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DISS AND ALFA PLANT FIBERS POTENTIAL INTO SLAG/GLASS POWDER METASILICATE-ACTIVATED BINDERS

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Abstract. Inorganic polymeric Alkali-Activated Binders (AABs) is a promising sustainable alternative for concrete technology today. In this work, alkali-activated vegetable fibers (VFs) with glass powder and slag (GP/S) were studied. The main objective of our study was to assess the engineering properties of GP/S mixtures with different native Algerian plant fibers: Alfa (A), Diss (D) along with Polypropylene fibers as reference fibers for comparison. Hence, Six mortars formulations were studied MC, MS, MSG, MSGA, MSGD and MSGP have been made. The conducted tests were flow, mechanical strengths, density/water porosity properties and thermal conductivity. The pastes were also tested by means of FTIR and SEM observations. The results show higher strength of Alkali-activated formulas especially at later ages in compression; lower mortar flow with AABs; density, porosity and thermal conductivity were slightly affected by the fibers. SEM observation exhibit mainly the formations of CSH and (C,N)ASH as hydration products.

Keywords: *AABs, Alfa/Diss vegetable fibers, Polypropylene, physical properties, microstructure*.

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1. Introduction

In our modern world, sustainable development is become a public concern. Thus, all industrial sectors turn to low carbon footprint solutions to mitigate global warming (Dinneen, 2023). Accordingly, construction area reuse and recycle with waste-based materials are becoming popular. As they are widely available and renewable, bio-based fibers composite or natural fibers (NF) may be a sound solution to lower the carbon footprint of the construction sector. Yet, this solution still needs more investigations to be accepted and standardized by construction specifiers.

Plant fibers are lignocellulosic composites of cellulose, hemicellulose, lignin and pectin (Babu, 2012). According to literature (24chemicalresearch, 2023), the global

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natural fiber market was valued at US$ 61760 million in 2022 and projected to reach US$ 67340 million by 2029. For their annual production worldwide, there are no accurate statistics about VF production for yet. While some workers report 4 billion tones (Rowell, 2008; Stokke *et al*., 2013), others state only 30 million tones (Azammi *et al*., 2020). However, plant fibers are available worldwide. Actually, they are used conventionally in textiles, polymer matrix, insulation materials, pharmaceutical carries (Cook, 2001; Mohanty *et al*., 2005). The history of use and benefits of plants fibers is well documented (Cook, 2001; Rowell, 2008; Shanks, 2014).

In addition to their renewable aspect and low cost, VF technically enhance toughness of materials prone to microcracking (Lv *et al*., 2023) and some physical properties of the recipient matrix. So, they should be very competitive to traditional high-cost energy consuming fibers (steel and synthetic).

Literature records on using VF in cementitious materials have flourished in recent years. Concerning cementitious materials, the most studied aspects are: necessity of appropriate treatment of VF; the influence of fiber morphology, chemical composition and optimum content. Generally applied treatment are mercerization (alkali) (Poletanovic *et al*., 2021; Ribeiro *et al*., 2024), cross-linking, acetalization, dyeing, cyanoethylation, bleaching. The goal of such treatment is the removal of wow, impurities and lignin recover materials (Rippon & Evans, 2020). Indeed, it is proven previously that the fibers covering raw coating cause weak interfacial transition zone (ITZ) and the properties of the mixture are affected (Achour *et al*., 2017; de Azevedo *et al*., 2021).

First, workability is reported to be reduced in the presence of the fibers (Afriansya *et al*., 2023; Rippon & Evans, 2020) because fibers make a physical barrier against materials flow and absorb water at the fresh state, especially with higher contents.

Second, porosity and bulk density of cemented materials records state a proportional decrease of density and increase of porosity with vegetable fibers, due to their low raw fiber's density (Amran *et al*., 2022; Khan *et al*., 2018) and eventually to the created porosity around the fiber during the mixing process (Abdelhak, 2017).

Third, for mechanical properties, there is a lot of work with some adverse findings. Scholars in references (Khan *et al*., 2018; Ribeiro *et al*., 2024) state a reduction of compressive strength with VF (Terai, 2023), but Alshaaer et al. (2017) record improved values. Also, similar or better to OPC flexural strength are documented in literature (Frydrych *et al*., 2019; Lazorenko *et al*., 2020). Those findings are explained by the variability of mixture composition, fiber nature, morphology and fiber treatment method.

Hence, the main objective of this work is to assess some mortars' performance based on two abundant natural fibers in Algeria (Diss and Alfa) and waste glass. We tested for mortars mechanical properties, porosity and thermal conductivity. In addition, microstructure observation of the pastes was carried out to get more information's needed to explain the macro properties. Scientifically, we are willing to address the nature of bonding in the case of NaOH treated grass fibers.

Diss and Alfa are two grass fibers abundant in Mediterranean countries and poorly known and valorized (Kaid-Harche & Djabeur, 2020). Their prepared fibers are investigated in OPC matrix by some authors. Sellami et al. (2013) with distillation and waterproofing treatments found weak mechanical properties of Diss based mortars; Bourahli and Osmani (2013) state 37 % better tensile strength with Diss; Kozlowski et al. (2020) found high records with 8% of Alfa substitution in NaOH geopolymers. Chiker et al. (2021) take note of good performance of CaOH treated Diss and Alfa in metakaolin bases mortars.

In fact, regarding the use of those fibers in geopolymers and alkali-activated materials there is little work on Alfa and even little work on Diss plant.

About the work significance, the authors believe the findings of the present study may provide a good support tobio-based fibers researchers in alkali-activated materials. In particular, it highlights a promising solution to construction sector in Algeria and Mediterranean countries to develop Alfa and Diss bases cementitious materials for application as structural elements or claddingand roofing's materials.

