Experimental Research on New Sustainable Geopolymer Mortars Reinforced and Not Reinforced with Natural Fibers

Alberto Viskovic[1(✉)], Michał Lach[2], Łukasz Hojdys[3], Piotr Krajewski[3], and Arkadiusz Kwiecien[3]

[1] University "G. d'Annunzio" of Chieti-Pescara, Chieti, Italy
a.viskovic@unich.it
[2] Faculty of Materials Science and Physics, Cracow University of Technology, Kraków, Poland
[3] Faculty of Civil Engineering, Cracow University of Technology, Kraków, Poland

Abstract. The paper describes experimental research voted to verify the mechanical characteristics of very sustainable geopolymer mortars and to try to improve their mechanical characteristic using natural fibers.

Particularly, there were used and tested geopolymer mortars using a geopolymer matrix obtained from very cheap and sustainable fly ashes (as they come from the waste recycling). About the natural fibers, they were used hemp short fibers as they are very durable and sustainable.

It was designed and performed a bending test program for twelve reference samples of geopolymer mortar beams (without fibers) divided into two group of six samples with different proportions between fly ashes and sand. Then, it was designed and performed the bending test program for the geopolymer mortars with the addition of hemp short fibers: twelve samples with a lower percentage of fiber and twelve samples with a higher percentage of fibers. Each group of twelve samples was divided into two group of six samples with different proportions between fly ashes and sand.

Then it was designed and performed a compression test program: there were used the same beams, after their cracking in the bending tests, to obtain cubic samples (after a regularization of the faces). Thus, there were twenty-four reference cubic samples without fibers and forty-eight cubic samples with fibers for the compression tests.

Keyword: Sustainable mortars · Geopolymer mortars · Fly ashes · Natural fibers · Hemp fibers · Experimental laboratory tests · Mortars mechanical characteristics

1 Introduction

1.1 The Research Background Philosophy

To improve human sustainability on our planet, the buildings construction and the buildings use sectors are also involved.

In the last decades the attention was mainly oriented to the reduction of energy demand during the use of the buildings and to the reduction of a building footprint considering all its life, from the construction up to the demolition. But for the building footprint the attention was initially focused only on the non-structural materials and elements. It is time to consider that the structural materials are a big part of the total material used in a building.

In all the buildings' structural typologies, today in use, there is a large use of steel and Portland cement mortars and concretes. Both steel and Portland cement, have a large footprint in terms of CO_2 emissions during their production processes. Thus, improving of sustainability is directly related to a reduction of the use of steel and Portland cement. The Portland cement may be substitute with natural hydraulic cements (like the pozzolanic cement) or geopolymers. Geopolymers may be obtained starting from pozzolanic or tufa rock powder or by fly ashes produced in waste incinerators.

1.2 The Research Specific Target

The specific target of the research was to verify the mechanical characteristics of a geopolymer mortar and to try to improve their mechanical characteristic. For this last aim it was interesting to use a dispersion of short natural fibers.

Particularly they were used and tested geopolymer mortars using a geopolymer matrix obtained from very cheap and sustainable fly ashes (as they come from the waste recycling).

2 Background Knowledge

2.1 Materials Defined "Geopolymers"

The term "geopolymer" identifies a vast class of inorganic materials with a polymeric structure, (both synthetic and natural). In the broadest sense of the term, they are inorganic materials whose structure resembles that of classic organic polymers. However, the same term can also refer to ceramic materials formed by chemical reaction at relatively low temperatures (below 350 °C), whose mechanical resistance is comparable to that of many traditional high temperature consolidated ceramics [1].

Geopolymers are therefore synthetic materials which, obtained by chemical reaction between an activating solution and a reactive powder, are consolidated at low temperatures from the environment to a maximum of about 120 °C, obtaining a material with excellent chemical and physical properties and with a wide range of potential applications [2]. Depending on the raw materials, extremely different geopolymer products are obtained.

2.2 Historical Notes About Geopolymers

In the 40s of the last centuries, it was discovered that alkaline activation was an efficient method to accelerate the latent pozzolanic activity of aluminosilicate minerals rich in calcium such as blast furnace slag. In these systems the water-mineral interactions are

accelerated by the presence of alkali in the solution, with a consequent rapid hardening [3–5]. These "alkali-activated slag cements" or Trief, were used on a large scale in building materials until the early 1950s.

