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Study on the Production of a Geopolymeric Cement from Rice Husk Ash in Madagascar

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Abstract - Currently the Malagasy government is launching a policy of building housing in each region. However, the supply of construction materials (cement, iron, sheet metal…) is made through imports which is not within the reach of all households, and constitutes the main obstacle to achieving its objectives.

Madagascar has large quantities of natural resources. Kaolin clay deposits with very large reserves have been identified in different parts of the island, for example in IBITY (Vakinakaratra) or ANJIRO (Alaotra Mangoro), and as a rice producing country, we have large quantity of husks (hard outer shell of the rice grain that are agricultural waste).

After metakaolin (obtained by calcining kaolinic clays), active fine silica (obtained by burning rice husks) is essential for the manufacture of sodium silicate and weakly basic calcium silicate (mixture of rice husk ash and Portland cement), which are the raw materials required to develop and study the production of out geopolymeric cement.

The aim of our research is to find a new building material that can become a real alternative to artificial Portland cement, in economic, technical and environmental terms.

The interest in geopolymer cements is obvious for the economies of developing countries.

Keywords - Cement, Geopolymerization, Metakaolin, Rice Husk Ash, Soda Ash, Silica, Alumina.

I. INTRODUCTION

The objective of our study is to master at the laboratory level and on a small scale, the technology of producing geopolymeric cement from: rice husk ash, metakaolin, Portland cement, sodium silicate.

In this context, the aim of our research is to define from the experimental data the optimal conditions that are directly related to the mechanical strength performance of the prepared and tested samples.

This publication is devoted to a part of our experimental study, which is the identification and optimization of the parameters that influence the mechanical strength of our geopolymeric cement.

II. METHODS

3.1 Standardized tests in accordance with official specifications

To confirm a certain number of results noted in the bibliography concerning the mechanical performance of geopolymeric mortars, we carried out a test campaign at the National Laboratory of Public Works and Building (LNTPB) in accordance with standard EN 197-1 on our geopolymeric cement, these tests are:

- Determination of mechanical resistance
- Chemical analysis
- Determination of setting time and stability
- Determination of the fineness

3.2 Determination of mechanical resistance

The objective of the test is to determine the compressive strength in accordance with EN196-1

Compressive strength is made up of current and short-term strength

3.3 Chemical analysis

The purpose of chemical analysis is to determine the contents of the various chemical elements (expressed as a percentage by weight) contained in our cement, in accordance with EN 196-2

3.4 Determination of setting time and stability

The determination of setting time and stability must comply with the requirements of EN 196-3

3.5 Determination of fineness

According to EN 196-6, the determination of fineness is the measurement of the specific surface area by Blaine permeability

3.6 The materials used to prepare the samples

Mechanical test samples are made from mortars made of materials such as geopolymeric cement, water and sand

3.6.1) Sand

Normal sand according to EN 196-1 is clean sand with rounded and dry grains. It is a product from Leucate (France). As we do not have this famous product, we have prepared our own experienced sand, so that our tests will be comparable to the standard tests of other hydraulic binders such as COA Artificial Portland Cement.

Our experienced sand is a dune sand that we took from Amborovy Mahajanga, and its silica content is about 98%. It has undergone the first essential treatment such as: washing and drying in order to ensure that our experienced sand is free of clay elements and organic matter (humus) that compromise stability, solidity and harm the mortar's construction.

After undergoing a series of crushing and sieving operations, our experienced sand has been manufactured with a varied grain size, respecting the same grain proportions as normal sand

Table 1 shows us the normal sand division. These suitable fractions are mixed to obtain the desired granularity.

Table 1: Normal sand division

Sieve: square mesh openings (mm)	AFNOR module	Percentage by mass	Cumulative percentage refusals	Weight for 1350g of sand
2,00	34	0	0	0
1,60	32	5	5±5	68
1	31	28	33±5	378
0,5	28	34	67±5	459
0,16	23	21	88±5	283
0,08	20	12	98±2	162

3.6.2) Water

The mixing water must be clean, if possible, potable, and must not contain any salts that may affect the setting time. For this reason, we use JIRAMA water.

3.6.3) Geopolymeric cement

Our geopolymeric cement is comprised of: rice husk ash, metakaolin, Portland cement, and sodium silicate.

Each raw material is weighed to obtain the molar ratio of silica, alumina, cement, metakaolin and sodium silicate for our geopolymeric composition.

Metakaolin, Portland cement and rice husk ash are finely ground and then intimately mixed to obtain a homogeneous grain mixture.

III. RESULTS AND INTERPRETATIONS

To better observe and interpret the mechanical test results of our samples, we adopt the presentation of these results in the form of curves and histograms.

But above all, we would like to specify the most important parameters to study in order to obtain a good quality of geopolymeric cement

4.1) Mechanical test results

4.1.1) the parameters to be studied

The parameters are classified into two categories:

- Fixed parameters
- Variable parameters

a) <u>Fixed parameters</u>

These fixed parameters are:

- The calcination temperature of kaolin clay= 750°C for 3 hours
- The incineration temperature of rice husks = 600°C
- The preparation temperature of the sodium silicate is the ambient temperature
- The fineness of the metakaolin is less than 0.16 mm or 160 microns
- The quantity of binder for specimen production = 450 g
- The quantity of sand for specimen making= 1.350g
- The quantity of water for specimen preparation=245g (W/C= 0.54)

b) Variable parameters

- After preliminary studies, we have identified the parameters that directly influence the mechanical strength of our geopolymeric cement, these parameters are :
- The molar ratio SiO2/Al2O3
- The molar ratio of soda silicate to metakaolin= n SS /n MK
- The molar ratio CaO/MK=n CaO/ n MK
- The molar ratio SiO2/Na2O= n SiO2/ n Na2O
- Age of the sample: 2 days, 7 days, 28 days
- How the samples are stored: in water and in the open air

We would like to point out that n_TSiO_2 is the mole number of total silicas, say the sum of the mole number of silicas in metakaolin and the mole number of silicas in Portland cement and the mole number of silicas in rice husk ash.

Our method consists in varying a parameter, by setting the other parameters

4.1.2) influence of the molar ratio n SiO2/ nNa2O

Figure 1 shows the evolution of compressive strength (Cs) as a function of the molar ratio SiO_2/NaO_2

We studied six formulations: n SiO_2/ n Na_2O = 1.12 / 1.3 / 1.56 / 1.96 / 2.6 / 3.96

The specimens were made by setting the following variable parameters:

- The molar ratio n SS/ n MK = 0.67
- The molar ratio nT SiO2/ n Al2O3 = 3.86
- The molar ratio n CaO/ n MK = 2.9
- The age of the sample is 07days
- How the samples are stored: in water

By gradually increasing n SiO_2/ n Na_2O, that means the modulus of the soda ash passes from detergent to abrasive (by decreasing the quantity of soda), the evolution of the resistances is remarkable.

For values below n SiO_2/ n Na_2O= 2.6, the resistance starts to decrease.

The explanation is that the excess soda in the soda ash is harmful because it is no longer necessary for the dissolution of the metakaolin and the rice husk ash, in addition the excess makes the mortar more fragile especially the sample is kept in water. Moreover, soda ash is a very corrosive product, and as such the excess is very dangerous for its use.