2. Materials and methods

The conducted methodological campaign is presented here. The different formulations are as follow: MC (the cement-based mix as a control mixture); MS: Alkali-activated slag mix, MSG (Activated slag/Glass waste powder) and MSGA, MSGD and MSGP are alkali-activated mixes with Alfa, Diss and Polypropylene respectively. The pastes' series included: C (cement), S (slag) and SG (slag/Glass) samples.

2.1. Materials

A standardized normal sand was used; its characteristics match ASTM C778 Standard (2021). Waste Glass powder (G) collected was from a local Algerian glass fabricant (AFRICAVER). It was finely grinded until a median particle size around 100 μm. An Algerian-made blast furnace granulated slag (BFS) was used from El-Hadjar Steel factory. The mineral SEM observation and physicochemical characteristics are presented in Figure 1 and Table 1.

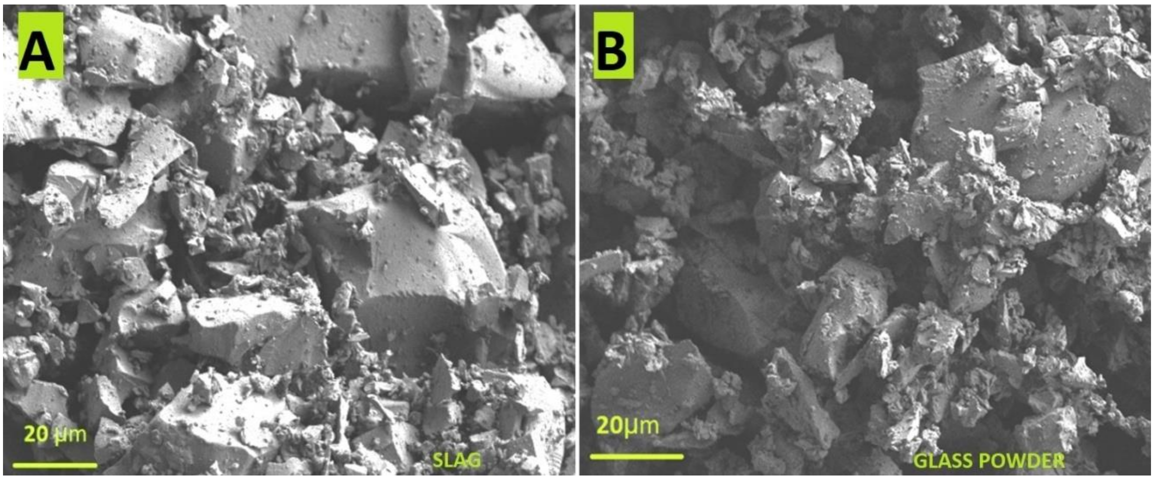

Figure 1. Precursors minerals in microstructural scale a) slag; b) glass powder

In addition, two vegetable grass-type fibers (VFs) were considered.

Alfa fibers (Esparto grass) from Alfa grass belong to Graminacies family, an abundant plant in ride areas in Algeria; called scientifically *Stipa Tenacissima L*. Diss fibers were prepared from Diss plant (Stramma, Mauritanica grass, rope grass) (Kozłowski *et al*., 2020), *Ampélodesmos Mauritanicus*, also a wild grass of the family of Poaceae (Abdelhak, 2017) in the North Africa and Southern Europe.

Table 1. Physico-chemical main characteristics of mineral powder materials

Oxide compositions (%)	OPC	BFS	GP
CaO	64.3	47.3	13.4
$CaCO_3$	-	-	-
SiO_2	19.9	34.5	70.1
Al_2O_3	5.1	6.45	1.71
MgO	0.9	3.85	0.520
MnO	-	2.18	-
Na_2O	0.25	0.203	13.4
FeO_3	3.3	1.57	0.331
K_2O	0.85	0.724	0.282
SO_3	3.2	1.46	0.246
TiO_2	-	0.320	-
Physical properties			
Blaine area (m^2/g)	4200	2795	2876
Density (g/cm^3)	3.1	2.35	2.5

Polypropylene (P) fibers were used in this study to be compared with VF findings.

The VF and P fibers in their natural forms are shown in Figure 2 and their surface under microstructural observations are exhibited in Figure 3. It is important to highlight the initial fibers preparation. So, those VF were first dried in natural conditions until getting a visual yellowish color (for ten days). Then, treated by 1% sodium hydroxide (NaOH) for one hour in ambient temperature and oven-dried in 45°C for one hour. Such chemical treatment is conventionally used to enhance surface properties of natural fibers, their moistures sensibility and the matrix fiber interface. Some fibers measured characteristics are indicated in Table 2.