Between 1960 and 1970, Victor Glukhovsky, a scientist of Ukrainian origin active at the Kiev Institute of Civil Engineering (KICE, USSR), made the greatest contribution in the identification of hydrated calcium silicates and calcium and sodium alumino-silicates hydrates as phases which formed during the above process [6]. In his studies, he also noticed how the clays reacted during the alkaline treatment to give sodium aluminosilicates hydrates. Glukhovsky called the cements produced with this technology "silicate concrete soil" (1959) and the binders "cement soil" (1967).

Flint et al. [7], in 1946, at the National Bureau of Standards, developed an alumina extraction process starting from a bauxite with a high silica content, in which one of the intermediate stages involved the precipitation of a compound simil-hydrosodalite. Only three years later, Borchert and Keidel prepared hydrosodalite by reacting kaolinite with a concentrated solution of NaOH at 100 °C. In 1963, Howell achieved the synthesis of zeolite "A" using calcined kaolin instead of kaolinite. In 1969, Bessons, Caillér and Hénin [8], at the French museum of natural history in Paris, developed the synthesis of hydrosodalite starting from various phyllosilicates (kaolinite, montmorillonite and halloysite) at 100 °C in concentrated caustic soda.

The geopolymers properly named were developed only in 1976 by Joseph Davidovits. The great interest on these materials developed as a result of numerous catastrophic fires that occurred in France between 1970 and 1972; events amplified by the extensive use of common organic plastics. The development of non-combustible and non-flammable plastic materials thus became the main target of Davidovits' career, who developed aluminosilicate materials with amorphous to semi-crystalline networks, which he called "geopolymers" [9, 10].

These new materials saw their first application as fire resistant construction products, between 1973 and 1976, for example, as chipboard panels coated on both sides with geopolymer nanocomposite material produced in a single stage [11]. However, the real turning point came between 1978 and 1980, when the Davidovits' company developed a geopolymeric liquid binder by reacting a meta-kaolin with an alkaline silicate solution. The patent for this discovery was filed in 1979 and was called "Geopolymite" [12]. From the first Davidovits patent to today, interest in these materials has grown enormously, especially since the early 21st century, with a relative increase in scientific publications and congresses on the subject.

3 Experimental Research

3.1 Experimental Tests Program

It was designed a bending test program for twelve reference samples of geopolymer mortar beams (without fibers) divided into two group of six samples with different proportions between fly ashes (Fig. 1a) and sand. Then, it was designed the bending test program for the geopolymer mortars with the addition of hemp short fibers (Figs. 1b, 1c): twelve samples with a lower percentage of fiber and twelve samples with a higher

percentage of fibers. Each group of twelve samples was divided into two group of six samples with different proportions between fly ashes and sand.

Fig. 1. Fly ashes (a), different type of natural (not treated) hemp fibers (b) and (c).

Thus, it was also designed to use the same beams, after their cracking in the bending tests, to obtain cubic samples (after a regularization of the faces) for compression tests. The program was to have up to a maximum of twenty-four reference cubic samples without fibers and forty-eight cubic samples with fibers for the compression tests.

3.2   Sample Preparation

Two variants of geopolymer mortar samples with the addition of natural mixed fibers (dry) and reference samples without the addition of fibers were made. The samples were made in the form of 4 × 4 × 16 cm, 6 pieces of each batch:

1) samples based on fly ash (70%), general construction sand (30%);
2) samples based on fly ash (50%), general construction sand (50%);
3) samples based on fly ash (70%), general construction sand (30%) and natural fibers in the amount of 1% by weight;
4) samples based on fly ash (70%), general construction sand (30%) and natural fibers in the amount of 3% by weight;
5) samples based on fly ash (50%), general construction sand (50%) and natural fibers in the amount of 1% by weight;
6) samples based on fly ash (50%), general construction sand (50%) and natural fibers in the amount of 3% by weight.

All the samples solidification was activated with 10M r-r NaOH and sodium glass in a ratio of 2.5 by weight at a temperature of 75 °C for at least 16 h. Then the samples were disassembled and left in the same conditions (ambient temperature 20 °C and 50% air humidity) for 28 days of seasoning. In the case of samples with fibers, the fibers were added just after a first mixing with the activator solution.

3.3   Experimental Tests

There were carried out two typologies of test:

a) four-point bending test on beams 40x40x160mm^3, based on the standard EN 12390–5,

b) compression test on cubic samples (halves beam, 40x40x40mm^3) obtained cutting the cracked sample after the bending test.

There were calculated the mean and the characteristic values of strengths. The characteristic values were determined according to EC0 Annex D (values of kn factor are taken from Table D1).

