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COMPARISON BETWEEN LEEB AND KNOOP HARDNESS ON METAKAOLIN-BASED GEOPOLYMERS

Abstract - S. Pagnotta, M. Lezzerini, *Comparison between Leeb and Knoop hardness on metakaolin-based geopolymers.*

Geopolymers represents a relatively new material, for sustainable building and Cultural Heritage restoration, that are of greatest interest in the field of Earth Sciences, not only because they are made starting from natural inorganic materials, but also for their peculiar mechanical features and for a low CO_2 emission. In the present work, it was experimented the use of a portable Leeb D instrument to determine the microhardness of some metakaolin-based geopolymer specimens by comparing its results with those obtained by the Knoop microdurometer. The Knoop test is indicated by the ASTM standards for determining the hardness of advanced ceramic materials. In our idea, the method can be used for a fast *in situ* measurement of the hardness properties of the materials, to be subsequently verified after sampling with laboratory measurements.

Key words - geopolymer, metakaolin, alkaline activator, Leeb D, Knoop microhardness

Riassunto - S. Pagnotta, M. Lezzerini, *Confronto tra la durezza di Leeb e Knoop di geopolimeri a base di metacaolino.*

I geopolimeri rappresentano un materiale relativamente nuovo, per architettura sostenibile e restauro del patrimonio storico, di grande interesse nel campo delle Scienze della Terra, non solo perché sono fabbricati a partire da materiale inorganico naturale, ma anche per le loro peculiari proprietà meccaniche e per la bassa emissione di CO_2. In questo lavoro si è sperimentato l'uso di uno strumento portatile Leeb D per determinare la microdurezza di campioni di geopolimeri a base di metacaolino, in rapporto a quella misurata con microdurometri Knoop. Gli standard ASTM indicano il saggio di Knoop per la determinazione della durezza di materiali ceramici avanzati. Nella nostra proposta, il metodo può essere usato per una rapida misurazione *in situ* delle proprietà di durezza dei materiali, da sottoporre a successiva verifica tramite analisi di laboratorio su campioni dei materiali.

Parole chiave - geopolimeri, metacaolino, attivatore alcalino, Leeb D, microdurezza di Knoop

Introduction

Among the new materials, geopolymers are those that have aroused great interest from the building sector for their innovative method of preparation, for the use of raw materials from industrial waste materials and for their low CO_2 emissions. Davidovits creates the term and start to use it from the end of '70 (Davidovits, 1982, 1989, 1991, 2002, 2015; Joseph Davidovits & Cordi, 1979) even if similar materials with the name of 'soil-cements' was introduced by Russian scientists in 1950s (Gluchovskij, 1959). The physical and mechanical properties are very good and someone considers them as a future 'green substitute' of Portland cements (Okoye, 2017). In general, a geopolymer is made up of two fundamental elements: a solid precursor and an alkaline activator. Among the most used solid precursors there are fly ash, blast furnace slag, red mud and metakaolin, while the alkaline activators consist of solutions of sodium silicate, potassium silicate and hydroxides of an alkali metal or alkali earth metal (Dadsetan *et al.*, 2019). Generally, the pH of these solutions is kept between 11 and 12 (Gharzouni *et al.*, 2015; Phair & Van Deventer, 2001; Weng *et al.*, 2005). Geopolymerization process starts from the dissolution of the aluminosilicate in an alkaline solution, subsequently siloxanes (Davidovits, 1978) and, therefore, poly-siloxanes are arranged in a disordered lattice until reaching long amorphous chains in which elements such as sodium and potassium can take place into the bond between the silicon and aluminium tetrahedra to balance the charges brought by oxygen (Khale & Chaudhary, 2007). Although these are long disordered chains, the packing of these tetrahedra after the curing period makes the material capable of reaching very high compressive strengths even over 100 MPa (Heah *et al.*, 2011; Mo *et al.*, 2014; Patil *et al.*, 2014; Rovnaník, 2010). Further interesting properties of these materials concern the possibility of making them acoustically and thermally insulating (Zhang *et al.*, 2015).