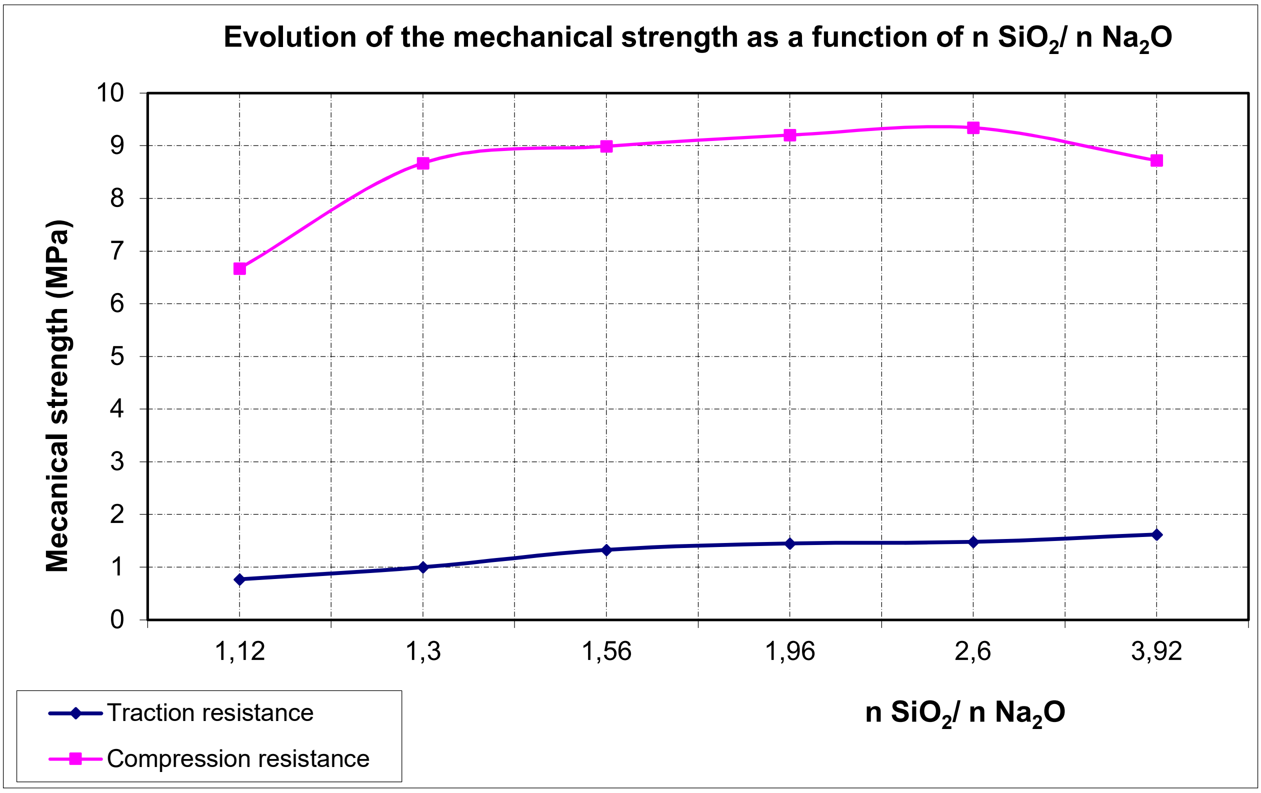

Figure 1: Evolution of the mechanical strength as a function of n SiO_2/ n Na_2O

For values above n SiO_2/ n Na_2O=2.6, the resistance decreases because a large part of the metakaolin and rice husk ash is not dissolved due to insufficient soda ash, that means, the higher the ratio n SiO_2/ n Na_2O, the lower the chance that a sodium ion can attack the metakaolin and rice husk ash.

Most bibliographies indicate that they use detersive modulus sodium silicate, i.e. n SiO_2/ n Na_2O<2 and in addition it does not specify how the samples are stored, but our experimental study confirms that the strength reaches the maximum Cs= 9.34 MPa for n SiO_2/ n Na_2O=2.6 and we have chosen to store our samples in water, because not all mortars or concrete that is not resistant in water is not acceptable for its use.

In conclusion, the objective is to dissolve the metakaolin and rice husk ash in order to release the reactive elements silica and alumina, i.e. the more the metakaolin and rice husk ash dissolves completely in silicate and aluminate monomers to recombine into aluminosilicate oligomers (first step in geopolymerization), the higher the resistance.

As such for n SiO_2/ n Na_2O=2.6 the dissolution of metakaolin and rice husk ash is optimal, and in addition the use of sodium silicate in the habitat is acceptable because it did not show corrosivity, in other words it will respond to the same classification of hydraulic binders that is irritating.

The general appearance of the tensile strength curve is almost identical to that obtained for compressive strength.

But it should be noted that the optimal tensile strength (Ts= 1.62 MPa) is significantly lower than that of the control cement (Holcim's Manda Ts= 4.83 MPa)

4.1.3) influence of the molar ratio nTSiO2/ n Al2O3

Figure 2 shows the evolution of the compressive strength (Cs) as a function of the molar ratio n_T SiO_2/ n Al_2O_3

The specimens were made by setting the following variable parameters:

- The molar ratio n SiO2/ n Na2O = 2.6
- The molar ratio n SS/ n MK = 0.67
- The molar ratio n CaO/ n MK = 2.9
- The age of the sample is 07 days
- How the samples are stored: in water

Six formulations were studied $n_T\ SiO_2/\ n\ Al_2O_3$= 3.37 ; 3.60 ; 3.85 ; 4.04 ; 4.50 ; 4.70 ; (corresponds for Si/Al = 1,7 ; 1,8 ; 1,9 ; 2 ; 2,25 ; 2,35)

As we increase $n_TSiO_2/\ n\ Al_2O_3$, i.e. increase the silica content, we see that the resistance increases and has a maximum value Cs= 1O.33 MPa for $n_TSiO_2/\ n\ Al_2O_3$= 4.5 (equivalent to Si/Al = 2.25)/

It should be noted that the higher the silicon concentration, the higher the proportion of silicate oligomers, and the proportion of monomer decreases

We measured the weight of each sample, and the average weight of the test pieces of each formulation shows us that is goes from 533g to 543g. As such, we can see that the binder becomes more homogeneous and denser when $n_T\ SiO_2/\ n\ Al_2O_3$ increases

