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Fiber-reinforced one-part alkali-activated slag/ceramic binders

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Abstract

In the present experimental/numerical study, a combination of ceramic waste and ground-granulated blast furnace slag were used in the preparation of one-part alkali-activated binders. Moreover, the effect of fiber type and content on hardened-state properties and shrinkage was studied under two different curing conditions. In the first stage of this study water absorption, compressive strength, and flexural strength were assessed. Subsequently, the flexural performance of fiber-reinforced binders was simulated and predicted using finite element models under concentrated and distributed flexural loading, respectively. The experimental results showed that fibers improved mechanical properties, and enhancement was governed by fiber type and curing conditions. Moreover, the numerical results indicated that the developed fiber-reinforced binders offer a flexural load-carrying capacity in the range of 10 to 40 kN/m² and permissible service loads were well below the ultimate capacity.

Keywords: One-part alkali-activated binders, Fiber-reinforced binders, Ground-granulated blast furnace slag, Ceramic waste, Hardened-state properties, Numerical simulations

1. Introduction

Alkali-activated binders have been investigated as one alternative to ordinary Portland cement (OPC). Alkali activation is based on the dissolution of reactive aluminosilicates in alkaline conditions. The most commonly used raw materials include kaolinitic clays [1-3], fly ash [5-8], blast furnace slag [9, 10], and combinations of these materials as hybrid blends [11-14].

Through alkali activation, many large-scale waste streams can be converted into sustainable construction materials [15]. The sustainable concrete industry is a research area that has been investigated broadly, with researchers specifically focusing on how to convert industrial by-products into cement and concrete [6-9].

The alkali-activation process in conventional alkali-activated binders (or two-part alkali-activated binders) involves alkali solutions, which are corrosive, viscous, difficult to cast and not user-friendly [16]. Therefore, one-part or “just add water” alkali-activated binders, in which the dry ingredients of these mix compositions comprise a solid aluminosilicate precursor and a solid alkali source [16-18], have increasingly been studied as an alternative approach. Finally, similar to the preparation of OPC, water is added to the dry composition. These binders eliminate the disadvantages of traditional alkali-activated binders and better enable cast-in-situ applications, making them appropriate for the construction market.

In general, the reaction mechanisms of alkali-activated materials are different when compared to ordinary Portland cement (OPC) [19]. Binders with a high calcium content could be activated with moderate alkalinity and calcium aluminum silicate hydrate (C-A-S-H) gel forms as the main reaction product, which would lead to a dense matrix with acceptable mechanical properties. Ground-granulated blast furnace slag (GGBFS) is commonly used as a calcium-rich aluminosilicate precursor in alkali-activated materials. GGBFS, which is a by-product of pig iron manufacturing in blast furnaces, consists of silicate and aluminate impurities present in iron ore and coke. Alkali-activated GGBFS binders typically have a high compressive strength at an early age, great durability performance (especially against acid and sulfate attack) [20], rapid setting and hardening [20], low hydration heat [21], high temperature resistance [21], and lower CO₂ emissions [22-25] compared to OPC.

Due to the benefits of one-part alkali-activated binders and ground GGBFS as a calcium-rich aluminosilicate precursor, various efforts have been implemented to develop one-part alkali-activated slag binders [26]. However, alkali-activated slag binders have some drawbacks, including volumetric instability and high shrinkage compared to OPC [27]. A high rate of shrinkage leads to the formation of cracks, which could subsequently affect mechanical and durability properties as well as long-term performance [23].

Several studies have demonstrated that the combination of GGBFS as a high-calcium-level precursor with another low-calcium-level precursor reduces shrinkage compared to alkali-activated binders containing solely GGBFS [28-39].

Lately, ceramic waste has been proposed as a silica source in the preparation of alkali-activated binders [40]. Besides the content of active calcium in the mix composition, which plays an

important role in gel formation and strengthening, the content of reactive silica and alumina are important in the formation of the gel structure [41]. Thus, the use of ceramic waste as the silica source in the preparation of alkali-activated binders could have advantages, including the conservation of natural resources, energy, and lower cost, as well as reductions in CO₂ emissions and other greenhouse gases. Accordingly, the use of ceramic waste as a partial replacement for cementitious binders has been the subject of several studies [42-47]. In most published studies on the use of ceramic waste as a replacement for cementitious binders [42-47], it has been proposed that ceramic waste could comprise 10–35% as a replacement.

One of the effective and relatively simple ways to reduce shrinkage compared to other methods is to use fibers to reinforce the mix compositions. That said, the amount of reduction in shrinkage depends on the type, content, and mechanical properties of the fibers as well as their bond properties at the interface of the fiber and the matrix. The addition of fibers may not only reduce shrinkage in regard to the abovementioned parameters, but could also improve the mechanical properties of the reinforced mix compositions [69].