With the aim of using geopolymers as materials for restoration, we tested a non-destructive method that can be exploited for hardness measurements directly *in situ*. The use of portable instruments already known in the geological (Alberti *et al.*, 2013; Anan, 1997; Aoki & Matsukura, 2008; Kawasaki *et al.*, 2002; Verwaal & Mulder, 1993; Wilhelm *et al.*, 2016; Yılmaz, 2013) and metallographic (Leeb, 1979) field such as Leeb D

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(HLD) could be useful for verifying its hardness directly on the applied material and verifying its mechanical stress over time. The choice of this instrumentation type rather than a more conventional Schmidt hammer (Cargill & Shakoor, 1990; Haramy & DeMarco, 1985; Katz *et al.*, 2000; Kolaiti & Papadopoulos, 1993; Poole & Farmer, 1980; Sachpazis, 1990) is dictated by the fact that it could cause decreases in the strength of the material and cause micro-fractures, as demonstrated by an interesting work by Kovler et alii (2018), which could lead, as a not negligible secondary effect, to an accelerated degradation of the material. As for the hardness reference measurements, we decided to use Knoop hardness, the most used one for advanced ceramic materials hardness determination, which are excellent for a measurement on polished samples, but do not lend themselves to being used directly on site. In conventional hardness tests, the surface of solid specimen is indented by a tip of various shapes and the imprint left on the surface of the material is measured. The most used tips are those for Knoop (HK) and Vickers (HV) hardness testing. The hardness of the material is given by the ratio between the applied load P and the surface of the impression left by the indenter on the surface of the specimen. Knoop hardness test use a lozenge-based pyramid with two different semi-apex angles (86°15′ along the length L and 65° along width b) as schematized in Fig. 1.

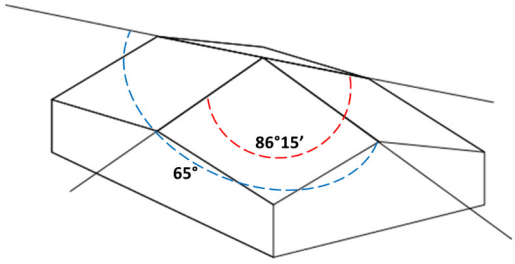

Figure 1. Geometry of the Knoop indenter.

The projected contact area (AP) is determined using the measured length of the indent by knowing the theoretical relationship between the length and the width expressed by the formula (1):

$$HK = 14.229 \cdot \frac{P}{L^2}$$

where L is the measured (in mm) long diagonal of the residual impression and P (in N) is the applied load.
We know from several studies (Ben Ghorbal *et al.*, 2017; Chicot *et al.*, 2007; Gong *et al.*, 2002; Mukhopadhyay *et al.*, 1990; Ullner *et al.*, 2001, 2002) that even if the hardness of a material is an intrinsic property, the Knoop hardness is lower than Vickers one. Several comparative studies on some materials report that the ratio between HV / HK for hard materials is be between 1.05 and 1.15, while negligible for materials as demonstrates by Gong et alii (2002) on different silicon nitride ceramic samples. The difference has been attributed by Marshall *et al.* (1982) to a different Knoop mark elastic response, which is more pronounced for harder materials. For a more exhaustive explication, we refer to Ben Ghorbal *et al.* (2017) where many aspects and some interesting conclusions are exposed and discussed.
While directly comparing a volume measurement such as the Leeb D with surface measurements such as the Knoop microhardness is not possible, our aim was to test whether a trend in hardness can be identified with a non-invasive and non-destructive portable instrument that may be indicative of the current physical conditions of the material in order to subsequently plan any sampling for laboratory testing and / or replacement in place of the material.

Material and methods

For the purposes of this study, we have made twelwe metakaolin-based geopolymer samples. Starting from commercial products both for metakaolin solid precursor (MK) and alkaline activator (AA). We have prepared four sets of samples with different recipes, ranging from 1.8 to 3.0 for SiO_2/Al_2O_3 ratios, from 0.4 to 0.8 for Na_2O/Al_2O_3 ratio and from 11.5 to 17.7 for H_2O/Al_2O_3 ratio. Among these recipes, we have chosen the end-members and the central one as listed in Tab. 1.