Bending Tests. In Table 1 are reported the bending test results, for the reference samples based on fly ash (70%) and general construction sand (30%). Data are provided in terms of tensile strength by bending (f$._{\text{t.fl}}$) and the evaluations of the mean value (f$._{\text{t.fl.mean}}$), the standard deviation (SD) and the characteristic value (f$._{\text{t.fl.k}}$).
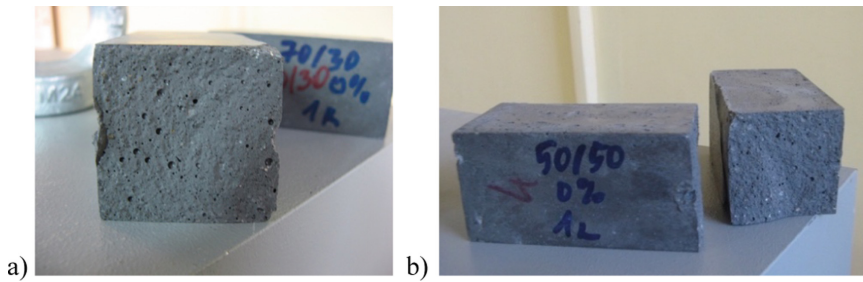
Table 1. Bending tests (1) reference samples. Fly ash (70%), sand (30%): tensile strength by bending (f$._{\text{t.fl}}$), mean value (f$._{\text{t.fl.mean}}$), standard deviation (SD) and characteristic value (f$._{\text{t.fl.k}}$)

Sample	F_{max}	f$._{\text{t.fl}}$	f$._{\text{t.fl.mean}}$	SD	CV	kn $_{EC0}$	f$._{\text{t.fl.k}}$
	N	N/mm^2	N/mm^2	N/mm^2			N/mm^2
7030_0p1	2014.375	4.72119					
7030_0p2	2530.448	5.93074					
7030_0p3	3530.756	8.27521					
7030_0p4	2565.255	6.01232					
7030_0p5	2390.918	5.60371					
7030_0p6	3415.918	8.00606	6.42	1.41	0.22	2.18	3.36

In Table 2 are reported the same bending test results for the reference samples based on fly ash (50%) and general construction sand (50%). In Fig. 2a and b are reported, respectively, the fracture surfaces in reference samples based on fly ash (70%) and general construction sand (30%) (a) and based on fly ash (50%) and general construction sand (50%) (b).

Table 2. Bending tests (2) reference samples. Fly ash (50%), sand (50%).

Sample	Fmax	$f_{.t.fl}$	$f_{.t.fl.mean}$	SD	CV	kn $_{EC0}$	$f_{.t.fl.k}$
	N	N/mm^2	N/mm^2	N/mm^2			N/mm^2
5050_0p1	4006.95	9.39129					
5050_0p2	3196.602	7.49204					
5050_0p3	3440.173	8.06291					
5050_0p4	3540.54	8.29814					
5050_0p5	3760.65	8.81402					
5050_0p6	3843.399	9.00797	8.51	0.69	0.08	2.18	7.00

Fig. 2. Fracture surface in a reference sample based on: fly ash (70%) and general construction sand (30%) (a); fly ash (50%) and general construction sand (50%) (b).

In Table 3 is reported the summary of the bending test results, in terms of tensile strength by bending mean value, standard deviation and characteristic value, for each different sample typology: "7030_0p" fly ash (70%), general construction sand (30%) and fibers (0%); "5050_0p" fly ash (50%), general construction sand (50%) and fibers (0%); "7030_1p" fly ash (70%), general construction sand (30%) and fibers (1%); "5050_1p" fly ash (50%) and general construction sand (50%) and fibers (1%); "7030_3p" fly ash (70%), general construction sand (30%) and fibers (3%); "5050_3p" fly ash (50%) and general construction sand (50%) and fibers (3%).

Table 3. Summary of the bending tests results.