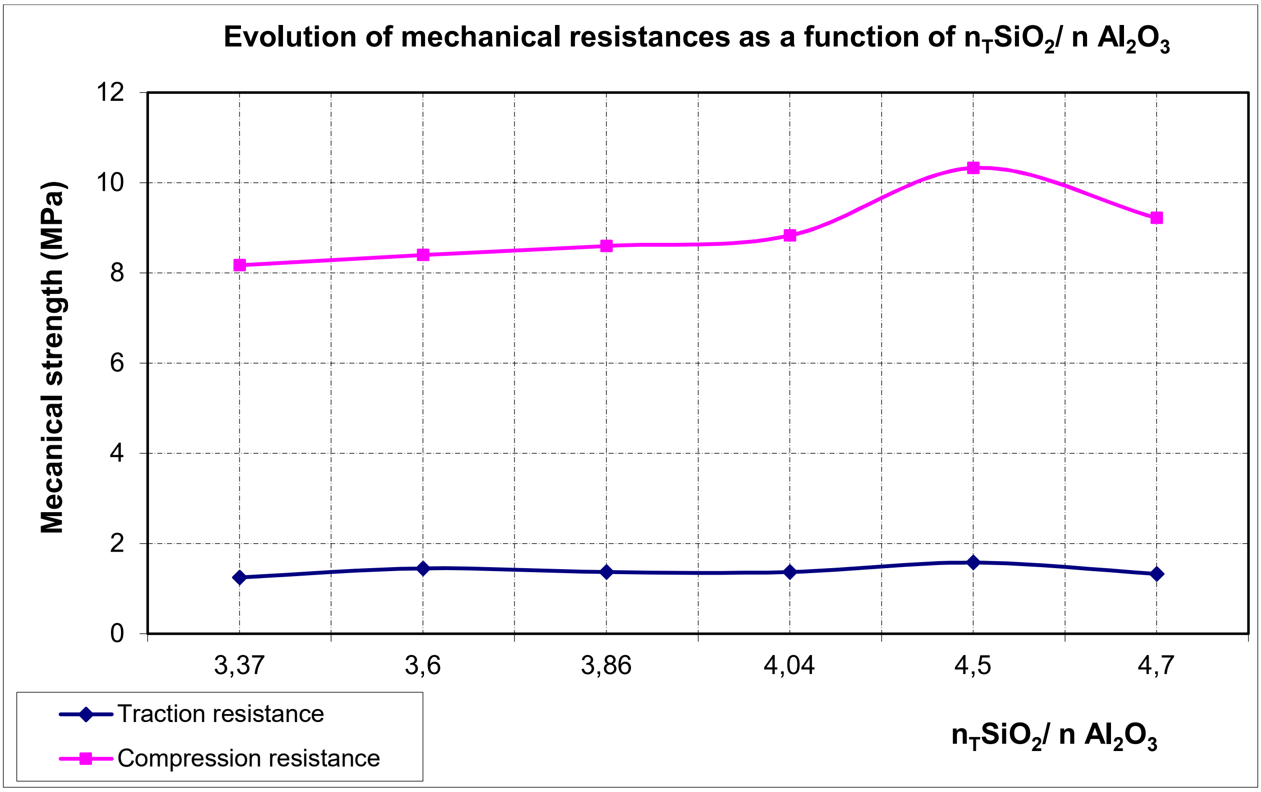

Figure 2: Evolution of mechanical resistances as a function of $n_TSiO_2/\ n\ Al_2O_3$.

For $n_TSiO_2/\ n\ Al_2O_3$< 4.5 (Si/Al< 2.25), we find that the resistances are low, this means that the proportion of silicate oligomers is low compared to the silicate monomer, for this purpose, the rate of polycondensation or geopolymerization is low which leads to the decrease in resistance. In addition, the average weight of each formulation is about 530g, which explains why the specimens contain voids, and this microstructure is responsible for the low mechanical properties.

Thus, for Si/Al> 2.25 or $n_TSiO_2/\ n\ Al_2O_3$> 4.5, we notice that the resistance drops, i.e. the excess silica is harmful, because this excess silica is no longer transformed into silicate oligomers so does not participate in the last stage of geopolymerization which is polycondensation or freezing hence the decrease in resistance.

Our experimental study shows that the compressive strengths of our samples reach maximum for a ratio (Si/Al=2.25) to ($n_T\ SiO_2/\ n\ Al_2O_3$= 4.5). So, at this point, the dissolution is almost total for metakaolin and rice husk ash, in addition, the proportion of silicate oligomers is high so the degree of geopolymerization is maximum, and finally we can see that the test pieces are homogeneous and dense because their average weight is 543g.

The majority of publications indicate that they have the maximum resistance for (Si/Al=2) or ($n_T\ SiO_2/\ n\ Al_2O_3$= 4), but this ratio is lower than our result. The explanation is that they use silica fumes from the filters above the melting furnaces of ferro-silicon steels (SiO_2 content= 92% and average diameter= 10microns), while we use rice husk ash (SiO_2 content= 85% and average diameter of 160 microns), and above all the reason for our high ratio is that our active silica (rice husk ash) contains a large part of overheating dur to the uncontrolled burning of rice husks.

The molar ratio of oxides Al_2O_3/Na2O=3.14 is between 1.5 and 4; these values allow to confer a very long stability over time.

Similarly, the pervious curve shows the general appearance of the tensile strength curve and is almost identical to that obtained for compressive strength, and the tensile strength Ts reaches 1.58MPa.

To conclude, the control of the incineration of rice husks and its fineness is important, because it has a significant influence on the reactivity of silica and therefore on the mechanical resistance of our sample.

4.1.4) Influence of the molar ratio n CaO / n MK

Figure 3 shows the evolution of compressive strength (Cs) as a function of the molar ratio n CaO / n MK

The specimens were made by setting the following variable parameters:

- The molar ratio nTSiO2/ n Al2O3=4.5
- The molar ratio n SiO2/ n Na2O=2.6
- The molar ratio n SS/n MK= 0.67
- The age of the sample is 07days
- Sample storage method: in water

We studied seven formulations n CaO/ n MK= 0 ; 0,60 ; 2,86 ; 3,89 ; 4,88 ; 7,11 ; 11,34

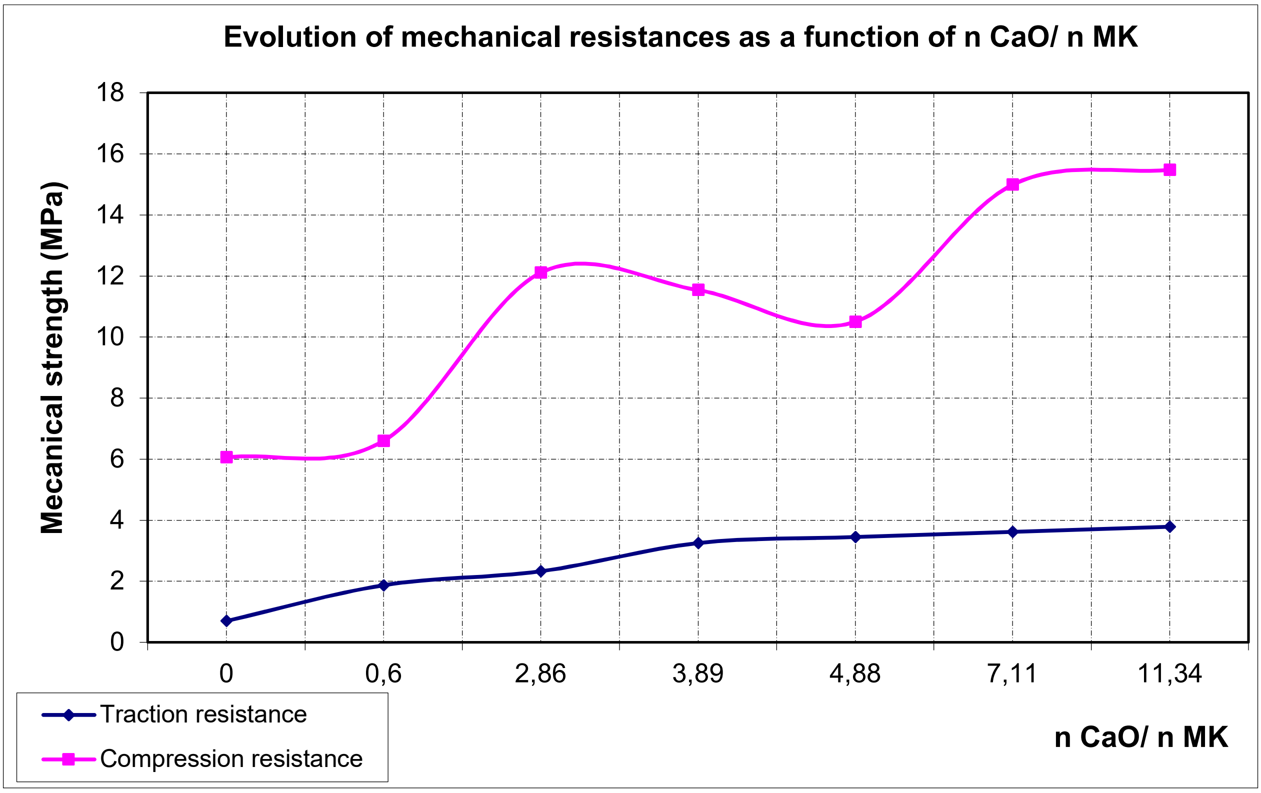

Figure 3: Evolution of mechanical resistance as a function of n CaO/ n MK

Calcium oxide CaO belongs to the weakly basic silicate, more precisely to the Artificial Portland Cement.