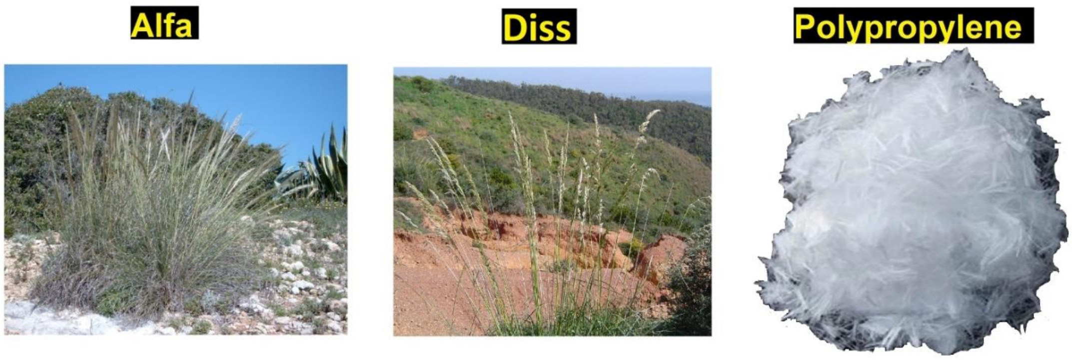

Figure 2. Raw fibers materials used in the study

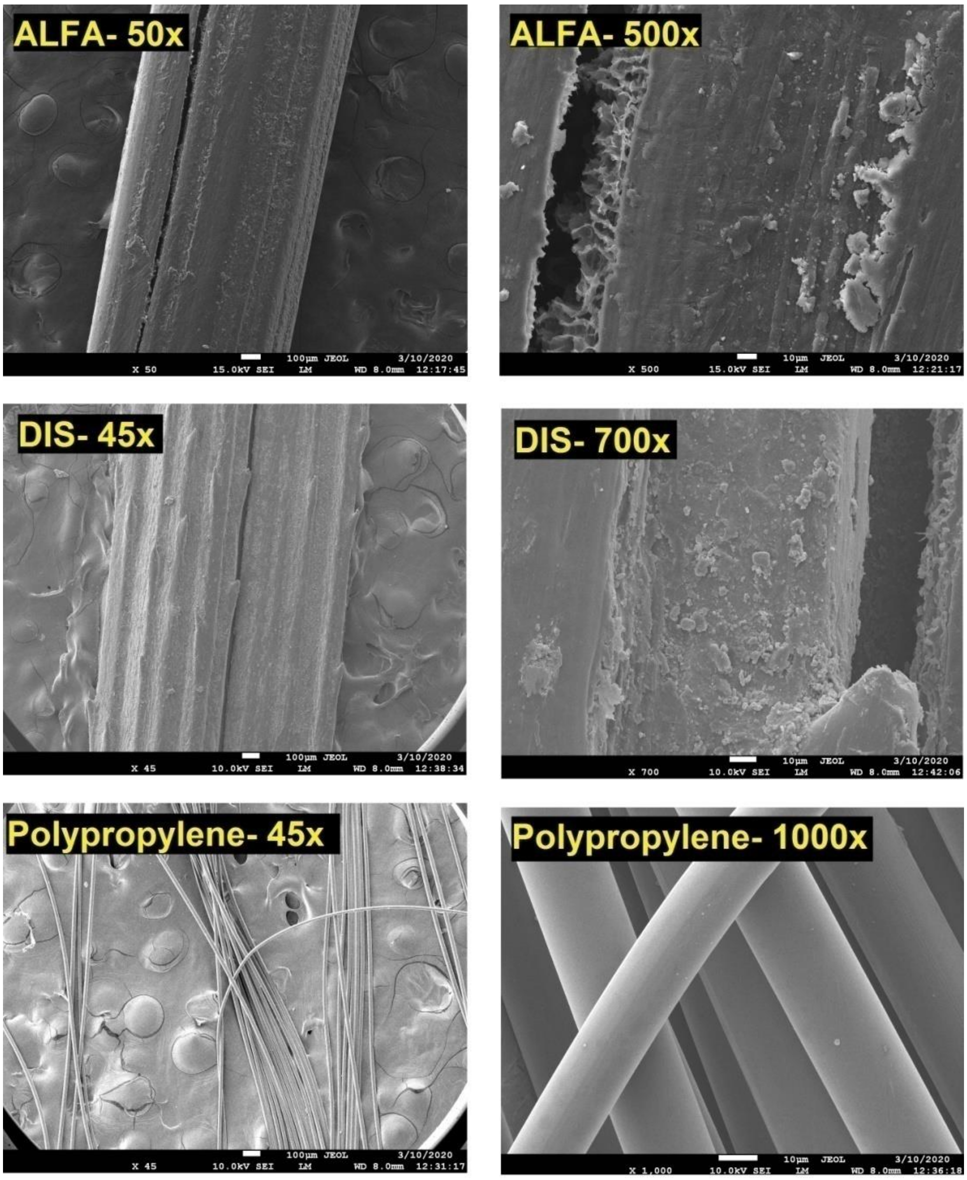

Figure 3. SEM observations of the prepared Fibers

Table 2. Physico-chemical and mechanical characteristics of VFs and polypropylene fibers (personal characterization (Abdelhak, 2017))

Characteristics/Fiber	Alfa	Diss	Polypropylene
density	1.4	1.23	0.9
Lignin (%)	25	27.5	-
Cellulose (%)	45	41.5	-
Hemicellulose (%)	24	22	-
Tensile strength (MPa)	250	174	450
Diameter (µm: Figure 3)	1000	1500	20
Used length (cm)	2	2	0.5

A metasilicate waterglass prepared solution was used to activate the solid precursor admixtures (slag and glass powder). It is a two-liquids solution containing sodium silicate Na_2SiO_3 and extra pure sodium hydroxide NaOH (50%wt solutions in water). Both obtained from (CEM-LAB NV, Belgium). Na_2SiO_3 composition (13% SiO_2, 11.16% Na_2O and 75.84% H_2O) by weight, a density of 1.39 g/ml and PH >10. In addition, NaOH has a density of 1.50 kg/l. The final activator solution with (SiO_2/Na_2O = 1) molar ratio was prepared 24h before each casting by mixing NaOH solution and sodium silicate with mass ratio (NaOH= 0.513 Na_2SiO_3).

2.2. Mixtures

While ordinary cement mixtures had a W/C ration equal to 0.5, the alkali-activated formulas had a liquid/solid (L/S) ration equal to 0.4. The pastes' mixture design for 100 grams of binder is presented in Table 3.

Table 3. 100 gr binder mixture design of study pastes

Paste	OPC (g)	Water (g)	Slag (g)	Glass powder (g)	Activator (g)
C	100	50	-	-	-
S	-	30	100	-	25.3
SG	-	30	80	20	25.3

For mortars mixes, MC had W/C=0.5 and Sand/cement=2.75, according to ASTMC1329 (2005). Besides, for AAMs liquid/solid =0.4 and Sand/binder=2.75 were kept. The mortars' composition is shown in Table 4.