Type of sample	$f_{.t.fl.mean}$	SD	$f_{.t.fl.k. (EC0)}$
	N/mm^2	N/mm^2	N/mm^2
7030_0p	6.42	1.41	3.36
5050_0p	8.51	0.69	7.00

(*continued*)

Table 3. (*continued*)

Type of sample	$f_{t.fl.mean}$	SD	$f_{t.fl.k.}$ (EC0)
7030_1p	7.30	0.87	5.40
5050_1p	8.31	0.41	7.43
7030_3p	6.70	0.35	5.92
5050_3p	7.76	0.99	5.59

In Fig. 3a, b, c and d are reported, respectively, the fracture surfaces in samples based on: fly ash (70%), general construction sand (30%) and fibers (1%) (a); fly ash (50%), general construction sand (50%) and fibers (1%) (b); fly ash (70%), general construction sand (30%) and fibers (3%) (c); fly ash (50%) and general construction sand (50%) and fibers (3%) (d).
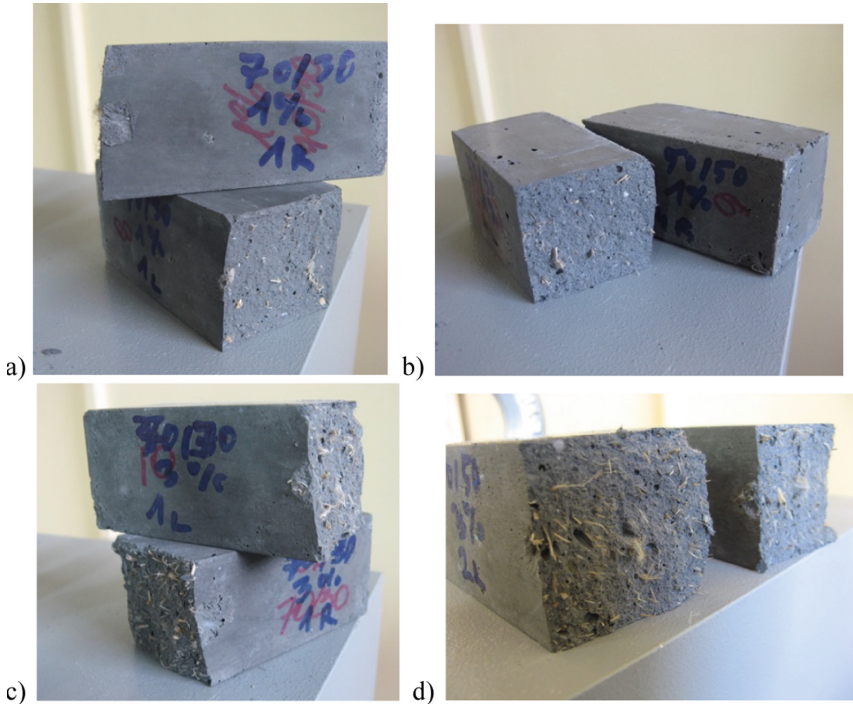

a)

b)

c)

d)

Fig. 3. Fracture surface in a sample based on: fly ash (70%), general construction sand (30%) and fibers (1%) (a); fly ash (50%), general construction sand (50%) and fibers (1%) (b); fly ash (70%), general construction sand (30%) and fibers (3%) (c); fly ash (50%), general construction sand (50%) and fibers (3%) (d).

Compression Tests. In Table 4 are reported the compression test results, for the reference samples based on: fly ash (70%) and general construction sand (30%). Data are provided in terms of compression strength (f_c) and the evaluations of the mean value ($f_{c.mean}$), the standard deviation (SD) and the characteristic value (f_{ck}).

The final letter L and R in the sample name simply indicate that they are obtained two sample, "half Right" and "half Left", from the same sample used in the previous bending test. The mean value, the standard deviation and the characteristic value are calculated on all the samples, L and R together.

Table 4. Compression tests (1) reference samples. Fly ash (70%), sand (30%): compression strength (f_c), mean value ($f_{c.mean}$), standard deviation (SD) and characteristic value (f_{ck}).

Sample	Fmax	f_c	$f_{c.mean}$	SD	CV	kn $_{EC0}$	$f_{ck\ EC0}$
	kN	N/mm^2	N/mm^2	N/mm^2			N/mm^2
7030_0p1L	89.27	55.79					
7030_0p1R	88.05	55.03					
7030_0p2L	100.02	62.51					
7030_0p2R	98.25	61.41					
7030_0p3L	93.65	58.53					
7030_0p3R	91.16	56.98					
7030_0p4L	99.18	61.99					
7030_0p4R	98.02	61.26					
7030_0p5L	97.71	61.07					
7030_0p5R	96.32	60.20					
7030_0p6L	90.87	56.79					
7030_0p6R	100.85	63.03	59.5	2.79	0.05	1.87	54.3

In Table 5 are reported the same compression test results for the reference samples based on fly ash (50%) and general construction sand (50%).

Table 5. Compression tests (2) reference samples. Fly ash (50%), sand (50%).