Some studies have examined the effects of using ceramic waste as an aggregate or as an pozzolanic admixture in OPC-based and two-part alkali-activated binders (conventional alkali-activated binders) [42-47]. However, an experimental/numerical study on fiber-reinforced one-part alkali-activated slag and ceramic binders has not yet been performed. Therefore, this paper shows the feasibility of using fiber-reinforced one-part alkali-activated blast furnace slag and ceramic binders. Moreover, the effects of using different curing regimes (sealing and water bath) on the physical and mechanical characteristics of fiber-reinforced mix compositions were investigated. Lastly, the drying shrinkage was studied in terms of crack formation on the surfaces of specimens.

2. Experimental program

2.1. Materials and mix designs

The designed alkali-activated mortars are composed of GGBFS (Finnsementti, Finland), ceramic waste (IDO, Finland), fine aggregate (standard sand according to EN 196-1), anhydrous sodium metasilicate (Na₂SiO₃, Alfa Aesar, molar SiO₂/Na₂O ≈ 1), and water. The chemical composition of the blast furnace slag and porcelain ceramic waste, which were determined by X-ray fluorescence (XRF, PANalytical AxiosmAX), are presented in Table 1. Regarding the chemical compositions, the slag was rich in calcium (CaO: 38 wt.%) and ceramic waste was a source of silica (SiO₂: 67 wt.%). A size distribution was obtained for sand grains

with respect to the ASTM D6913 recommendation [48], with minimum and maximum grain sizes of 0.08 and 2 mm, respectively. The used slag had a d_{50} value and density of 10.8 μm and 2.93 g/cm^3 , respectively. The porcelain ceramic wastes were milled and sieved to obtain a grain size less than 75 μm [75].

In this study, three different fibers (steel fiber (ST), polypropylene fiber (PP), and polyvinyl alcohol (PVA) fiber) with 0.5 and 1% of the total binder mass were used to reinforce the compositions. The physical and mechanical properties of industrial steel, PP, and PVA fibers are listed in Table 2.

The mix ingredients were combined with slag in 80% of the total binder mass, ceramic waste in 10% of the total binder mass, and anhydrous sodium silicate in 10% of the total binder mass. The sand-to-binder ratio was kept constant at 2. Finally, water was added to the mixture using a water-to-binder ratio of 0.35. Table 3 lists the molar ratios related to the designed one-part alkali-activated binder. It is worth stating that the preliminary experimental tests showed that replacing more than 10% of the slag with ceramic waste significantly affected fresh state properties, followed by mechanical properties. Thus, the maximum slag that could be used was replaced by ceramic waste.

Table 1. The chemical composition of slag and ceramic waste

Table 2. The mechanical properties of industrial steel, PP, and PVA fibers

Table 3. Molar ratios of the designed mix composition

2.2. Specimen synthesis and aging

Dry ingredients (slag, ceramic waste, sodium silicate, and sand) were initially mixed for 3 minutes in a mixer. Afterward, water was added to the mixture, and the mix composition was then stirred for a further 2 minutes. Finally, the mixtures were prepared by gradual addition of fibers to the fresh mix compositions to avoid balling of fibers, while adding enough fibers to the admixture so that the desired ratio of fibers was achieved. Then, the compositions were mixed for a further 2 minutes. To avoid of inhomogeneous distribution of fibers, a low mixing speed (mixing paddle at a rate of 140 ± 5 rpm and planetary motion of 65 rpm) was used. This approach was imposed for preparing all reinforced mix compositions with different fiber types. Fresh mortar was cast into cubic molds (40 × 40 × 40 mm) and prismatic beams (40 × 40 × 160 mm) to assess its physical and mechanical properties. These characteristics were addressed in terms of water absorption, compressive strength, and flexural strength.

After casting, the specimens were kept at a temperature of 23 °C and a relative humidity of 35% for 24 hours; the specimens were then demolded and cured in two different regimes for 28 days

(until the test day): (1) a water bath with a temperature of 20 °C, and (2) sealed in plastic bags. The control specimens (the plain composition) were also kept sealed until the test day.

Regarding the use of the three different fibers (PP, PVA, steel) with two different percentages (0.5 and 1% of the total binder mass), a total of 21 prismatic beams (considering the plain alkali-activated binder as the reference mix composition) were cast and tested to assess their flexural and compressive strength. For each mix composition, three prismatic beams were cast, prepared, and tested. The flexural responses of the prismatic beams made with plain or reinforced one-part alkali-activated slag and ceramic binders were obtained from the average of three replicated prismatic beams.

In the present study, the specimens were nominated as follows: The first letters indicate the fiber type (steel fiber (ST), polypropylene fiber (PP), and polyvinyl alcohol (PVA) fiber); the following number shows the fiber mass (0.5 or 1% of the total binder mass); and the last letter depicts the curing regime (if a water bath was used to cure the specimen, it has the letter “W”; no letter indicates sealed curing).