The following tests clearly show the importance of the role of CaO in the geopolymer matrix, as it is the calcium ions Ca^{++} that determine the curing rate, in addition it makes mortars and geopolymeric concretes less soluble in water, and finally the optimal polycondensation reaction is achieved when these Ca^{++} ions are integrated into geopolymer structure.

For n CaO/ n MK = 0, i.e. the geopolymeric mortar has no CaO contribution, or more precisely no CPA addition. The first observation is that the specimens were not taken until after forty-eight hours, and then the samples were removed from the mould and stored in water. After seven days, the compressive strength is about 6MPa. This value is low, the reason being that the mortar is less resistant to water because the CaO carrier and also as a setting activator.

For 0< n CaO/ n MK ≤ 2,86, i.e. by increasing the CaO content in this range, first we notice the possibility of demoulding the specimens after twenty-four hours of mixing and secondly, we observe a rapid evolution and then we see a stabilization of the mechanical strength Sc=12.11 MPA for n CaO/ n MK= 2.86. The justification for the increase in compressive strength is due to the total substitution of Na^+ ions (sodium silicate) by Ca^{++} ions (Portland cement) which are integrated into the geopolymeric matrix. Experience shows that rapid curing requires the addition of Portland cement, in addition to acting as a catalyst that promotes the geopolymerization reaction, but the strength of the mortar is ensured by that of the geopolymer, i.e. by mixing metakaolin, active silica and sodium silicate.

2.86< n CaO/ n MK≤ 4.88, in other words when the molar ratio n CaO/ n MK approaches 3 to 4.88, there is a decrease in the compressive strength of the specimens to 10.5 MPa. In the meantime, the experimental study has shown us that antagonistic physico-chemical phenomena appear which act at the same time. Our explanation is that there is a disruption between the polycondensation of aluminosilicate oligomers and the hydration of silicates and aluminates in Portland cement.

For n CaO/ n MK > 4.88, i.e. the molar ratio is 7.11, there is a sudden rise in Cs= 15MPa. The reason of this sudden increase in compressive strength is a massive precipitation of hydrates for Portland cement and aluminosilicate oligomers for geopolymer, therefore the strength of the samples is ensured by mixing metakaolin, active silica, sodium silicate and Portland cement.

For n CaO/ n MK>7.11 or about 11.34, the compressive strength of the specimens is stable at about 15.5MPa. The reason is that the excess Portland cement is harmful, i.e. the excess Ca^{++} ions are no longer integrated into geopolymeric matrix.

Part of the literature uses Portland cement as a source of CaO, and others use blast furnace slag, but only a small portion of the latter mentioned the result of the molar ratio n CaO/ n MK which is the order of 0.6. This value is very much lower than our experimental result of 2.86 and 7.11.

Our explanation is as follows: we have made samples which are then stored in water corresponding to n CaO/ n MK=0.6 and we have found that the corresponding compressive strength is low, in the order of 6.60MPa, which means that the addition of Portland cement is a small proportion, for this purpose, the quantity of Ca^{++} ions is insufficient to replace Na^+ ions in the geopolymeric matrix which leads to a decrease in the rate of hardening, and subsequently to the decrease in the mechanical strength of test pieces.

In conclusion, figure 3 of our experimental curve shows two optimums, one for n CaO/ n MK= 2.86 and the other for n CaO/ n MK=7.11; which means that we have two types of cement, in other words, two different uses.

4.1.5) Influence of the molar ratio n SS/ n MK

Figure 4 shows the evolution of compressive strength (Cs) as function of the molar ratio n SS/ n MK

The specimens were made by setting the following variable parameters:

- The molar ratio nTSiO2/ n Al203=4.5
- The molar ratio nT SiO2/ n Na20= 2.6
- The molar ratio n CaO/ n MK=7.11
- The age of the sample is 07days
- Sample storage method: in water

Six formulations were studied n SS/ n MK= 0,32 ; 0,41 ; 0,57 ; 0,71 ; 0,86 ; 1,02

Geopolymerization begins with the alkaline attack of the monovalent Na^+ cations of sodium silicate on the surface of the metakaolin and active silica (rice husk ash), followed by the dissolution phase, i.e. the breaking of the Si-O-Si and Si-O-Al bonds of the metakaolin to form reactive precursors $Si(OH)_4^-$ and $Al(OH)_4^-$ in the mortar.

If the sodium silicate content is between 0.32 ≤ n SS/ n MK < 0.57, we find that the resistance decreases slowly to Cs=14.5MPa. our explanation is the following, the oxide ($Si_2O_5Al_2O_2$) or metakaolin reacts first of all with sodium silicate, and if the quantity of the latter is mall, this means that a significant part of the metakaolin is not dissolved, therefore these aluminums and siliciums of the metakaolin do not participate in the structuring of the geopolymeric matrix and in addition the mortar is not sufficiently alkaline to generate the geopolymerisation.

