Geopolymer mortar: metakaolin-based recipe for Cultural Heritage Application

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Abstract – **Many papers have been written about geopolymers and their application, but only few of them report about the recipe to obtain suitable product for Cultural Heritage application. As regards to the production costs of geopolymers, we realized that these are not, now, comparable with those of cement Portland mortars, but the possibility of obtaining products for specific applications, makes the geopolymers suitable for specialist uses in the field of Cultural Heritage despite their cost. Starting from a simple metakaolin-based geopolymer we would try to find the right ingredients and proportion between them to obtain a simple white geopolymer mortar suitable for restoration and reintegration purposes with physical and chemical characteristics like the commercial restauration standard products. In order to determine the mechanical and chemical properties, we follow up the processes with standard analytical techniques such as XRD on solid precursor and compressive strength on the final products.**

I. INTRODUCTION

Geopolymers are alkaline-activated aluminosilicate materials [1–7]. Even if geopolymer were known from the 50s, its applications have started to spread in recent years thanks to its low CO_2 [8] emission and the possibility of being made with waste materials from different types of industrial processes [9–17]. As a potential substitute for mortars and special cements, it immediately attracted interest for what concerns the possible applications in the field of cultural heritage and, although there is very little literature on the applicative potential, it seems to be interesting [18–22]. There is a large literature about geopolymers and choose a basic recipe for a self-made production of this material is not so trivial as reported in literature. Even choosing basic materials such as metakaolin [23], potassium silicate and sodium hydroxide as precursors, to understand how to put the components together and in what proportions is difficult to extrapolate even many papers reports their correct ingredients ratios. So, we decided to proceed empirically through several tests until we reach our recipe. Starting from this mix we have obtained a material that solidifies in about an hour at room temperature. At the end we have evaluated the mechanical properties of the material obtained.

II. MATERIAL AND METHODS

We have selected and used a metakaolin (MK) as alkaline source and a commercial Potassium Silicate (SiK) and Sodium Hydroxide (NaOH) as alkaline activators as reported in Table 1:

Table 1 The selected materials for the reported recipe.

Product	Specification
Metakaolin	Rock powder-Kaolin, calcinated at 1100°C
Alfa Aesar	Potassium Silicate, anhydrous, -48 mesh, SiO_2:K_2O 2.5:1 wt%
Honeywell Fluka™	Na(OH)

The Metakaolin source (MK) is a pure kaolin, calcinated at 1100°C in order to obtain a metakaolin. We have tested it by powder X-ray diffraction (pXRD) by mean of a Bruker D2 analyzer equipped with an X-ray tube with a copper anode (CuKα, λ = 0.154 nm). A scan of the metakaolin powder was performed at low and high angles (2θ = 5 ° - 65 °) with a rotation of the sample of 15 revolutions per minute (RPM). We found that there is some residual anhydrite probably coming from some gypsum present inside the original kaolin powder (Fig. 1). For evaluating the elasticity of the material, we have used the Shore C scale, used for rubber and polymeric materials. We have tested the sample with a Leeb durometer Proceq Equotip Piccolo 2 to measure indirectly the compressive strength too.
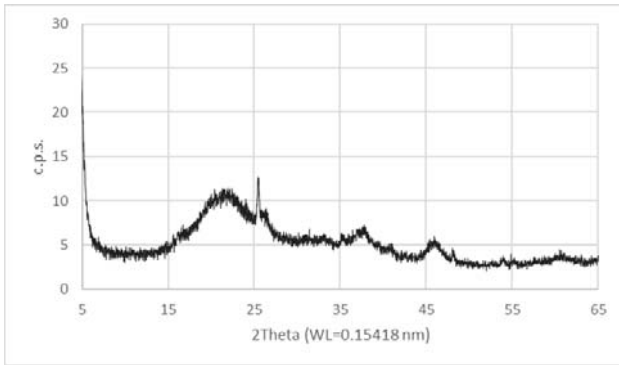
Fig. 1 Diffraction pattern of the metakaolin utilized for sample preparation.