2.3. Test procedures and setups

2.3.1. Water absorption by immersion

In total, 21 cubic specimens with 40-mm edges were cast and cured under the aforementioned conditions for 28 days. The cubic specimens were immersed in a water bath (the average water temperature was 23 °C) for 24 hours at room temperature conditions. Then, the mass of each specimen was measured as W_{Im} (immersed weight) as it was suspended with a thin wire and completely submerged in water. Afterward, the specimens were removed from the water and placed on a wire mesh for 1 minute to allow the water to drain. Next, using a damp cloth, the visible surface water was removed and the mass was recorded as W_{Sat} (saturated weight). All specimens were heated by using an oven at 105 ± 2 °C for no less than 24 hours. The masses of the specimens were measured every 2 hours. The increment of the mass loss should not be greater than 0.1% of the last mass measured for the specimen. The mass of the dried specimens was obtained as W_{Dry} (oven-dry weight). Water absorption by immersion was obtained by the following equation:

$$Abs(\%) = \frac{W_{Sat} - W_{Dry}}{W_{Dry}} \times 100 \quad (1)$$

2.3.2. Three-point bending test (TPB)

Twenty-one prismatic beams (40 × 40 × 160 mm) were used to assess the flexural performance of reinforced and plain beams made with one-part alkali-activated slag and ceramic binders under a three-point bending (TPB) test in accordance with the ASTM C78 recommendation [49]. The flexural load was recorded by using a load cell with a capacity of 100 kN. The prismatic beams were evaluated under a flexural load with a deflection rate of 0.6 mm/min. The flexural load was submitted to the beams under a displacement control. Equation (2) was used to calculate the flexural strength of the specimens under the TPB test:

$$\sigma_f = \frac{3FL}{2bh^2} \quad (2)$$

where F is the total flexural load, L is the span length (equal to 120 mm), and b and h are the width (40 mm) and height (40 mm) of the beams, respectively.

2.3.3. Compressive test

As described above, two different regimes were used to cure the specimens. According to the ASTM C116-90 recommendation, the compressive strength of the specimens was measured using portions of prismatic beams broken in the flexural test [50]. All the broken prismatic beams were assessed under a compressive load with a constant displacement rate of 1.80 mm/min. The compressive load was measured with a Zwick Z100 Roell testing machine with a maximum load capacity of 100 kN. The results were obtained from the average of six tested replicates.

2.3.4. Microstructural analysis

After assessing the compressive strength of the mixtures, their morphology was observed by scanning electron microscope (SEM) images. To take the SEM images, small samples of the mixtures were selected and submerged in acetone (ACS grade) to stop the reaction products. In this study, the SEM images were produced by using an acceleration voltage of 15 kV and a distance varied in the range of 6–8 mm.

3. Results and discussion

3.1. Water absorption

Fig. 1 depicts the water absorption of different mixtures reinforced with different fiber types and contents. Water absorption in the fiber-reinforced mixtures depended on the porosity and thickness of the transition zone [51, 52]. This test was implemented to assess the resistance of concrete to the ingress of aggressive ions and, indirectly, the porosity network. The addition of

fibers increases both the porosity and thickness of the transition zone, which could increase and decrease water absorption, respectively. Therefore, interactions between these two effects can either increase or decrease water absorption [51, 53]. Increasing the porosity and thickness of the transition zone depends on the fiber content, type, and length. However, a higher increase in the thickness of the transition zone was obtained, as compared to an increase in porosity, and a reduction in water absorption could be measured [51].

The results in Fig. 1 show that compared to the plain mixture, adding fibers to the mixtures can either increase or decrease water absorption, depending on the curing regime. This can result from the introduction of more air voids due to the addition of fibers and an increase in thickness at the transition zone. The curing regime affected the porosity at the fiber/matrix interface.

Curing conditions significantly affect the mechanism of binders where C-A-S-H gel in the main strength-giving phase. The relative humidity can affect the structural incorporation of alkali cations in the C-A-S-H gel, which reduces the stacking regularity of C-A-S-H layers. Then, the drying facilitates the C-A-S-H redistribution and reorganization, silicate polymerization, new interlayer formation and alteration of the pore refinement [81]. Through this mechanism, relative humidity affects the alkali-activated binder systems incorporating C-A-S-H gel.

As indicated in Fig. 1, water absorption in the reference plain mixture was recorded 6.85%. The plain mixture was cured by sealing in plastic bags. The maximum increase in water absorption was recorded as about 5% in the composition reinforced with 1% PVA fiber and sealed in plastic bags until the test day, as compared to the plain composition. Meanwhile, the maximum reduction was recorded as more than 6% for the mixture reinforced with 0.5% PVA fiber and cured in a water bath.