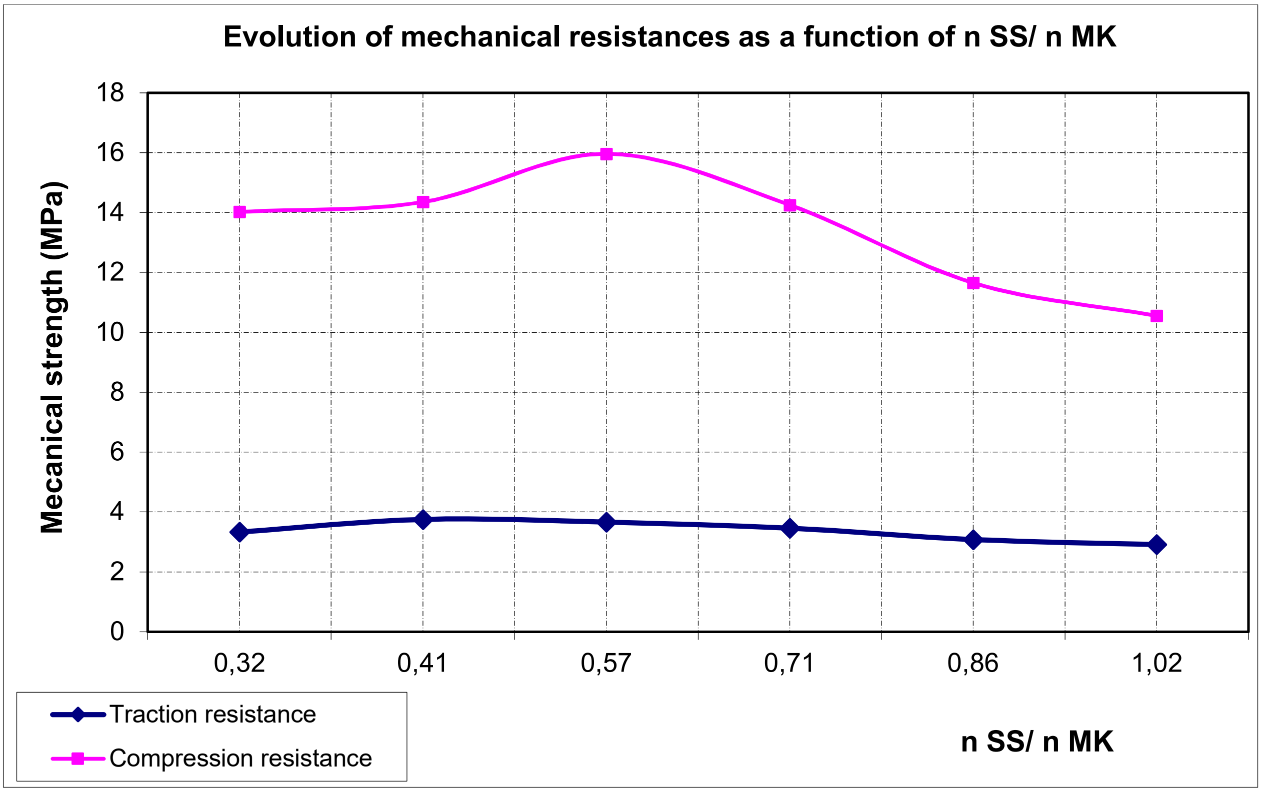

Figure 4: Evolution of mechanical resistance as a function of n SS/ n MK

On the other hand, if the quantity of sodium silicate is high for n SS/ n MK< 0.57; the first effect observed is that the resistance decreases significantly. Cs= 10MPa, for this effect it will require a large quantity of Ca^{++} ion to achieve the same curing rate. Consequently, the excessive solubility of the reaction medium slowed down the precipitating action of Ca^{++} ions from Artificial Portland Cement, thus contributing to the drop-in resistance.

For n SS/n MK=0.57, the solubility of metakaolin and rice husk ash is optimal, which results in maximum sample resistance: Cs=16MPa

In conclusion, the solubility of minerals in alkaline sodium silicate solution has a direct influence on the compressive strength of the specimens, because the more soluble the metakaolin and rice husk ash, the more resistant the samples are.

By comparing our result with that of the bibliography, we notice that they are perfectly in agreement because the latter has published the value of n SS/ n MK=0.60

N.B: we would like to point out that up to now the test pieces made from our experimental sand have been deprived of the last fraction of the normal sand grain size, which is 80 µ, and the corresponding proportions have been changed with the upper mesh sizes, because Amborovy dune and contains a small quantity of fine fraction such as 160 µ and 80 µ.

4.1.6) Influence of the age of the specimens

Figure 5 shows the evolution of compressive strength (Cs) as a function of the age of the specimens.

The specimens were made by setting the following variable parameters:

- The molar ratio nTSiO2/ n Al203=4.5
- The molar ratio nTSiO2/ n Na20= 2.6
- The molar ratio n CaO/ n MK=7.11
- The molar ratio n SS/ n MK= 0.57
- Sample storage method: in water

We studied three ages for the test pieces: 02 days, 07 days, and 28 days; and they are made with normal Leucate sand.

We would like to point out that after the setting follows the hardening period

The experimental study shows that compressive strength increases as the samples get older and older.

At an early age, two days after mixing, there is a slow evolution of compressive strength Cs=8.5MPa, due to the high content of silica in our geopolymer mortar $n_T SiO2/ n Al_2O_3=4.5$ as silica-rich systems react more slowly with a pause in the reaction steps.

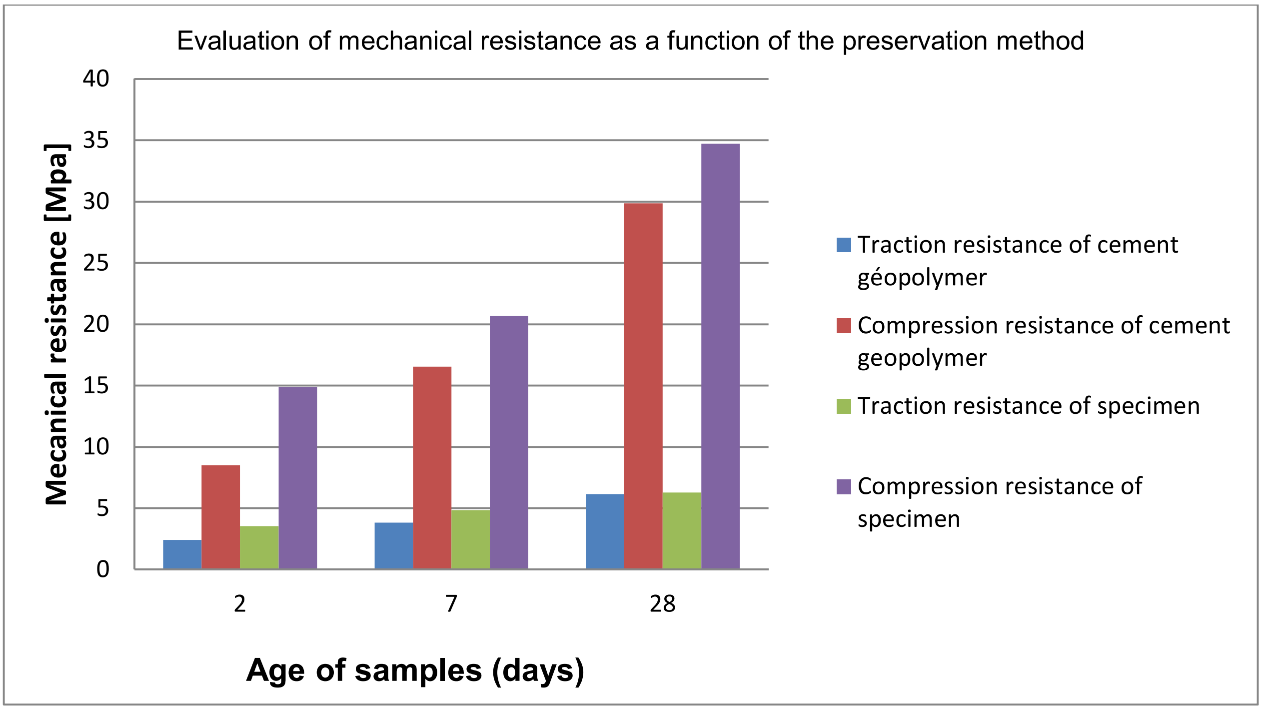

Figure 5: Evolution of mechanical strength as a function of the age of the specimens

After seven days, we see a rapid evolution of resistant with a 100% increase, from 8.50 MPa in 02days to 16.55 MPa in 07 days.

But between 07 days and 28 days of age, we observe an increase in compressive strength 81%, because in 28 days Cs=30MPa.

In our experimental study we see Holcim's Manda cement, and we use it as control samples i.e. 100% Manda cement samples.

By comparing our geopolymeric cement with that of the control cement, the following conclusions can be drawn:

According to table 2, the first observation is that at an early age, Holcim's Manda cement (control cement) is much higher than our geopolymeric cement, which is why the control cement belongs to a high short-term strength class rated R.

However, between 07 and 28 days of storage in water, we notice that the difference in compressive strength is no longer significant.

Table 2: comparison of mechanical strength between our geopolymeric cement and that of the control cement

Mechanical resistance	02 days		07 days		28 days	
	Cs	Ts	Cs	Ts	Cs	Ts
Our geopolymeric cement	8,51	2,41	16,55	3,83	30	6,16
Control cement	14,91	3,04	20,66	4,83	34,71	6,29

It should be noted that the bending tensile strength of our specimens at 02 and 07 days is significantly lower than that of the control samples, while at 28days, the Ts of the two cements are almost the same

We would like to inform you that the experimental conditions are almost the same for both cements (e.g. W/C=0/54), but the only difference is that our geopolymeric cement is made from Leucate sand, while the test pieces of the control cement are prepared using our experimental sand without the 80 µ fine fraction.