Sample	Fmax	f_c	$f_{c.mean}$	SD	CV	kn $_{EC0}$	$f_{ck\ EC0}$
	kN	N/mm^2	N/mm^2	N/mm^2			N/mm^2
5050_0p1L	99.52	62.20					
5050_0p1R	102.34	63.96					

Table 5. (*continued*)

Sample	Fmax	$f_{.c}$	$f_{.c.mean}$	SD	CV	kn_{EC0}	$f_{ck\ EC0}$
5050_0p2L	96.81	60.51					
5050_0p2R	103.59	64.74					
5050_0p3L	105.04	65.65					
5050_0p3R	97.92	61.20					
5050_0p4L	107.61	67.26					
5050_0p4R	103.42	64.64					
5050_0p5L	109.86	68.66					
5050_0p5R	108.14	67.59					
5050_0p6L	108.89	68.06					
5050_0p6R	102.96	64.35	64.9	2.68	0.04	1.87	59.9

In Fig. 4a and b are reported, respectively, the photo of the compression test on a reference sample based on fly ash (70%) and general construction sand (30%) (a) and based on fly ash (50%) and general construction sand (50%) (b).
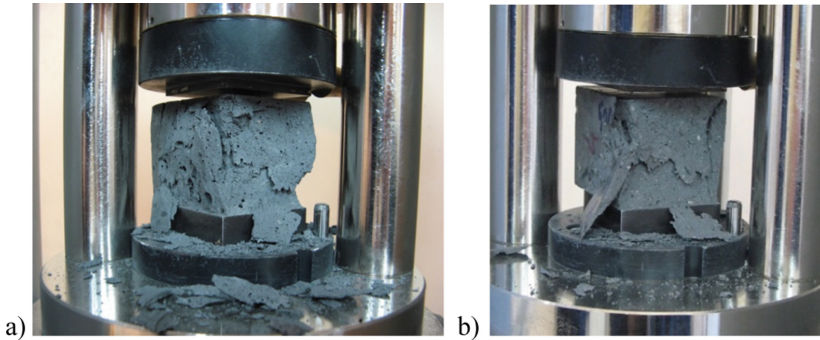

Fig. 4. Compression collapse in a reference sample based on: fly ash (70%) and general construction sand (30%) (a); fly ash (50%) and general construction sand (50%) (b).

In Table 6 is reported the summary of the compression test results for each type of sample typology, in terms of compression strength mean value, standard deviation and characteristic value.

Table 6. Summary of the compression tests results.

Type of sample	$f_{c..mean}$	SD	$f_{ck. (EC0)}$
	N/mm^2	N/mm^2	N/mm^2
7030_0p	59.5	2.79	54.3
5050_0p	64.9	2.68	59.9
7030_1p	54.7	4.49	46.3
5050_1p	53.8	3.84	46.6
7030_3p	41.7	3.36	35.4
5050_3p	43.9	2.73	38.8

In Fig. 5a and b are reported, respectively, the compression collapses in samples based on fly ash (70%), general construction sand (30%) and fibers (1%) (a); based on fly ash (50%), general construction sand (50%) and fibers (1%) (b).
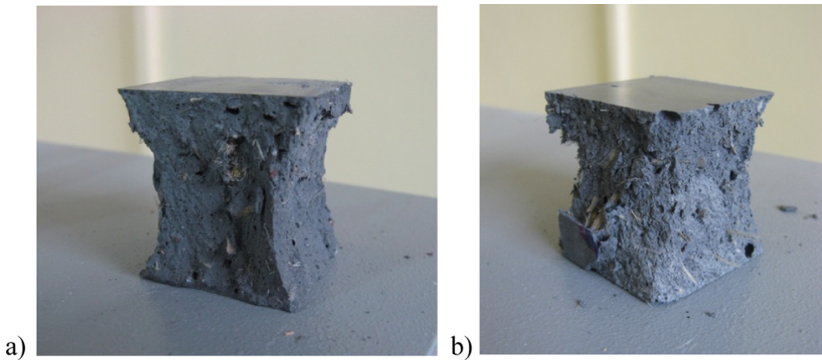

a)   b)

Fig. 5. Compression collapse in a sample based on: fly ash (70%), general construction sand (30%) and fibers (1%) (a); fly ash (50%), general construction sand (50%) and fibers (1%) (b).

In Fig. 6a and b are reported, respectively, the compression collapses in samples based on fly ash (70%), general construction sand (30%) and fibers (3%) (a); based on fly ash (50%) and general construction sand (50%) and fibers (3%) (b).