With respect to the results, as shown in Fig. 1, using different curing regimes had a greater impact on the porosity network of the compositions as compared to the use of different fiber types and contents. This means that an increase in the thickness of the transition zone due to the formation of reaction products and a reduction in porosity had a higher impact in terms of reducing water absorption in comparison to the introduction of air voids and to increases the porosity causes to add the fibers. In general, the incorporation of fibers and the use of different curing regimes did not have a substantial effect on increasing or decreasing water absorption. It will be shown (in sections 3.2 and 3.3) that the mechanical performance of the reinforced mixtures will be significantly affected by the fiber type and curing regimes through affecting the chemical bond and friction. In general, fibers begin to arrest further crack opening after the initial crack has been formed. With respect to the curing regimes and their effect on the bond properties at fiber/matrix (chemical bond and frictional behavior), the mechanical performances

of the reinforced mixtures will be shown to have significant differences. On the contrary, the effect that fiber addition has on water absorption is less clear; depending on whether increasing air voids or ITZ thickness dominates, it can be either increased or decreased. Therefore, variables such as fiber length and content can have a defining impact on hardened-state properties.

Fig. 1. Effect of curing conditions on water absorption of the reinforced compositions containing different fiber contents and types.

3.2. Compressive strength

The effects of using different fiber types, contents, and curing regimes on the compressive strength of the compositions are illustrated in Fig. 2. The addition of fibers increases air voids while simultaneously limiting the propagation of cracks. Therefore, the interaction of these two effects could either increase or decrease compressive strength [54, 55].

According to the results, regardless of fiber type and content, fibers can effectively increase compressive strength. In other words, the capacity of the fibers to prevent the further opening of cracks was greater than the induced porosity they caused. The compressive strength for the plain mixture was recorded around 42 MPa. According to Fig. 2, the maximum increase of compressive strength was more than 50% in the compositions incorporating 1% PVA or steel fibers and cured in the water bath, as compared to the plain composition. Moreover, the maximum compressive strength was obtained with compositions reinforced with 1% PVA or steel fibers and cured in the water bath. Furthermore, the lowest increment of compressive strength was measured in the reinforced compositions using PP fibers. This finding could be explained by the smooth surface of PP fibers, which provides a weak bond at fiber/matrix interfaces [56, 57]. In general, employing a water bath had a greater impact on increasing compressive strength when compared to sealing, such that the maximum increment of compressive strength in the case of using a water bath instead of sealing was more than 13% for the reinforced composition with 1% PVA fiber. This improvement resulted from increasing the degree of polymerization and, subsequently, forming more reaction products and decreasing the porosity. Additionally, the maximum increment of compressive strength was measured at around 40% in the reinforced mixtures with 1% PVA fiber and cured by sealing, as compared to the plain composition.

Fig. 2. Effect of curing conditions on the compressive strength of the reinforced compositions containing different fiber contents and types.

3.3. Flexural strength

The effect of fiber type and content have significant effect on the flexural performance of the reinforced composites. The pull-out behavior of smooth fibers consists of debonding and frictional pull-out behavior. Based on the debonding criterion in the strength-based models, debonding initiates when the interfacial shear stress exceeds the shear strength [58]. Ascending the load to the peak load is associated with the adhesive bond (or chemical bond) and the debonding process until full debonding occurs.

The adhesion or chemical bond represents the resistance offered by interface transition zone (ITZ) between the fiber and matrix that surrounds it. The chemical bond is the first mechanism to be activated on the pullout. During this initial phase, the fiber and matrix deformations are fully compatible, in such a way that the bond of the fiber/matrix interface does not suffer any damage.

The properties of ITZ depend on many parameters, such as the nature of the binder, the presence of additives, and water/binder ratio. The properties and bonding quality of the interface plays a crucial role on the behavior of the composite material. In fiber reinforced composites, the ITZ behavior has, even, a more relevant role on the mechanical behavior than in plain concretes, since the fiber aspect ratio is considerably higher, and, therefore, the contact surface area is larger than that of the aggregates [58]. The formation of ITZ is affected by the wall-effect close to the fiber surface, since there is a non-efficient packing of the binder particles in the fresh state, which leads to a high amount of empty space that can only be partially filled with reaction products. Generally, the ITZ is strengthened by densification and thus, the fiber/matrix interfacial bond strength is enhanced. After full debond is attained, frictional stresses are generated due to the abrasion and compaction processes on the ITZ throughout the slipping of the fiber along its channel, which is governed by wall roughness [58].