4.1.7) Influence of the conservation method

Figure 6 shows the evolution of compressive strength (Cs) as a function of the storage method

The tests were carried out by setting the following variable parameters:

- The molar ratio nTSiO2/ n Al203=4.5
- The molar ratio nTSiO2/ n Na20= 2.6
- The molar ratio n CaO/ n MK=7.11
- The molar ratio n SS/ n MK= 0.57
- Age of samples: 02 days, 07 days, 28 days

We would like to point out that the specimens are made from our experienced sand, which respects the grain size of normal sand.

Two methods of storage were used: in water and in the air

The evolution profile of the mechanical properties of our samples stored in water and air is the same. We are witnessing a slow evolution, then a sudden rise and finally a stabilization of the resistance.

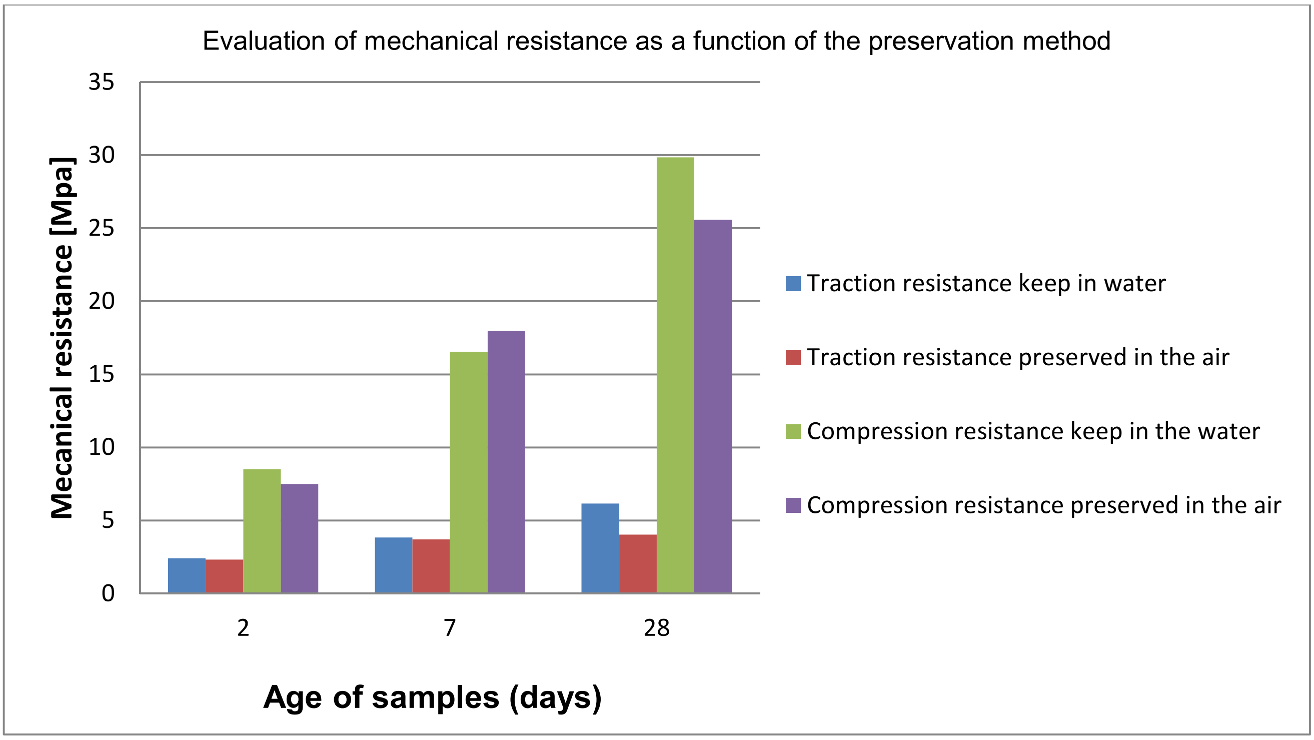

Figure 6: Evaluation of mechanical resistance as a function of the preservation method

By comparing the storage method, the compressive strengths of the specimens stored in air for 02 days and 28 days are significantly lower than those stored in water.

In air, we have maximum Cs=7.5 MPa in 02 days and 25MPa in 28days. Under the same conditions, the specimens stored in water have an optimal Cs=8.5MPa in 02 days and 30 MPa in 28days? To this end, the loss of resistance is 20% in 07 days and 30% in 28 days.

While the resistances of the specimens kept in the open air for 07 days: Cs=18MPa is slightly higher than those of the samples kept in water for 07days: Cs= 16.5MPa. This can be explained by the fact that our cement is a geopolymeric binder, because curing is done by polycondensation of aluminosilicate oligomers, but not as hydraulic cements as the hydration of silicates and aluminates is the cause of curing.

Our experimental result found that the bending tensile strength of our air-preserved specimens is significantly lower than that of water-preserved samples regardless of the age of the samples.

4.1.8) Influence on the modulus of soda ash silicate

Figures 7 and 8 show the evolution of Compressive strength (Cs) as a function of the modulus of soda ash silicate

The tests were carried out by setting the following variable parameters:

- The molar ratio n_T SiO2/ n $Al_2$03=4.5
- The molar ratio n CaO/ n MK= 2.86
- The molar ratio n SS/ n MK= 0.67
- Age of samples: 02 days, 07 days, 28 days
- How the samples are stored: in water and in the air

We would like to point out that all previously mechanically tested specimens are made from metakaolin from Anjiro kaolin clay (Alaotra Mangoro), but the result we present as follows is from samples made from Prochimad and metakaolin (Ibity quarry in Vakinankaratra)