Table 1. Sample recipes.

Sample ID	SiO_2/Al_2O_3	Na_2O/Al_2O_3	H_2O/Na_2O
A1	1.8	0.6	13.3
A6	2.1	0.6	13.3
A11	2.4	0.6	13.3
B1	1.8	0.6	17.6
B11	2.4	0.6	17.6
B21	3.0	0.6	17.6
C1	2.0	0.4	17.7
C11	2.0	0.6	15.5
C21	2.0	0.8	14.4
D1	2.3	0.6	11.5
D11	2.3	0.6	12.5
D21	2.3	0.6	13.5

For the 1st and 2nd sets of samples (A and B respectively), we have fixed the Na_2O/Al_2O_3 and H_2O/Na_2O ratios, changing the SiO_2/Al_2O_3 ratio; for the 3rd series (C), we fixed the SiO_2/Al_2O_3 ratio, changing the Na_2O/Al_2O_3 and H_2O/Na_2O ratios; for the 4th series (D), we have fixed the SiO_2/Al_2O_3 and Na_2O/Al_2O_3 ratios, changing the H_2O/Na_2O ratio.
As we can see from the Fig. 2, some samples (A1, B1, B11, C11, C21) at 14 days result not yet hardened, while the same samples at 28 days results perfectly solidified similarly to the others.

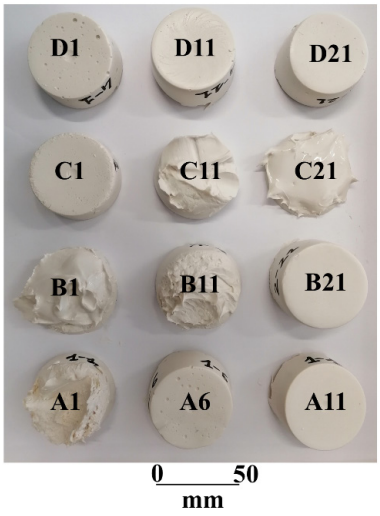

Figure 2. Selected samples used for this study, photographed 14 days after their production.

First, we carried out the XRD analysis on the solid precursor to verify the presence of crystalline (i.e. kaolinite, halloysite, dickite) or amorphous (metakaolin) phases. We have used an X-ray powder diffraction system, Bruker D2 analyzer, equipped with an X-ray tube with a copper anode (CuKα, λ = 0.15418 nm). A scan of the sample in the 5 - 65° 2θ range was performed, and the sample was run at 15 revolutions per minute (rpm).
To test the specimens setting, we have used direct empirical observation and a Shore C durometer, usually used to test medium-hard rubber and plastics in a scale range that vary from 0 to 100. For the application of Shore C durometer (HC), we referred to ASTM D2240-15 (ASTM, 2015) standard, repeating the measure for ten times. For Leeb D testing, we have used a Proceq Equotip Piccolo 2 with D mass, usually suitable for metals and alloys hardness testing. As regards the method used, we referred to ASTM A956/956M-17a (ASTM, 2017) standard, repeating the measurement for ten times for each specimen and discarding the first three measurements for a total of seven measures for each specimen. We have used a durometer Leitz for Knoop microhardness measurements with a 490.3 mN load. Considering geopolymer like very homogeneous ceramic material, we have follow the ASTM C1326-13 (ASTM, 2018) standard for Knoop testing of advanced ceramics, polishing each specimen on the cross-section and performing ten indentation for each of them (Tab. 2).

Table 2. Summary of standards and number of measurements.

	Normative	N° measurements
Shore C	ASTM D2240-15	10
Leeb	ASTM A956/956M-17a	10 (7)
Knoop	ASTM C1326-13(2018)	10

Results and discussion

XRD analysis performed on the solid precursor MK, formed by calcining kaolinite at 750 °C, and analyzed samples permitted to verify their amorphous structure both in the solid precursor (MK) and in final geopolymer samples (A1, A6, A11; B1, B11, B21; C1, C11, C21; D1, D11, D21) (Fig. 3).