Starting from these materials, we have prepared several samples from GS1 to GS5 mixing the components for about 15-20 minutes each one, by mean of a self-made mixer. In the Table 2, we report the formulation of each prepared samples.

Table 2 Formulation of the samples created.

Sample	MK (g)	Alkaline Solution (g)	SiK/Na(OH) ratio	Mixing water (g)
GS1	10.14	5.12	1:3.2	16.22
GS2	10.09	10.01	1:1.8	17.67
GS3	10.40	2.52	1:6.6	16.68
GS4	17.29	14.42	1:1	14.37
GS5	10.18	7.53	1:2.2	16.82

We have measured the weight-loss in function of the time with a Mettler Toledo PB4002-S/FACT for all the samples for a total of seven hours: GS1 (Fig. 3 and Table 3), GS2 (Fig. 4 and Table 4), GS3 (Fig. 5 and Table 5), GS4 (Fig. 5 and Table 6) and GS5 (Fig. 7 and Table 7). We report that after this period the sample GS1 was completely fractured and sample GS3 was not yet completely solidified Fig. 2. for this reason, we have decided to completely discard the aforementioned samples as they are not suitable for our purpose.
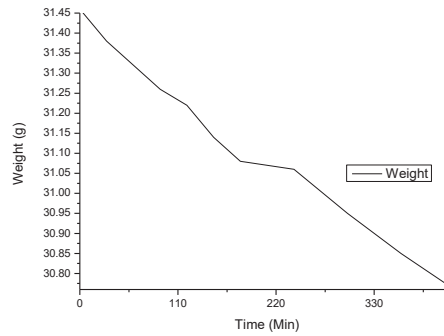

Fig. 2 On the left GS1, on the right GS3 (Ø= 4.9 cm).

Fig. 3 Weight-loss of the sample GS1.

Table 3 Weight-loss data of the GS1 sample.

T(min)	Weight (g)
0	31.46
30	31.38
60	31.32
90	31.26
120	31.22
150	31.14
180	31.08
240	31.06
300	30.95
360	30.85
420	30.76
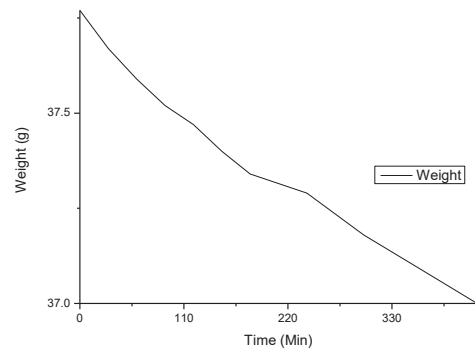
Fig. 4 Weight-loss of the sample GS2.

Table 4 Weight-loss data of the GS2 sample.

T(min)	Weight (g)
0	37.77
30	37.67
60	37.59
90	37.52
120	37.47
150	37.4
180	37.34
240	37.29
300	37.18
360	37.09
420	37.00
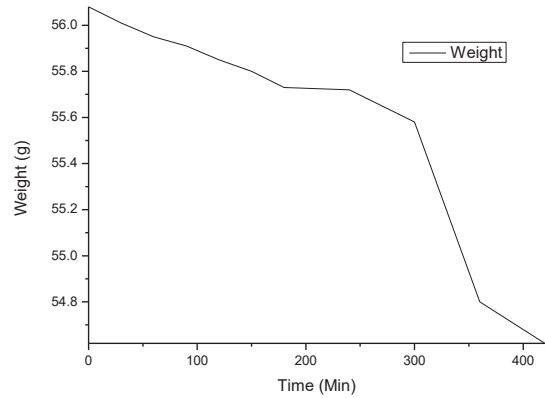
Fig. 6 Weight-loss curve for GS4 sample.

Table 6 Weight-loss data of the GS4 sample.