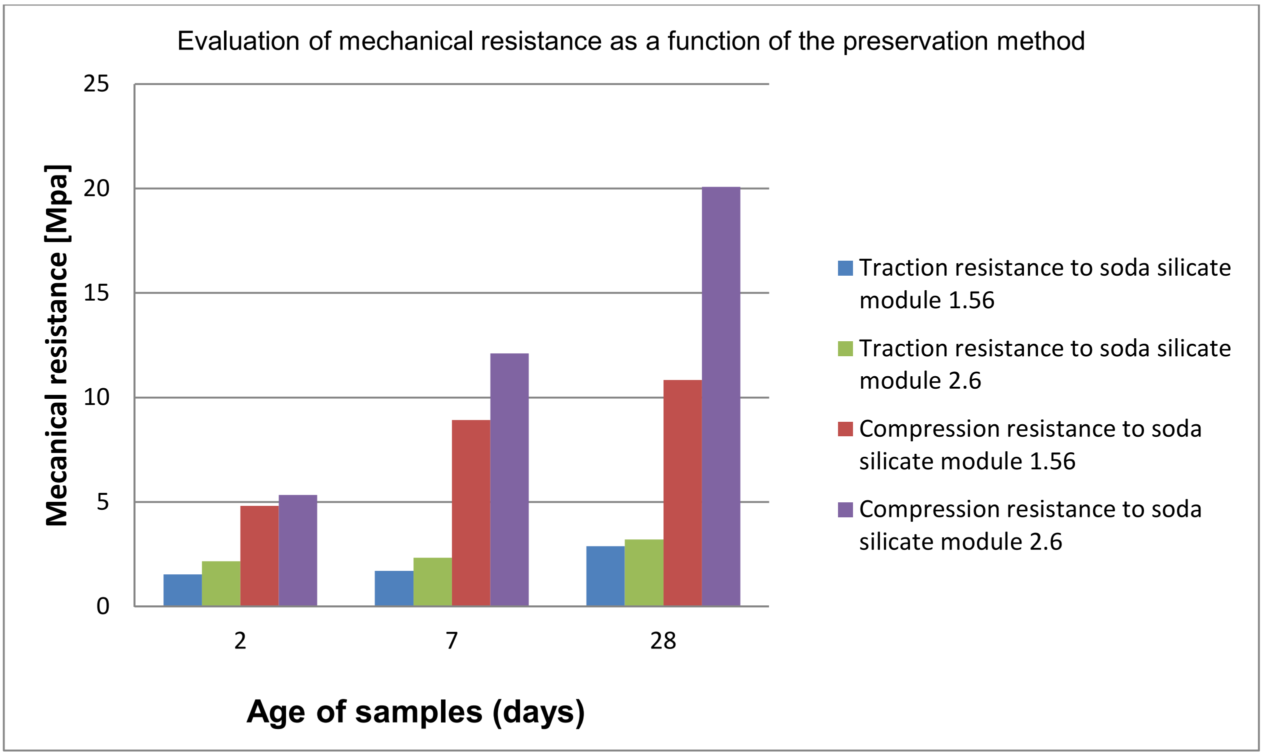

Figure 7: Evolution of mechanical strength as a function of age for two different soda ash silicate modules (water conservation)

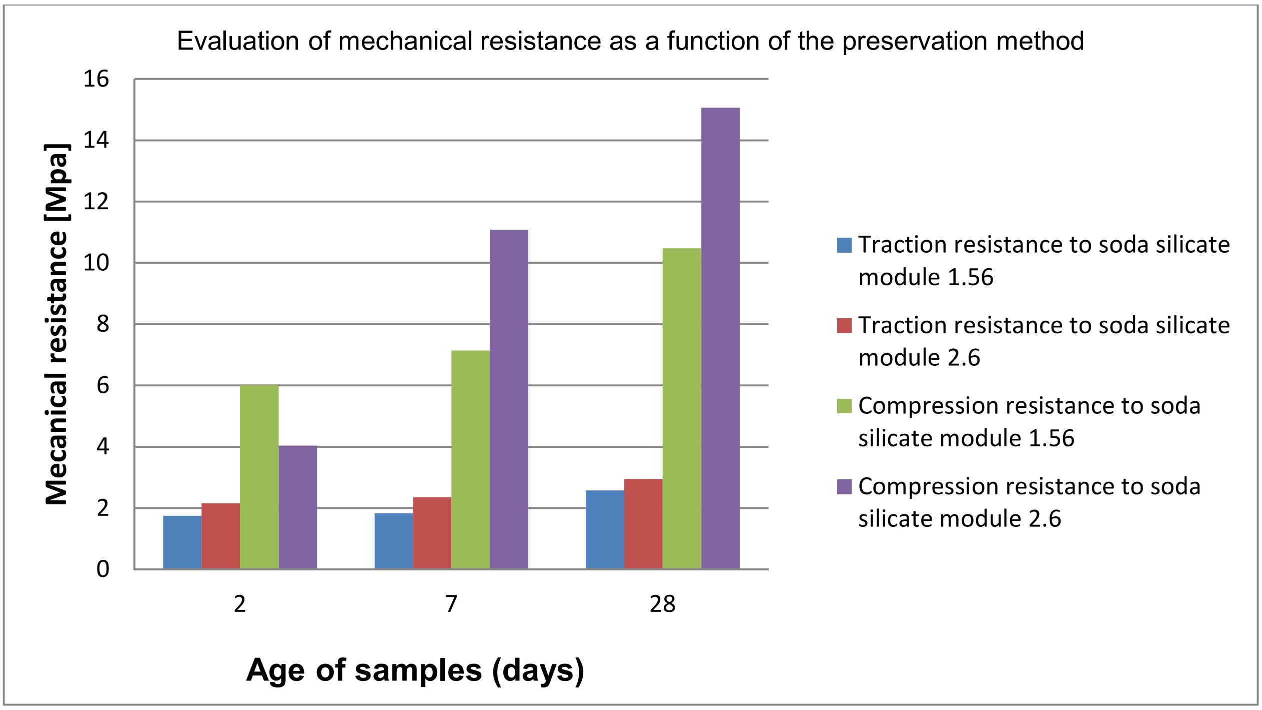

Figure 8: Evolution of mechanical strength as function of age for two different soda ash silicate modules (air preservation)

Looking at figure 7 and figure 8, the first finding leads us to conclude that our experimental study to very clearly identify that the mechanical strengths for specimens made from abrasive modulus sodium silicate (n SiO_2/ n Na_20= 2.6) are much higher than those made from detersive modulus sodium silicate (n SiO_2/ n Na_2O=1.56), regardless of the age and storage method of the samples.

For example, at 28 days storage in water, the compressive strength of the specimens made by soda ash silicate of modulus n SiO_2/ n Na_2O=2.6 (Cs=20.07 MPa) is twice that made by modulus soda silicate: n SiO_2/ n Na_2O= 1.56 (Cs= 10.84 MPa). This superiority is still valid for samples kept in open air, except for the tensile strength Cs of the abrasive module which is lower than that of detergent module if the age of the specimens is 02 days.

In conclusion: first, we note that the replacement of Anjiro's metakaolin by Prochimad's metakaolin (Ibity) does not have a major influence on mechanical resistance, and second, the result obtained experimentally for the molar ratio n SiO_2/ n Na_2O disagrees with the majority of the results of the other researchers mentioned in the bibliography.

4.1.9) Influence on Portland cement content

Figure 9 shows the evolution of compressive strength (Cs) as a function of Portland cement content.

The tests were carried out by setting the following variable parameters:

- The molar ratio nT SiO2/ n Al203=4.5
- The molar ratio n SiO2/ n Na2O= 2.6
- The molar ratio n SS/ n MK= 0.67
- Age of samples: 02 days, 07 days, 28 days
- Sample storage method: in water

The objective of this graphical representation is to show the two optimal strengths that correspond to two cement contents depending on the age and storage method of the samples: this allows us to have two types of products and then propose their respective possibilities of use.

In a few figures, for a cement content n CaO/ n MK=2.86 (corresponds to 31% Portland cement in our geopolymeric cement), the compressive strength of the specimens after 28 days stored in water is about 20MPa. This value is close to the strength of 22.5 MPa class cements. Indeed, our first type of cement will be used for simple masonry such as: plaster and mortar, soil stabilization, foundation slab.