Table 4. 600 gr binder mixture design of study mortars

Mortar	Sand (g)	OPC (g)	Water (g)	Slag (g)	Glass powder (g)	Activator (g)	Fiber volume rate (%)
MC	1650	600	300	-	-	-	-
MS	1650	-	180	600	-	151.8	-
MSG	1650	-	180	480	120	151.8	-
MSGA	1650	-	180	480	120	151.8	1
MSGD	1650	-	180	480	120	151.8	1
MSGP	1650	-	180	480	120	151.8	1

2.3. Procedures

After mixtures' design, the experimental investigation was done in ambient laboratory conditions.

For pastes, the following mixing procedure was applied:

- Mixing of dry materials with 75% of the used amount of water for 90s at slow speed;
- 25% water and alkaline solution for 90s at low speed.
- For mortars, the following mixing procedure was applied:
- Mixing of dry materials for 1min at slow speed;
- Fibers for 1 min at slow speed;
- 75%.wt of the used water for 1min at slow speed;
- 25% water and alkaline solution for 2min at low speed;
- 2min at high speed after a break of 1min.

Mortar flow test was conducted immediately after batching. To examine the workability of mortars, we used a mini-cone having a lower diameter d1=150 mm, an upper diameter d2 = 50 mm and a height h=100 mm. The fresh mortar is poured into the mold by two layers with applying 30 shocks for each layer. Hence, the obtained spread value is the average of two measured diameters.

The molds were covered by plastic films for 48 h at room temperature before demolding. Subsequently, they were placed in plastic bags and cured at 20 ± 2 °C in an air-conditioned room at relative humidity RH >55% until the specified testing ages.

The study tests are shown in Table 5.

Table 5. Summary of the study's testing procedures

Test conducted on mortar	Test conducted on paste
Flow Compressive strengths Flexural strength Open water porosity/density Thermal conductivity	FTIR SEM observations

Compressive strength of mortars was determined by 7,28 and 90 days on 4x4x4 cm^3 mortar samples according to EN 196-1standard (2006). For flexural strength, 4x4x16cm^3 samples were used.

Water porosity test was conducted on mortar bits according to NF EN18-459 standard (2010).

Fourier's transformed infrared spectroscopy (FTIR) technique was used to identify the involved chemical bonds and the molecular structure of the materials analyzed (Ex. C-H, C-OH, C-O, C-C). Thus, the links formed during geo-polymerization reaction should be assessed. The broken pieces of paste samples were prepared. They were placed directly on the device's supports and obtained graphs as peaks combinations. Characterization work is carried out then to better identify the found peaks.

The paste samples' morphology at microstructural scale was examined by scanning electron microscopy (SEM) on platinum-coated specimens' fragments using a Gemini 300 (ZEISS). The micrograph ware recorded at a working distance of 8-9 mm (Chiker *et al*., 2021; Cousture *et al*., 2021).

3. Results and discussions

3.1. Mortar flow

In Figure 4 the recorded flow values of mortar specimens are plotted.

The first obvious observed fact is the 40-50% lower flow of the activated mortars with or without fibers. This is explained by the rheological behavior of activated mixtures exhibiting higher viscosity compared to ordinary Portland cement materials (Stefanini *et al*., 2023). This is a subject of controversy for yet among the area workers. Some believe on the relevant negative influence of sodium metasilicate on workability (Li *et al*., 2018; Zhang *et al*., 2020).

Furthermore, by comparing MS and MSG flow values, it can be seen that glass powder led to increase mortar flowability. What is already reported for cement-based or alkali-activated mixes in previous works (Alioui *et al*., 2022; Tagnit-Hamou & Bengougam, 2012) and sought by the authors to adjust the loose of workability caused by the fibers in the set.

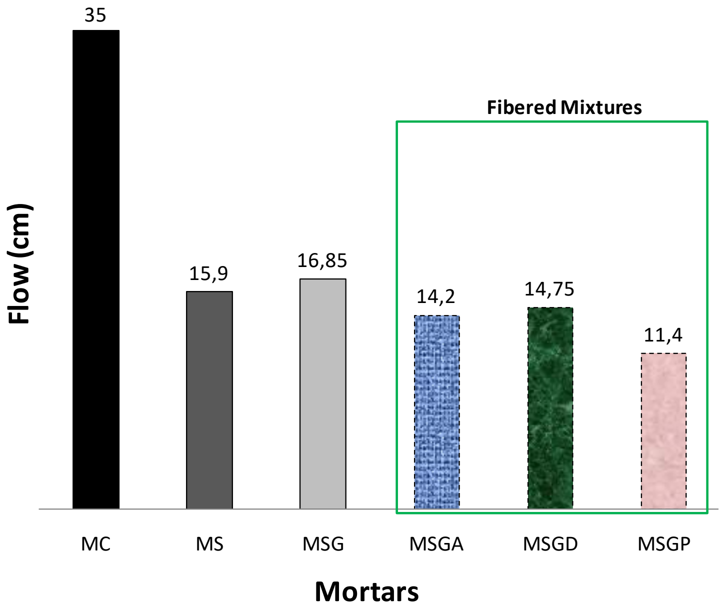

Figure 4. Mini-cone flow test records of mortar samples

Besides, the fibers presence in slag/glass samples caused a slight down of flow. This is an expected feature by considering their physical role as barrier to mortar flow, as reported previously in literature (Lv *et al.*, 2023; Terai, 2023).

By comparing VF and polypropylene samples flow values, it was seen that P induced lower flow. The authors explain that by the high fiber's dispersion in the case of polypropylene, which having very finer diameter compared to VF (see physical characteristics table). In other words, with a same volume amount in the mix, small diameter leads to higher fibers number dispersed in the total volume and hence to decreased flow.