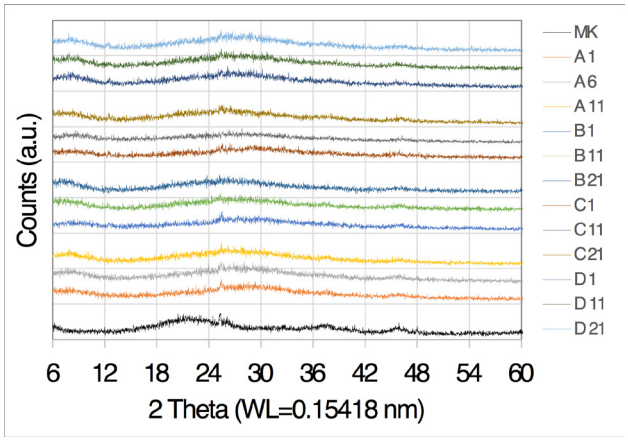

Figure 3. XRD pattern of raw solid precursor (MK) and final products (A1, A6, A11; B1, B11, B21; C1, C11, C21; D1, D11, D21).

By some empirical observation (opacity, elasticity, softness), we have found that samples Group A and Group D were already hardened after about one day. For Group B and Group C, the hardening of samples B1 and C1 was already almost occurred before 7 days, with a constant trend, samples B2 and C2 harden slowly and samples B3 and C3 began to harden after 14 days. The C-type Shore durometer allowed us to check these trends at 7 and 14 days (Fig. 4).