Using different fibers with different mechanical and physical properties affects the debonding and frictional pull-out behavior. For PVA fibers, the slip mainly depends on the breakdown of chemical adhesion and on friction [70], while it is generally accepted for steel fibers that the chemical bond can be neglected in favor of the mechanical bond between the fiber the surrounding matrix [71-73, 82, 83]. It is worth stating that the length of steel fibers has also great impact on the mechanical bond. Therefore, it can be expected that using short length steel fibers significantly improves the flexural strength, compared to the post-peak responses [74].

Outer surface of PP fiber is smooth, therefore, only weak interaction between fiber and matrix occurs during debonding, consisting of slip-dependent interface friction as well as snubbing

friction for fibers bridging at inclined angles [73, 84]. Moreover, the partial replacement of slag with ceramic wastes and curing regime affects the reaction products, which in turn influence the chemical adhesion and bond properties between fibers and matrix during the frictional pull-out stage.

The effect of fiber type and content on the flexural performance was investigated (Fig. 3). As indicated in Fig. 3a, increasing the PVA fiber content from 0.5 to 1% increased the flexural load by about 15% and improved the post-peak residual force, such that the post-peak residual force at a mid-span deflection of 0.3 mm ($\approx \delta: L/400$, where L is span length and equal to 120 mm) for the reinforced composition with 1% PVA fiber was almost twice that of the reinforced composition with 0.5% PVA. Moreover, no significant changes were observed for flexural stiffness due to an increase of the fiber content. The maximum load carrying capacity in the control beams was on average about 800 N. Fiber bridging action resulted in an increase of the load carrying capacity and deflection.

According to the results, using a water bath to cure the prismatic beams substantially changed the flexural performance of the fiber-reinforced beams. Regardless of the fiber content, a hardening deflection behavior was recorded for the specimens. For the reinforced beams with 0.5% PVA fibers, the curing regime had no significant impact on the crack form initiation force (≈ 2500 N). Above this force, a hardening deflection behavior was recorded and, subsequently, the force and ultimate deflection were increased to 3425 N and 0.87 mm ($\approx \delta: L/138$), respectively. The ductility (μ) of the mixtures was determined by the ultimate hardening deflection (above this deflection, the flexural behavior entered a softening phase (D_u in Fig. 3d)) to the deflection corresponding the crack initiation (D_y in Fig. 3d). Regarding this definition, a ductility higher than 3 was recorded. This significant enhancement may be correlated with the improvement in chemical and the frictional bond properties at the fiber/matrix interface. As mentioned earlier, increasing the degree of polymerization and decreasing the porosity effectively increased the thickness of the transition zone. Increasing the PVA fiber content from 0.5 to 1% in the beams cured in a water bath had no significant effect on the flexural stiffness, flexural strength, and hardening characteristics of the beams, while the residual force increased as compared to the beams incorporating 0.5% PVA fibers.

Sealing and reinforcing the prismatic beams with 0.5 or 1% micro steel fibers resulted in similar flexural performance, as shown in Fig. 3b. The only effect of increasing fiber content from 0.5 to 1% was a slight reduction in flexural stiffness. Using a water bath to cure the beams greatly affected the ultimate load carrying capacity and flexural stiffness when compared to sealing. The ultimate load carrying capacity and flexural stiffness increased more than 75% and 25%,

respectively, in the specimens cured in a water bath compared to sealing. An increase in the ultimate load carrying capacity and flexural stiffness could be explained by the short length of steel fibers. Employing longer steel fibers could significantly affect the hardening stage and post-peak responses of the beams. It could be concluded that the curing regimes affected the polymerization, which this influenced the chemical adhesion at the bond properties between short steel fibers and matrix.

The results in Fig. 3c demonstrate that the beams reinforced with PP fibers and sealed for 28 days presented an almost linear behavior up to a flexural force of 2000 N for PP0.5 and 1100 N for PP1, followed by an almost perfectly plastic behavior up to a mid-span deflection of about 4 mm. Like PVA fibers, changing the curing regime from sealing to water bath substantially changed the flexural performance of the fiber-reinforced beams. The curing regimes impacted the polymerization, which this influenced the chemical adhesion at the bond properties between PP fibers and matrix. While, sealing the reinforced prismatic beams with PP fibers affected the slip-dependent interface friction.

Fig. 3. Effect of curing conditions on the flexural performance of the reinforced compositions: a) containing PVA fiber; b) containing steel fiber; c) containing PP fiber; d) in terms of ductility.