3.2. Compressive and flexural strength of mortar samples

We present in this section the results of mechanical compressive and flexural strength tests at 7, 28 and 90 days (Figures 5 and 6).

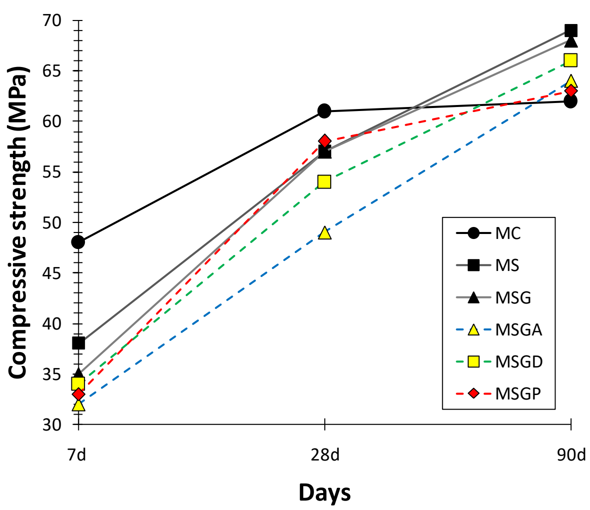

Figure 5. Compressive strength values of mortar samples

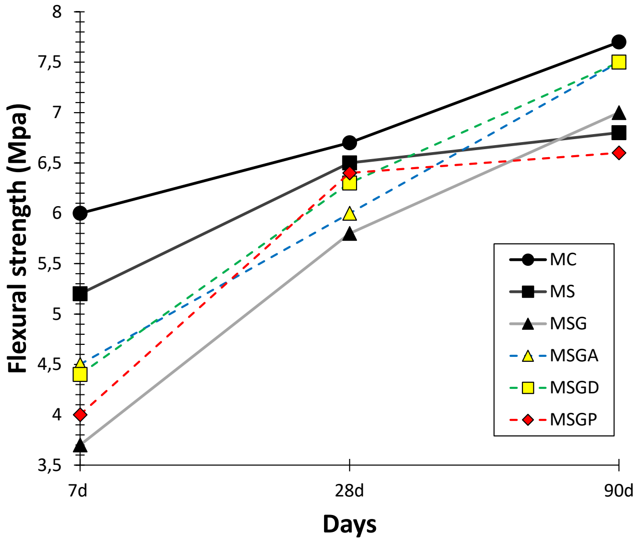

Figure 6. Flexural strength values of mortar samples

From Figure 5 we see that OPC mortar exhibited higher strength values than AABs at 28d. However, that was inversed at later hydration ages (90d). This fact is explained by the delayed recrystallisation process in high-CaO content precursor materials (GP and slag) (Leonelli & Palomo, 2022).

Furthermore, at 90 days compressive strengths of fibred mixtures was slightly lower than fiberless AABs, but still having superior values than OPC mortar. This confirms that fibers content in the present study have little influence on compressive strength, may be due to their small substitution rate (1% of the total mixtures' volume). Some similar results have been already published by Chindrasiriphan et al. (2023) on polypropylene fibers and Wongsa et al. (2020) with 1% coir fibers.

Besides, we can see that VF mortars recorded better compressive values than PP based mixture at 90 days. May be due to vegetable fibers' length (2cm) compared to PP fibers. Also, the authors believe that vegetable fibers rough surface can play a key role in strengthen interfacial zone (ITZ) between paste and fibers, as explained previously by Mohanty et al. (2005). This fact is more confirmed by observing the Alfa fiber cross section with longitudinal splits allowing strong matrix/fiber adhesion (Figure 3 and SEM monographs of fibers in Figure 7). The significant factor here is the surface morphology of the fiber and thus the bonding nature (Jawaid *et al*., 2018). In the case of plant fibers, the mechanism is governed by both mechanical interlocking and chemical bonding boosted by the good compatibility of the NaOH-treated fiber surface and the matrix.

Furthermore, it can be seen that Diss perform better than Alfa. What can be explained by the surface nature and the influence of cellulose content (de Azevedo *et al*., 2021; Santana *et al*., 2021).

Regarding flexural strength records in Figure 6, the first observed fact is the lower values in all hydration ages of AABs as compared to OPC mix. Thus, in the case of plant fibers alkali-activated mixtures flexural strength don't follow compressive strength. Even with good compressive behavior, weak flexural values may be explained by the influence of hardened paste with high interconnected pore set, especially near the fibers interface, fostering fracture under flexural loads.

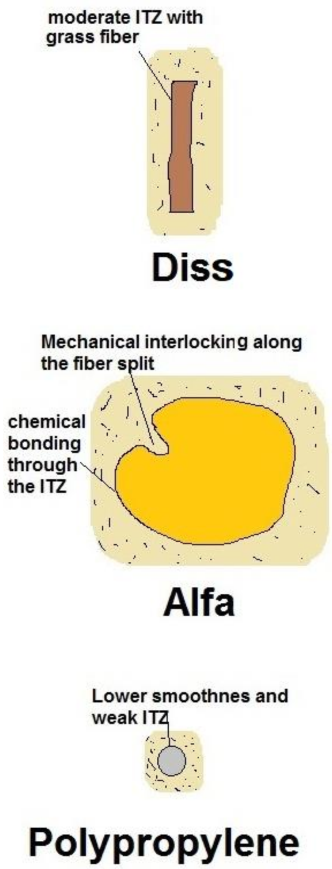

Figure 7. The proposed sets of fibers bonding mechanism

Nevertheless, vegetable fibers with lower fiber tensile strength (174-250 MPa) exhibited better mortar flexural records than PP mortar, having higher tensile (450 MPa). This may be the result of the conducted efficient fibers' NaOH treatment in one hand and the smooth PP fibers on the other hand (Ranjbar *et al*., 2016). In short, for flexural strength, the authors state that the fiber surface smoothness is more significant than the initial flexural resistance of the fiber.

