binder on the environment as compare to normal cement.

- The use of lime mortars is a contribution towards sustainability, due to lower temperatures (900°C) used in the production process and lower CO_2 emissions; additionally, during the carbonation process, CO_2 is absorbed from the atmosphere.
- At the optimum percentage of metakaolin for correct pozzalinicity reaction, the carbonation process still occurs as in the pure lime. Because They also have some free lime available for carbonation.
- Lime-metakaolin mortar can manage humidity and allow moisture to evaporate, helping to keep building free of damp and create healthier internal environment.
- With the respiration of slabs permitted with the perspiring property of lime, capillary effects on walls are reduced.
- The humidification of walls through condensation process in the house is reduced. This is because the wall can breathe due to the air permeability property of lime-metakaolin mixture.

12.1.3 On the domains that could be cover by lime-metakaolin mixture in civil engineering

- Metakaolin is an adequate pozzolanic addition for lime mortars, providing adequate mechanical and water behaviour characteristics for application in internal Pointing with soft masonry, plastering, tiling, Bedding, Flooring, or chimney flaunching.
- Lime-metakaolin is cheaper because it needs less energy consumption for its production
- Lime-metakaolin has a good appearance. And so, it can be used for aesthetical and decorative works. For instance, its white color and its plasticity which reduces cracking and spalling on pointing works, could be the advantages for the interior decorations.

12.2 Scientific Relevance

The use of greater percentages of pozzolan in a mortar doesn’t necessarily imply improved characteristics. For each particular pozzolanic product there are specific formulations that produce better results for the application that is being considered.

12.3 Recommendations

Following the results obtained in this work, the following recommendations are made for a future use of the Balengou Hallosyte clay and a new approach on the utilization of lime.

- Lime-metakaolin is viable with good physical properties such as plasticity, high strength, and breathability at 20% of metakaolin.
- Use lime-metakaolin binder for internal Pointing with soft masonry, plastering, Bedding, Flooring, rendering. but not for high structural works.
- Use Lime-metakaolin only for works where the need for breathability and lower strength is outweighed by the desire for an earlier and harder set such as working on bedding hard masonry, wall copings, chimneys and slate floors.

12.4 Further Research

- Study of the cracking susceptibility and durability (climate and salts) on lime-metakaolin binder
- Study of the acoustical and thermal isolation properties of lime-metakaolin mixture used in wall masonry.
- Resistance strength of our lime-metakaolin mixture to climate attacks such as frost and extreme dryness and the chemical attacks such as sulfate attack and alkali resistance.

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