For a cement content n CaO/ n MK=7.11 (corresponds to 68% Portland cement in our geopolymeric cement), after 2 days after mixing, our compressive strength is 8.5 MPa. This resistance is higher than specified limit applicable to each of the results for the resistance class 32.5R and 42.5R according to EN-197-1

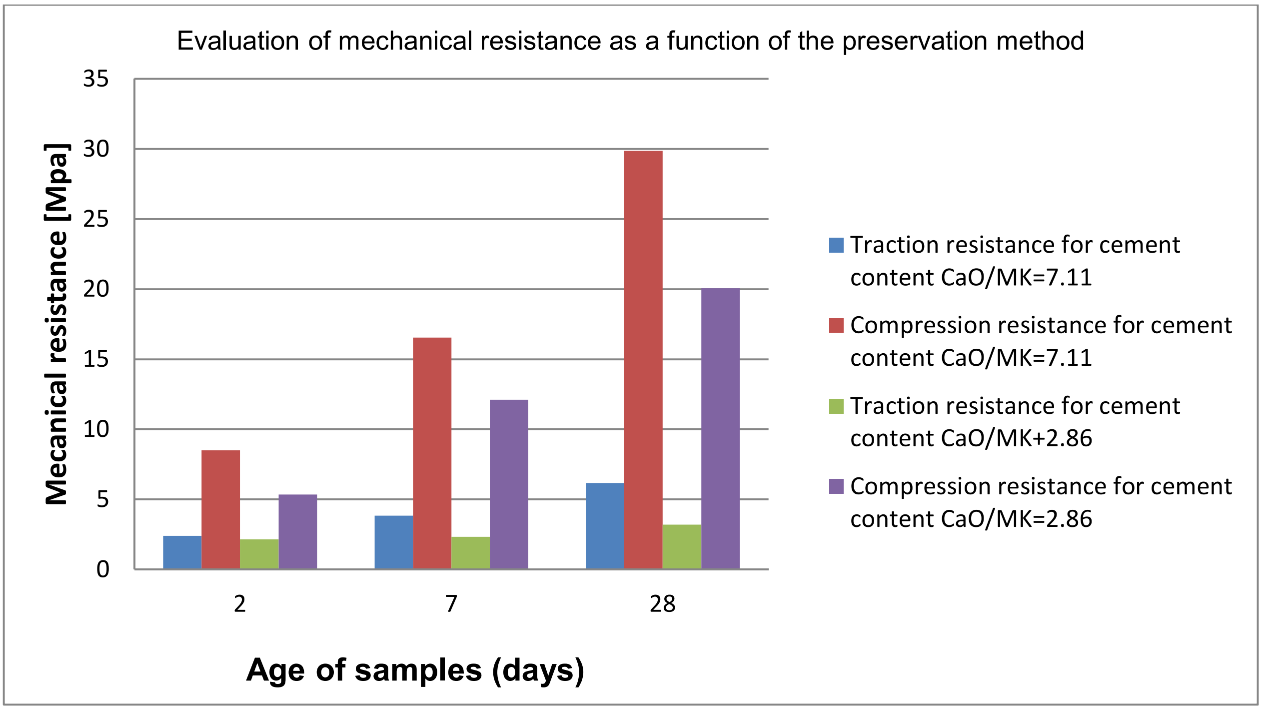

Figure 9: Evolution of mechanical strength as a function of Portland cement content (water conservation)

After 7 days of storage in water, the test pieces were crushed and the result was: Cs= 16.55 MPa. This is in perfect accordance with the mechanical requirements according to EN 196-1, for the short-term strength of strength class 32.5N.

Finally, the compressive strength reaches a maximum value of MPa in 28 days. This result complies with the limit value specified for the current strength class 32.5N, according to EN-196-1. This result allows us to obtain a cement of exploitable mechanical quality. As such, our second type of product will be used to make proprietary concrete and molded cements such as: breeze block, fibrocement tile, paving stone, claustra, pipe and especially for the construction of houses with less than 3 floors.

4.1.10) Conclusion on mechanical tests

In the first part we confirm that the mechanical test is the most important test for the study and development of the new binder.

Then, we mastered the parameters that directly influence the mechanical strength of our geopolymeric specimens, these parameters are: n SiO_2 / n Na_2O; n_T SiO_2 / n Al_2O_3 ; n CaO / n MK ; n SS / n MK, age and conservation of the samples. We have two types of cements based on the optimal compressive strength types: one for Cs=20MPa and the corresponding product will be dedicated to uses requiring the least mechanical performance, and the other for Cs= 30MPa will be used for structural construction.

Finally, other parameters can also improve our result, and which deserve to be studied carefully, these parameters are: control the incineration of the rice husk, the fineness of the metakaolin and the rice husk ash, the water content (W/C), the temperature of the soda silicate manufacture, the nature and granulometry of our experimental sand.

4.2) Chemical test results

In general, our chemical analysis results comply with the chemical requirements defined in terms of characteristic values according to EN-196-2, except for insoluble ones which have a very high percentage (30.55%), and they are formed by free silica, and especially by overcooked materials during the incineration of the rice husk, and this justifies our high silica content SiO_2 / n Al_2O_3 =4.5 during parameter optimization. Indeed, it is necessary to control the incineration of rice husks.

The following table shows us the chemical composition of our geopolymeric cement

Table 3: the chemical composition of our geopolymeric cement

Elements	Weight % of our geopolymeric cement	Chemical requirements according to EN 196-2
Loss on ignition at 975 °C	3.89	≤ 5%
Soluble SiO_2	21.77	
Insoluble in HCl	30.55	≤ 5%
Al_2O_3	16.28	
Fe_2O_3	1.34	
CaO	20.34	
MgO	2.9	
SO_3	1.38	≤ 3,5%
Free CaO	1.06	

4.3) Results of setting tests and stability

By successively groping the water to obtain paste of normal consistency and checking with the Vicat apparatus, we found a value of 35% for the mixing water and the corresponding setting times are:

- The start of the catch is 82 minutes away
- And the taking stops at 2.15 a.m,

We affirm that this result is in accordance with the resistance class at 32.5N and 32.5R.

The stability test was carried out using the Chatelier mould, which is filled with the normal paste and then enclosed from top to bottom by glass plates, and allowed to rest for 24 hours, then immersed in hot water at 100°C for 3 hours:

When measuring the spacing of the needles of the Chatelier, we notice that it is almost zero, this means that there is no increase in volume due to the presence of an excess of sulfate of free lime and/or MgO magnesia in the normal geopolymeric paste, therefore this result meets the conditions required by EN 196-3.

We would like to note that this result is in perfect harmony with the chemical analysis.

4.4) Result on the determination of fineness

- We found the following results:
- Apparent density= 0.94
- Specific weight=2.60 T/m3
- The specific surface Blaine= 7 638 cm2/g

First, we notice that the bulk density and specific gravity of our geopolymeric cement are much lower than that of Portland cement, the explanation is that rice husk ash and metakaolin are lightweight materials. While the Blaine specific surface area of our new binder is significantly higher than Portland cement (4.095 cm^2/g), this explains the high mixing water content of our normal paste (35%) compared to Portland cement (28%), because the greater the fineness, the greater the degree of hydration of the cement grows, and the greater the grinding fineness, the greater the compressive strength increases.

IV. Conclusion

The objective of this work is to identify and then control at the laboratory level and on small scale the parameters that influence the technical properties of a geopolymeric cement from metakaolin, Portland cement, rice husk ash and sodium silicate in order to contribute to its effective production.

To give the study of our new binder a relevant and standardized character, and especially to compare our results with international research work, we decided to carry out our tests at the National Laboratory of Public Works and Building (LNTPB) in Alarobia.

We have determined the most important parameters for obtaining a good quality cement: these parameters are: the molar ratio n SiO_2 / n Na_2O; n_T SiO_2 / n Al_2O_3 =4.5 , n CaO / n Metakaolin ; n soda ash silicate / n Metakaolin; the age of the samples: the method of sample storage.

The optimization of these parameters allowed us to have two optionals compressive strength at 28 days of storage in water, and this led us to two types of products:

- One for Cs=10MPa and the corresponding product will be intended for uses requiring less mechanical performance or simple masonry such as: plaster, mortar, soil stabilization, paving, etc.
- The other for Cs=30MPa, this result respects the limit value specified for the current strength class 32.5N, according to EN-196-1 standard, therefore our second type of product will be dedicated to the construction of structures, i.e. the manufacture of proprietary concretes and cast cements such as: block, fibrocement tile, pavers, claustra, pipes and especially to the construction of houses with less than 3 floors.

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