3.3. Water porosity and density of mortar samples

Figures 8 and 9 show respectively the obtained results of water porosity and density of hardened mortar samples.

AABs showed higher water porosity and lower density than control mix. This means that activated mixtures have a special set of water porosity and bulk density different from OPC based mixtures. In other words, the hydration products and paste pores' set in activated binders are quite different from cement (Alioui *et al*., 2022).

Some previous works state higher induced porosity with VF (Belkadi *et al*., 2018; Khan *et al*., 2018). In our case, fibred mixtures exhibit identical or lower porosity values than free-fibers mortar (MSG) and almost similar densities. This confirms again that the 1% substitution fibers rate have little influence on mortars' porosity and density. Also, those finding confirm also that the good interface fiber/matrix in our mixtures with no less generated porosity around fibers.

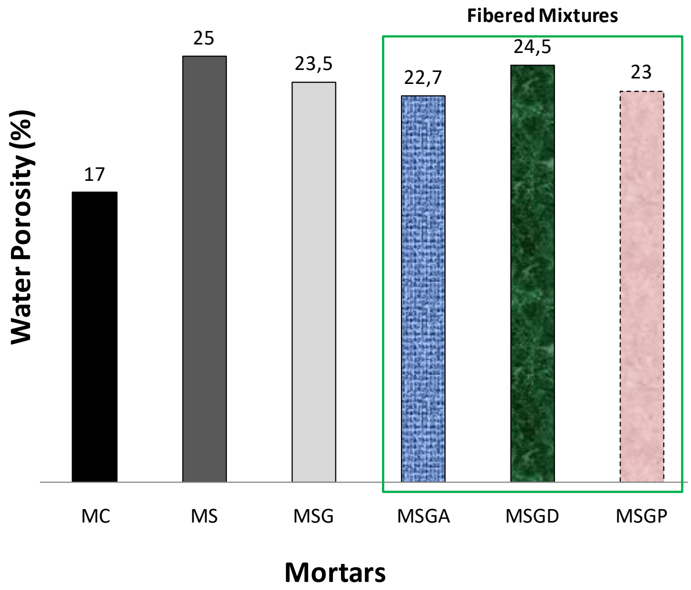

Figure 8. Open water porosityvalues of mortar samples

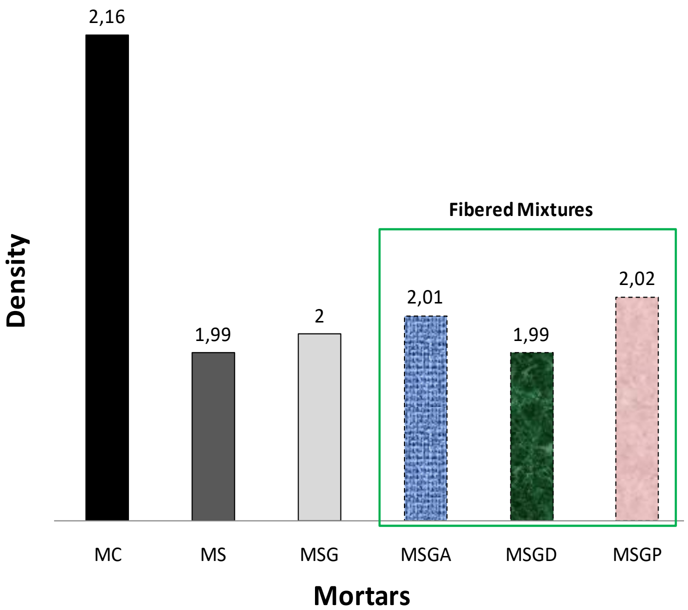

Figure 9. Density values of mortar samples

To get link with flexural strength authors suggest that the near ITZ composition is different than the rest of the paste, as found in Portland cement interface (Mehta & Monteiro, 2014; Ollivier *et al*., 2012). Thus, this ITZ may be formed by a thin layered brittle material influence flexural loadings than density and porosity network (Figure 10), more investigations are needed to support this fact.

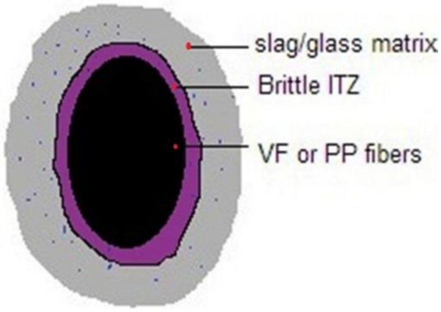

Figure 10. Proposed scheme of bonding interface and pastes porosity in AAM fibred mixes

3.4. Thermal conductivity

In Figure 11, the thermal conductivity recorded values of the studied mortars are presented. The plot was designed to figure out the influence of GP and fibers substitution rate.