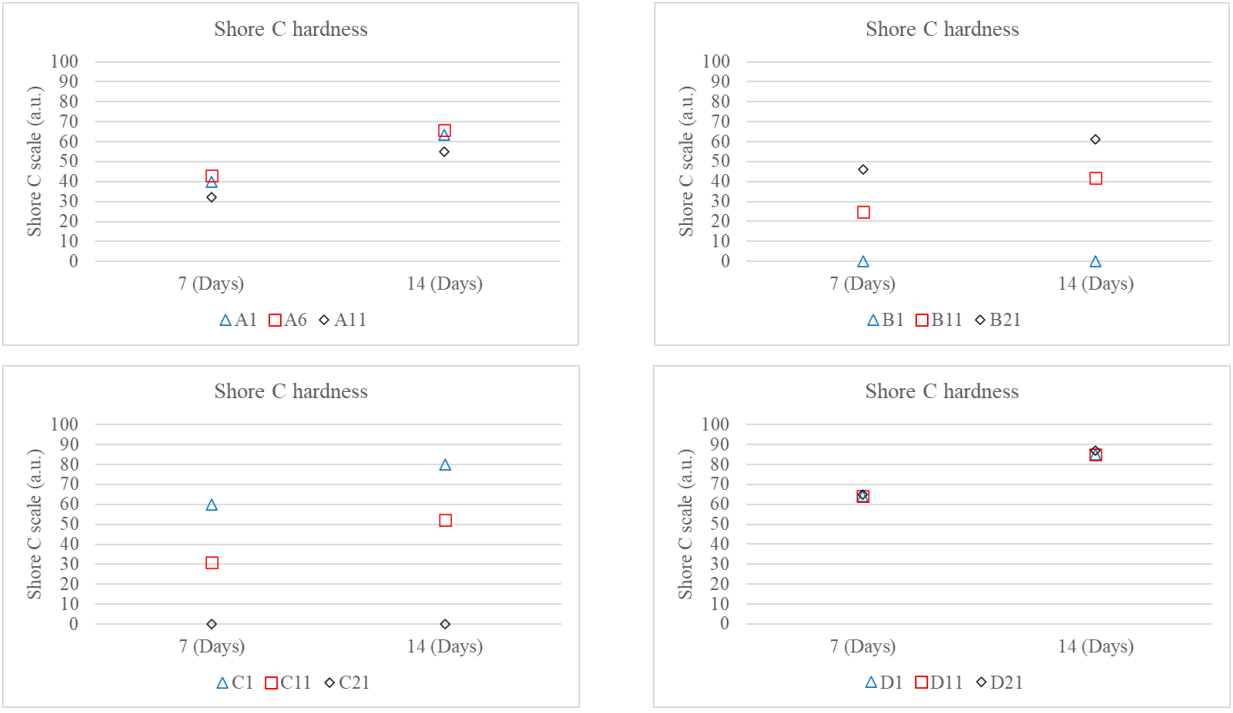

Figure 4. Hardening trends of the samples (7 and 14 days), according to Shore C durometer scale. The zeros value for both samples B1 and C21 means that the HC values are not measurable at 7 and 14 days.

After 14 days, the geopolymer samples are almost all hardened, except for B1 and C21 that starts their hardening after 14 days, and their macroscopic characteristics make them like highly homogeneous advanced ceramic materials. The Knoop microhardness results, taken at 28 days, show a trend similar with those of the Leeb D test (measured at the same time) carried out without sample preparation (Tab. 3).

Table 3. Summary table of HLD and HK results.

Sample	HLD	HK (MPa)	L (mm)	PK (N)
A1	475	172.6	0.201	0.4903
A6	552	327.1	0.146	0.4903
A11	562	355.7	0.14	0.4903
B1	514	133	0.229	0.4903
B11	554	177.8	0.198	0.4903
B21	632	250	0.167	0.4903
C1	541	400.1	0.132	0.4903
C11	526	179.7	0.197	0.4903
C21	512	17.4	0.633	0.4903
D1	552	309.9	0.15	0.4903
D11	533	282.9	0.157	0.4903
D21	510	265.7	0.162	0.4903

The 1st set of samples (A) shows same trends for portable Leeb D and micro-Knoop values (Fig. 5A and 5B): the increasing ratio of SiO_2/Al_2O_3 produces an effect of increase the HLD and HK of the material. As demonstrate by the 2nd set of samples (B) with a little higher ratio of H_2O/Na_2O, the increasing of SiO_2/Al_2O_3 produces no significative change in the trend as shown in Figures 5C and 5D. Fixing the SiO_2/Al_2O_3 ratio and increasing the Na_2O/Al_2O_3 ratio at the same time decreasing the H_2O/Na_2O ratio produce a drop in HLD and HK of the material (Fig. 5E and 5F) as shown in Group C trend. A decreasing of the HLD and HK as shown by trends of the sample group D (Fig. 5G and 5H) was produced by fixing the SiO_2/Al_2O_3 ratio and the Na_2O/Al_2O_3 ratio, while increasing the H_2O/Na_2O ratio.
HLD and HK correlation values are as follows:

$HLD = 2.0434 \cdot HK - 796.08$ $R^2 = 0.9983$
$HLD = 1.0015 \cdot HK - 379.43$ $R^2 = 0.9973$
$HLD = 13.353 \cdot HK - 6826.8$ $R^2 = 0.9945$
$HLD = 1.0078 \cdot HK - 248.25$ $R^2 = 0.9792$

The collected hardness data results in a linear correlation and in almost perfect agreement with R^2 better than 0.97.

Figure 5. Trends comparison of HLD and HK values in group samples (vertical bars represent the standard deviation on the set of measures).

Conclusion

The portable Leeb D hardness test results suitable for an accurate and fast analysis of geopolymers. In fact, this technique, although initially developed for testing metallic materials, demonstrated an interesting practical application in the field of constructions and it can quantitatively characterize the geopolymer hardness. Furthermore, the Leeb D durometer seems to be an instrument more sensitive to hardness variation than the Knoop or Vickers ones. The comparison of the two tests allowed us to hypothesize that even in the case of samples that show structural inhomogeneities, the rebound-based test (Leeb D) seems to be effective in giving a useful data, while the indentation-based test (Knoop) is more sensitive to inhomogeneities (i.e., the presence of microfractures). The advantage of using the Leeb D test, in addition to its obvious portability and no need for sample preparation, results in the possibility of observing directly in situ, hardness trends that are similar to that measured with Knoop microhardness carried out in laboratory.

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