Fig. 4 represents the flexural strength and deflection corresponding to the maximum flexural force of the compositions. The flexural strength and mid-span deflection corresponding to the flexural strength for the plain composition were measured 2.2 MPa and 0.1 mm, respectively. With respect to the results, the maximum flexural strength (7.2 MPa for PVA0.5 and 8.23 MPa for PVA1) and mid-span deflection were recorded in the beams reinforced with PVA and PP fibers, respectively, when the specimens were sealed. As expected, the minimum flexural strength was recorded for the beams reinforced with PP fibers as a result of their smooth surface and weak bond at the fiber/matrix interface [56]. It should be mentioned that the PP fibers indicated large post-peak deformation ($\approx \delta: L/30$) and the results presented in Fig. 4b were based on the deflection corresponding to the maximum flexural load. Since using a water bath greatly affected the flexural performance of the specimens. The maximum improvement in flexural strength and deflection was observed for the reinforced mixture with PP fibers (about 3 times in PP0.5W) and PVA fibers (more than 4 times in PVA0.5W), respectively. The minimum and maximum flexural strengths were recorded as 3.31 and 11.40 MPa for the specimens PP0.5 and ST1W, respectively.

Fig. 4. a) Flexural strength; b) mid-span deflection corresponding to the flexural strength.

SEM images were used to show the morphology of the compositions and better understand the fiber reinforcement mechanisms provided by different fibers. As shown in Fig. 5a, the hydrated

particles were attached to the fiber surfaces, which confirmed the excellent bond properties of PVA fibers to the matrix. Moreover, some scratches were observed on the PVA fiber surfaces, demonstrating high interactions between the pulled-out fibers from their surrounding matrix. These scratches explain the hardening deflection behavior of these materials under flexural loading.

As indicated in Fig. 5b, using a water bath led to some hydrated particles becoming attached to the PP fiber surfaces, which indicates improvements at the fiber/matrix interface. PP fibers were preponderantly failed by fiber debonding from their surrounded matrix in the reinforced specimens with PP fibers and cured using sealing (Fig. 5c), justifying an almost perfectly plastic behavior of the flexural capacity for the mid-span deflection in the interval of 0.3 to 4 mm.

Fig. 5d shows the fracture surface of the reinforced beams with short-length steel fibers that preponderantly failed by debonding. Using straight steel fibers with a short length explains the brittle behavior of the specimen reinforced with this type of fiber. Furthermore, not using hooked-end steel fibers did not provide an additional mechanical anchorage during the debonding.

Fig. 5. SEM images from: a) PVA0.5W; b) PP1W; c) PP0.5; d) ST1W.

It is worth noting that all specimens were visually monitored to evaluate the efficiency of replacing slag by ceramic waste and fibers to limit shrinkage and subsequently prevent crack formation. The formation of these cracks could significantly degrade the mechanical performance of the mix compositions. Regarding the observations, all specimens remained intact and no cracks were detected on their surfaces, which demonstrates the efficiency of the hybrid blends and fibers used in controlling the shrinkage.

In the first phase of the present study, mechanical properties were characterized and interpreted. Regarding the results, the prismatic beams were tested under a concentrated load using different curing regimes, which greatly affected the flexural performance of the specimens. Therefore, it was necessary to assess the flexural performance of the beams under a distributed load with a longer span. Thus, the second phase of the present study involved assessing the flexural performance of the compositions under a distributed load and a longer span. To achieve this aim, finite element models were initially developed to simulate the flexural performance of the prismatic beams using an inverse analysis. Afterward, the reliable models used to evaluate the flexural performance of compositions under distributed loads and longer spans.

The efficiency of this methodology was approved by the various studies in [76-80]. The previous findings used this approach to assess the flexural performance of fiber reinforced alkali-activated binders with larger spans as well as using for repairing the concrete beams [76-80].

Employing the numerical simulations provides this possibility to evaluate the mix compositions under different load conditions, which were not experienced experimentally.

4. Numerical simulations

A finite element method (FEM)-based software package capable of simulating nonlinear behaviors of several types of materials was employed at this stage of the study. The numerical simulation was carried out using FEMIX software. Fibers bridging micro cracks contribute to the formation of diffuse crack patterns since they offer resistance to the coalescence of earlier micro cracks into macro cracks. Therefore, smeared crack constitutive models are, conceptually speaking, more appropriate than discrete crack models to simulate crack initiation and propagation in reinforced specimens. Moreover, to capture inelastic deformations, compression and tension properties should be introduced to the smeared crack constitutive models. A trilinear stress–strain–softening diagram was used to capture the post-cracking behavior of reinforced beams in tension. The accuracy of FEM modeling largely depends on the fracture parameters (the fracture mode I crack propagation).