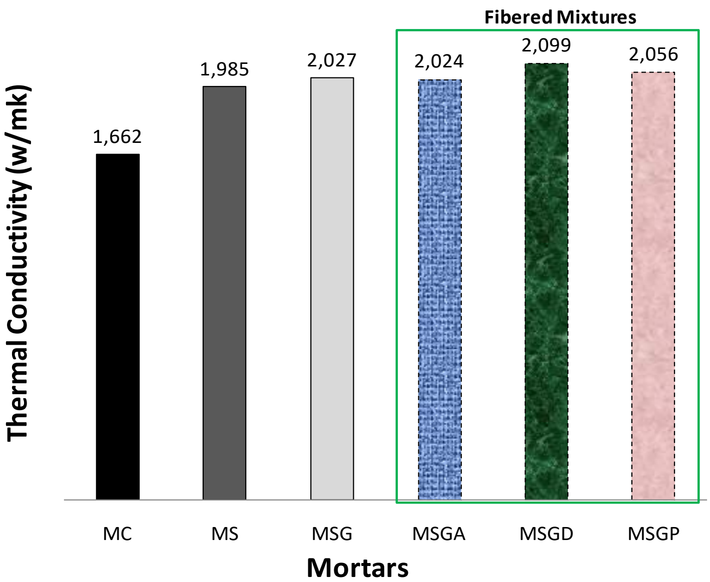

Figure 11. Thermal conductivity of mortars

Alkali-activated formula had higher thermal conductivity records. This may be explained by the set of porosity network of AABs, so different from OPC-based materials. Some previous works on cementitious materials report decreased thermal conductivity with vegetable fibers (Belkadi *et al*., 2018). They explained this by the reduced connected porosity by the physical deposit of the fibers within the matrix. In this study, with 1% fibers content, neither the presence of GP nor the fibers appear to have a significant role in connected porosity and thermal conductivity.

3.5. FTIR test results

Figure 12 shows the plots of the obtained results. The main task here is to identify the nature of hydration products, an explain the obtained results. The authors used the literature background of FTIR of alkali-activated materials as basic tool (Tables 6 and 7).

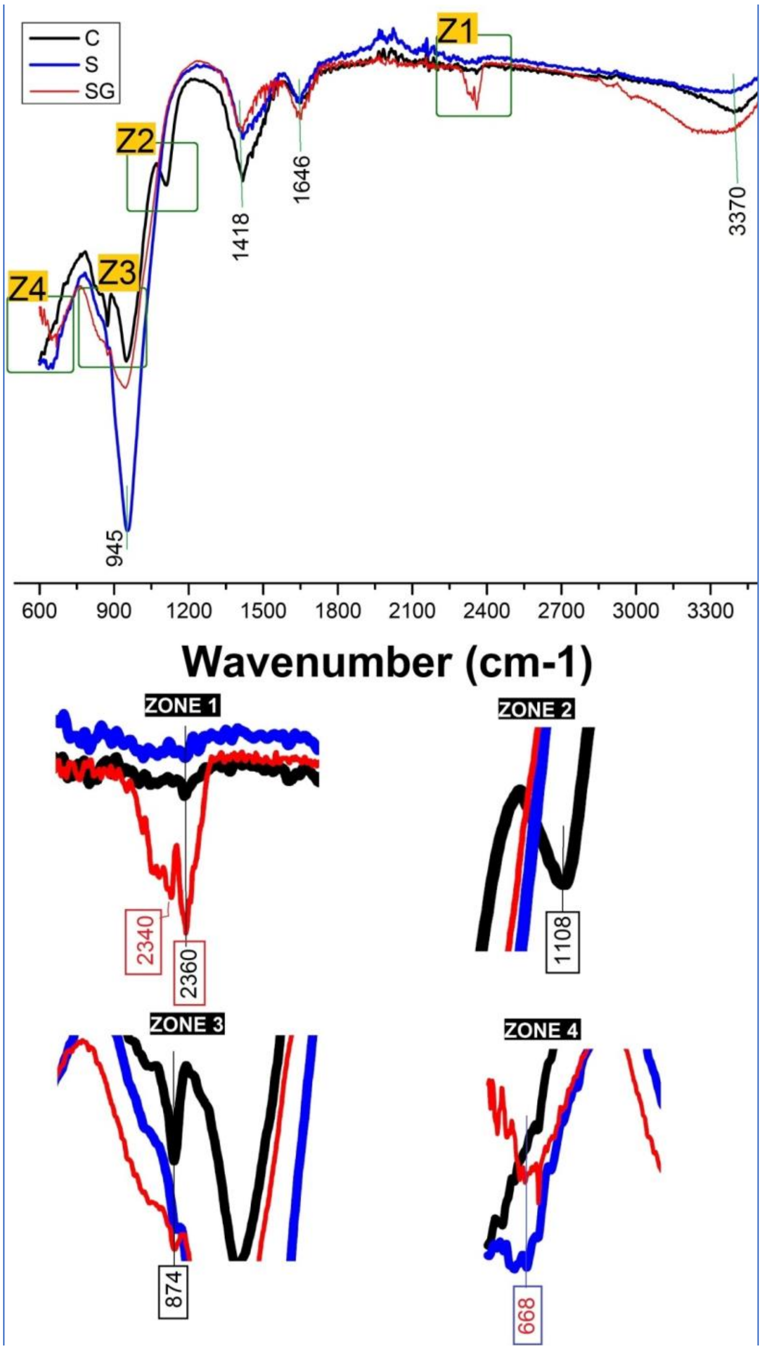

Figure 12. FTIR plots

Table 6. Compilation of FTIR's literature entries of hydration'bonds in the case of OPC mixtures

Compound	Bond/peak (wavenumber)	Reference
Anhydrous clinker	925,878,523	(Farcas & Touzé, 2001)
Portlandite	3640, 3415, 1638,1477	
(Si-O bond) C-S-H	970	
Gypsum (S-O)	1158-1121	
	670-603	
Calcite	1428, 878, 721	
Water band	3438	
	3440	(Shi *et al.*, 2019)

Table 7. FTIR entries of hydration' bonds in the case of AABs

Compound	Bond/peak (wavenumber)	Reference
OH or H-OH vibration	1643,3444,3448,1643,2000	(Abed *et al*., 2023; Zhao & Li, 2022)
C-O tensile vibration, (carbonates)	1410-1570, 870,1406,1436,1430	(Liu *et al*., 2019; Rios *et al*., 2023)
Si-O-(Al/Si) asymmetric stretching vibration	983, 900-1000	(Liu *et al*., 2020; Zhu *et al*., 2019)
Si-O	950, 940, 683-449, 668,673	(Hosseini *et al*., 2021; Tho-In *et al*., 2018)