T(min)	Weight (g)
0	56.08
30	56.01
60	55.95
90	55.91
120	55.85
150	55.8
180	55.73
240	55.72
300	55.58
360	54.8
420	54.62
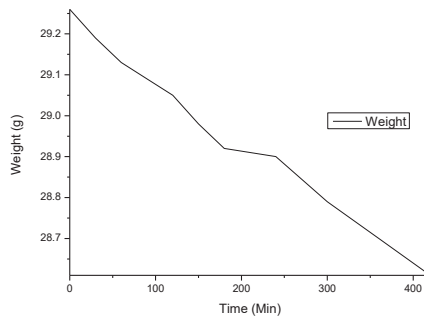
Fig. 5 Weight-loss of the sample GS3.

Table 5 Weight-loss data of the GS3 sample.

T(min)	Weight (g)
0	29.26
30	29.19
60	29.13
90	29.09
120	29.05
150	28.98
180	28.92
240	28.9
300	28.79
360	28.70
420	28.61
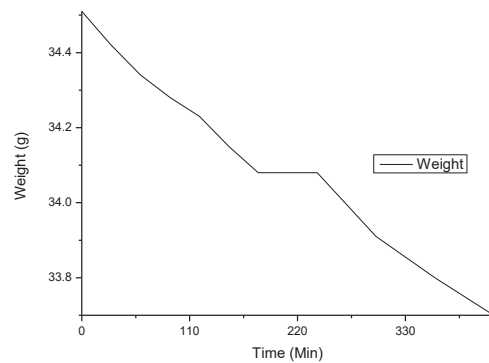
Fig. 7 Weight-loss of the sample GS5.

Table 7 Weight-loss data of the GS5 sample.

T(min)	Weight (g)
0	34.51
30	34.42
60	34.34
90	34.28
120	34.23
150	34.15
180	34.08
240	34.08
300	33.91
360	33.8
420	33.7

III.   DISCUSSION

As we can observe from the Fig. 3, Fig. 4, Fig. 5 and Fig. 7 these samples has a linear weight-loss in the first part, approximatively between 120 and 150 min there is a little slope followed by a plateau that continue till 250 min. After this period, the weight-loss results linear. The sample GS4 shows a slightly different behaviour: a linear weight loss till 180 min, a plateau between 180 and 240 min followed by a little weight loss from 240 to 300 min. After that there is a weight-loss with an hight slope value till 360 min followed by a less pronounced weight loss till 420 min.

IV.   CONCLUSION

Using a sol/gel made by SiK+Na(OH) help to slowing the hardening of the MK-geopolymer, the most critical things in the production of such kind of materials is the control of the water (mixing water) that is not so simple. As regards the recipe for a simple white metakaolin-based geopolymer we have found the mechanical properties reported in Table 8. For measuring the elasticity of the geopolymer we have considered the Shore C scale used in rubber and polymeric materials.

Table 8 The ratio and hardening time of our white MK-based geopolymer:H_t= Hardening Time; HC=Shore C Hardness;HRB=Leeb hardness.

Sample	H_t(min)	HC	Δ_s	HRB	Δ_s
GS2	120	91.5	1.2	535	14
GS4	60	97.7	0.9	484	6
GS5	150	99.4	0.7	412	32

Using a conversion table from the Leeb hardness value, we have obtained compressive strength values that are good for a material suitable for restoration (Table 9).

Table 9 Compressive strengt obtained from conversion table from Leeb HRB values. Σ_C=Compressive strength.

Sample	Σ_C (kg/cm$_2$)	Δ_s
GS2	24.1	1.4
GS4	19.5	0.5
GS5	14.6	1.9

Even if GS4 recipe seems to be the best considering the homogeneity (if we consider the Δ_s as an approximative indication of such parameter), we think that GS2 recipe is the best ones: it shows a good value for compressive strength elasticity with a good homogeneity.

V.   AKNOWLEDGEMENT(S)

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