In order to analyze the prismatic beams with finite element models, the beams were considered as plane stress problems. The explanation of the formulation of the multidirectional fixed smeared crack constitutive model is related to the case of cracked concrete, at the domain of an integration point (IP). The specimens were modeled by a mesh of 8-node “serendipity” plane stress finite elements. The Gauss-Legendre integration scheme with 2×2 IP was used in all elements. Smeared crack constitutive models are, conceptually, more appropriate than discrete crack models in the simulation of crack propagation in fiber reinforced cement concrete, mainly, in those with a high number of redundant supports. According to the adopted constitutive law for plane stress finite element, stress and strain are related to each other by:

$$\Delta\sigma = D^{Crcon} \Delta\varepsilon \quad (3)$$

Where $\Delta\sigma = \{\Delta\sigma_1, \Delta\sigma_2, \Delta\tau_{12}\}^T$ and $\Delta\varepsilon = \{\Delta\varepsilon_1, \Delta\varepsilon_2, \Delta\gamma_{12}\}^T$ are the components of stress and strain vectors, respectively. By decomposing the total strain in equation (3) into elastic and cracked concrete parts, i.e. $\Delta\varepsilon = \Delta\varepsilon^{ela} + \Delta\varepsilon^{cra}$, D^{Concra} will be determined using [59, 60]:

$$D^{Crco} = D^{Co} - D^{Co} [T^{cr}]^T (D^{cr} + T^{Cr} D^{Co} [T^{Cr}]^T)^{-1} T^{cr} D^{Co} \quad (4)$$

where D^{Con} is the constitutive matrix of concrete assumed to have a linear behavior:

$$D^{Co} = \frac{E_c}{1-\nu_c^2} \begin{bmatrix} 1 & \nu_c & 0 \\ \nu_c & 1 & 0 \\ 0 & 0 & \frac{1-\nu_c}{2} \end{bmatrix} \quad (5)$$

which E_c and ν_c are elasticity modulus and Poisson's ratio of concrete, respectively. T^{Cr} in equation (4) is the transform matrix of stress components from the coordinate system of element to local crack coordinate system. If n is the number of cracks, T^{Cr} will be defined as:

$$T^{Cr} = [T_1^{Cr} \dots T_i^{Cr} \dots T_n^{Cr}]^T \quad (6)$$

where, the crack orientation matrix is given by:

$$T_i^{Cr} = \begin{bmatrix} \cos^2 \theta & \sin^2 \theta & 2\sin \theta \cos \theta \\ -\sin \theta \cos \theta & \sin \theta \cos \theta & \cos^2 \theta - \sin^2 \theta \end{bmatrix} \quad (7)$$

and θ is defined as the angle between the x_1 axis and the vector orthogonal to the plane of the i_{th} crack. Furthermore, D^{Co} in equation (4) is defined as a matrix that includes the constitutive law of the n cracks:

$$D^{Cr} = \begin{bmatrix} D^{Cr}_1 & \dots & 0 & \dots & 0 \\ \dots & \dots & \dots & \dots & \dots \\ 0 & \dots & D_i^{Cr} & \dots & 0 \\ \dots & \dots & \dots & \dots & \dots \\ 0 & \dots & 0 & \dots & D_n^{Cr} \end{bmatrix} \quad (8)$$

In the above equation, D_i^{Cr} is the crack constitutive matrix of the i^{th} crack, which is defined as:

$$D^{Cr} = \begin{bmatrix} D_{I,i}^{Cr} & 0 \\ 0 & D_{II,i}^{Cr} \end{bmatrix} \quad (9)$$

$$D_{I,i}^{Cr} = -k_i \frac{l_{cr} f_{ct}^2}{G_f}$$

$$k_1 = \frac{(1-\alpha_1)(\xi_1 + \alpha_1 \xi_2 - \alpha_2 \xi_1 + \alpha_2)}{2\xi_1}$$

$$k_2 = \frac{(\alpha_1 - \alpha_2)(\xi_1 + \alpha_1 \xi_2 - \alpha_2 \xi_1 + \alpha_2)}{2(\xi_2 - \xi_1)}$$

$$k_3 = \frac{\alpha_2(\xi_1 + \alpha_1 \xi_2 - \alpha_2 \xi_1 + \alpha_2)}{2(1-\xi_1)}$$

where, $D_{I,i}^{Cr}$ and $D_{II,i}^{Cr}$ are the moduli corresponding to the fracture mode I (normal) and fracture mode II (shear) of the i^{th} crack, respectively. In this numerical approach, a new crack forms in an integration point (IP) when the angle between the new crack and the already existing cracks exceeds a certain threshold angle (This angle is selected between 30-60 degrees according to [59]). The crack opening propagation was simulated using the trilinear diagram, which is indicated in Fig. 6a.