4   Observations on the Geopolymer Mortars Tests Results

The results show a general better performance in the case of 50% - 50% proportions between fly ashes and sand. Moreover, in the case of that 50%-50% proportions, the reference samples show a higher tensile resistance which is only 1/8,5 of the respective compression strength, against a ratio 1/16 (between tensile and compression strength) in the case of 70%-30% proportions between fly ashes and sand. But what is more noticeable, it is a very high compression strength in both cases: $f_{ck} = 54,3 N/mm^2$ (for
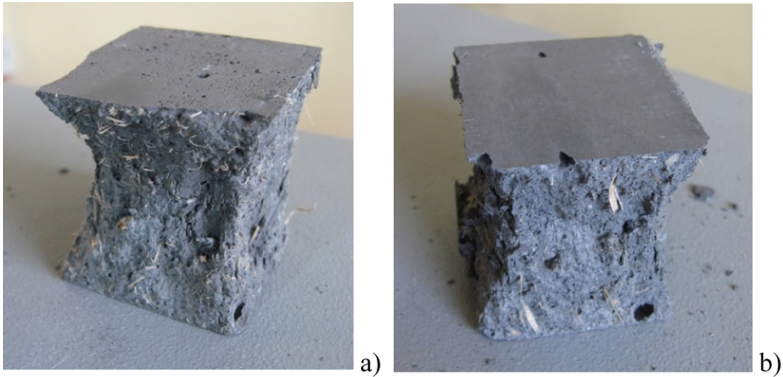
Fig. 6. Compression collapse in a sample based on: fly ash (70%), general construction sand (30%) and fibers (3%) (a); fly ash (50%), general construction sand (50%) and fibers (3%) (b).

70%-30% proportions) and $f_{ck} = 59,9 N/mm^2$ (for 50%-50% proportions), the double of the better traditional mortars. These geopolymer mortars may be considered very "super mortars" and named "M50" in comparison to the normal ones.

Adding a 1% of fibers by weight, there is an increment in tensile resistance in both cases of proportions between fly ashes and sand. On the contrary, adding a 3% of fibers there is an increment in tensile resistance in case of 70%-30% proportions, while there is a decrement in case of 50%-50% proportions. On the contrary to that was expected, adding hemp fibers, the compression strength decreases in any case: $f_{ck} = 46,3 N/mm^2$ and $f_{ck} = 46,6 N/mm^2$, respectively in case 70%-30% and 50%-50% proportions and 1% of fibers, while they are $f_{ck} = 35,4 N/mm^2$ and $f_{ck} = 38,8 N/mm^2$, respectively in case 70%-30% and 50%-50% proportions and 3% of fibers. This means that the increment in tensile strength don't give a confinement effect to the mortar (with a consequent supposed increment in compression strength) as it generally happens with normal mortars. These results may be explained with a different mechanical behavior of the geopolymer mortars respect to the traditional ones and it may be related to the higher performances of the binder basic geopolymer material.

In any case the geopolymer mortars with fibers have very high compression strengths in comparison to the traditional ones and may be considered "M40" in case of 1% of fibers and "M30" in case of 3% of fibers by weight. Moreover, there is a better ratio (respect to the normal mortars) between the tensile and compression strength: they are 1/8,5 and 1/6,3 respectively with 70%-30% and 50%-50% proportions between fly ashes and sand, with 1% of fibers; while they are 1/6 and 1/7 respectively with 70%-30% and 50%-50% proportions, with 3% of fibers.

5 Conclusions and Future Research

The results show as the geopolymer binder material has a very different behavior respect the Portland binder or the pozzolanic or others natural hydraulic mortar binders. The future research may follow some different paths and purposes:

a) to research of higher mechanical performances for the geopolymer mortars, without adding fibers,

b) to use geopolymer mortars as matrix for FRCM like reinforcements for historical masonries (to bond natural fiber fabric to masonry surfaces),

c) to search a better use of the short fibers, that may be useful to obtain lighter structural geopolymer materials.

In the (a) case, the research has to be oriented to find the better mix design in terms of proportions between fly ashes and sand and in terms of the better calibration of diameters of the sand granules or in terms of a good mix of different sand diameters; moreover, the research has also to find the better proportions among the activator, the fly ashes and the sand, in relation of the different porosity of different sands.

In the (b) case, using an improved geopolymer mortar (from case (a)), it may be interesting to study the debonding strength of FRCM like reinforcements.

In the (c) case, it may be useful to explore the field of prefabricated structural and non-structural elements.

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