We can see four mutual peaks among the paste's samples, OPC-based and activated (874,945,1418 and 1646). According to the references cited in Tables 6 and 7, 1646 and 3370 cm-1 represent the bending vibration of OH bonds, explained by Chen et al. (2022) by the presence of free and chemically bound water. In addition, 1418 cm-1 peak is attributed to C-O bond of carbonates, as a result of $Na_2Ca(CO_3)_2.5H_2O$ formation (Liu *et al*., 2019). However, the peak at 945 in the case of activated binders is due to the asymmetric stretching vibration of Si-O-(Si/Al) bond (Li *et al*., 2024; Liu *et al*., 2020). It indicates the geopolymerization degree of the gel phase (Zhu *et al*., 2019) and correspond to the CSH hydrate formation in cementitious control sample. Also, Tho-In et al. (2018) report the formation of more stable gel with 20% glass powder, same content with our study. The peak in 686 cm-1 wavenumbers for activated samples match the symmetrical tensile vibration of Al-O bond (Sedira & Castro-Gomes, 2020) or Si-O bond (Pan *et al*., 2018).

3.6. SEM observations

Figure 13 present the recorded morphological SEM observations conducted on paste samples. Remember that through this task the authors aim to assess the microstructure of the hydrated paste to explain the macrostructural observed facts.

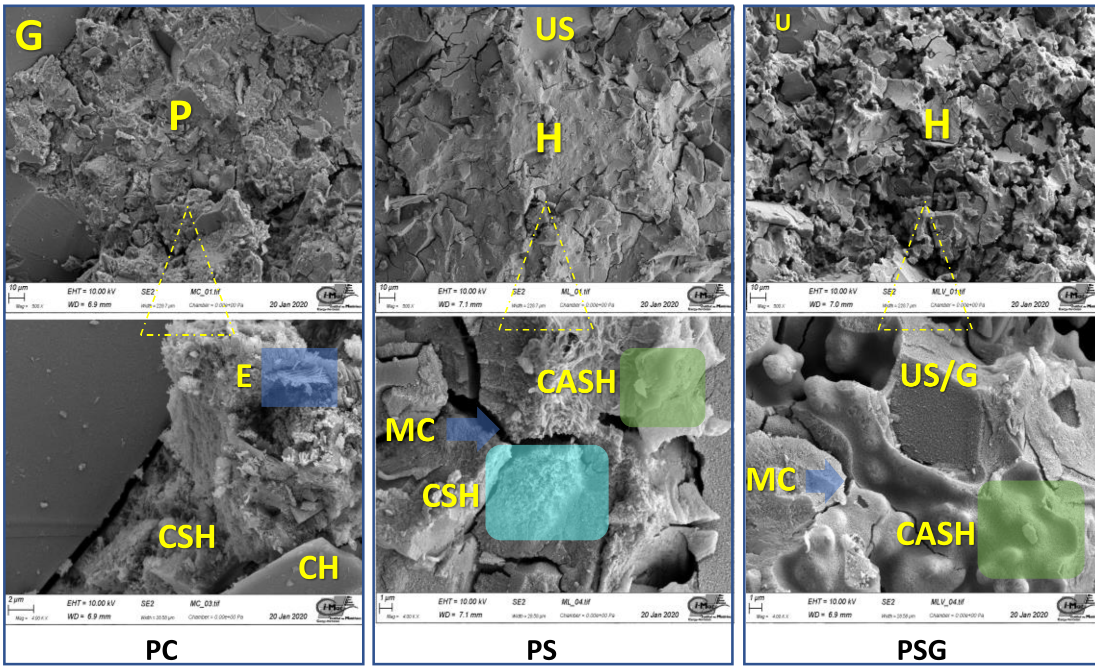

Figure 13. SEM morphology of PC, PS and PSG pastes

First, it can be seen that the hydrated paste in activated samples exhibits more microcracking. There is a general agreement that alkali-activated binders are prone microcracking due to autogenous shrinkage (Provis, 2014). Nevertheless, this is not for now proved to be a serious threat to the mechanical properties, see the recorded values here. The formation of amorphous CASH silica gel rich products was observed more in PSG sample. Thus, the presence of glass powder enhances the sample microstructure, limits non-reacted precursor particles and visible cracks in comparison with PG free sample. This reveals SiO_2 boost additional aluminosilicate gels production. Thereby geopolymerization degree is upgraded process (Sun *et al*., 2024) and mechanical properties are increased. Good agreement with previous studies (Ali *et al*., 2023).

SEM and FTIR findings the option of binary precursor to synthetize alkali-activated mortars was as good. This matrix is compatible with NaOH treated fibers with dense ITZ.

4. Concluding remarks and recommendations

This experimental investigation studied the performance of biobased fibered vegetable mortars, Diss and Alfa, compared to polypropylene fibers.

The following conclusions are observed:

- Compared to OPC mortar compressive strength was enhanced with plant fibers with a little decrease in flexural strength, flexural strength doesn't fellow compressive strength.
- NaOH treatment is an efficient process to boost bonding between the matrix and the fibers.
- We suggest that mechanical interlocking and chemical bonding are both involved in vegetable fibers/AAMs matrix ITZ.
- The roughness and morphology of the fibers is more reliable than the fibers physical properties, especially with a lower fiber content. Those factors dictate the ITZ behavior controlling all the material properties.
- Diss formulations mechanical strength is better than Alfa formulations due to its higher cellulose content.
- A 1 % fibers content has a little influence on thermal conductivity of AAMs.

The findings of this study provide some insights to future researchers and construction industries aiming to produce green alkali-activated materials with grass fibers.

Finally, we suggest advanced ITZ investigations, alternative treatment methods of fibers and optimizing fiber contents as a perspective to better understand the involved mechanics of AAM with alkali-activated natural fibers.

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