Normalized stresses (α_1 and α_2) and strains (ξ_1 and ξ_2) were defined and used for transposition points between the linear segments of the diagram. The ultimate crack strain (ε_n^{Cr}) is a function of the fracture energy, tensile strength (σ_t^{Cr}), crack bandwidth (l_{Cr}) and normalized strains and stresses. Therefore, the ultimate crack strain is given by [60]:

$$\varepsilon_n^{Cr} = \frac{2}{\xi_1 + \alpha_1 \xi_2 - \alpha_2 \xi_1 + \alpha_2} \left(\frac{G_f}{l_{Cr} \sigma_t^{Cr}} \right) \quad (10)$$

and the fracture mode II modulus, D_{II}^{Cr} , is defined by [61]:

$$D_{II}^{Cr} = \frac{\kappa}{1 - \kappa} G_c \quad (11)$$

where, G_c is the elastic concrete shear modulus and κ is the shear retention factor. The parameter κ is considered as a function of the current crack normal strain (ε_n^{Cr}) and ultimate crack normal strain (ε_n^{Cr}) [62]:

$$\kappa = \left(1 - \frac{\varepsilon_n^{Cr}}{\varepsilon_n^{Cr}} \right)^{P_1} \quad (12)$$

If $P_1=1$, κ will decrease linearly as ε_n^{Cr} increases. Higher values of the exponent results in more decrease in κ [61]. Regarding fracture mode II, the total and the incremental approaches, depicted in Fig. 6b, and more details explained in [61], are available in FEMIX, but in the numerical simulations implemented the total approach was adopted due to its better performance.

Fig 6. a) Trilinear stress-strain diagram in simulation of the fracture mode I crack propagation (

$\sigma_{n,1}^{Cr} = f_{ct}$, $\sigma_{n,2}^{Cr} = \alpha_1 \sigma_{n,1}^{Cr}$, $\sigma_{n,3}^{Cr} = \alpha_2 \sigma_{n,1}^{Cr}$, $\varepsilon_{n,2}^{Cr} = \xi_1 \varepsilon_{n,u}^{Cr}$, $\varepsilon_{n,3}^{Cr} = \xi_2 \varepsilon_{n,u}^{Cr}$); b) Relation between crack shear

stress and crack shear strain for the incremental and total approaches

It is worth mentioning that continuum models, such as micro plane constitutive law, can be successfully employed to simulate crack propagation in cementitious binders under dominant tension, compression, and complex loading [63-66]; meanwhile, in the present study, smeared crack constitutive models were used to simulate crack propagation in fiber-reinforced binders.

As there is no data on the experimental results of this work as related to the direct tensile test and tensile properties of the reinforced one-part alkali-activated slag and ceramic binders with PVA, PP, and steel fibers, the unknown parameters of tensile characteristics were obtained by executing an inverse analysis. Thus, an inverse analysis procedure was implemented to obtain the unknown fracture parameters of the tensile properties of the alkali-activated binders. This analysis was carried out to best fit the experimental results in terms of force-deflection curve. The parameters were obtained by fitting the force versus deflection curves with the minimum error, which was computed using the following equation:

$$Error = \frac{|A_{exp} - A_{Num}|}{A_{exp}} \times 100 \quad (13)$$

where A_{exp} and A_{Num} are the areas below the experimental and numerical force versus deflection curves, respectively.

Moreover, to obtain mesh refinement, a sensitivity analysis was conducted, which did not affect the simulation accuracy. The sensitivity analysis was accomplished by refining the mesh under linear elastic behavior only to obtain result stabilization. To achieve this aim, three levels of mesh were submitted to the numerical model, including fine (using 256 element and the element size 2.5×2.5 mm), medium (using 64 element and the element size 10×10 mm), and coarse (using 16 element and the element size 20×20 mm). Regarding the preliminary numerical results, the numerical model with medium and fine meshes produced almost similar load vs. mid-span deflection relations to the experimental one. Regarding the accuracy of the numerical simulations and the computational cost, the medium mesh was selected to simulate the numerical models.

In the numerical simulations, beams with all details were modelled using 64 elements, and the double symmetry technique was not used.

The loading and support conditions were simulated in agreement with the experimental flexural test setup. As shown in Fig. 7a, the supports with null displacements in the Z-axis were restricted at both ends of the beams; likewise, the supports with null displacements in the direction of the Y-axis were restricted to the left side. In the experimental tests, the flexural load was imposed on the beams through one load line. This disposition was copied to the numerical model. The total applied deflection in each slab was divided into a series of deflection increments. The Newton-Raphson incremental-iterative method was adopted by evaluating the tangent stiffness matrix in each iteration. A tolerance of 0.001 was adopted for assuring

convergence. Stability failure was considered as the main failure criterion and occurs when a very large displacement suddenly transpires in the force versus deflection curves of the beams. Fig. 8 represents the verified numerical results using the experimental tests. The numerical results were well-fitted to the experimental results and effectively captured the flexural performance of the beams. Regarding the calculated error in Table 4, the maximum error between the areas below the experimental and numerical force versus deflection curves was computed as 6%, which demonstrates the reliability of the developed finite element models to capture the flexural performance of the prismatic beams reinforced with different fibers and cured in different curing conditions. The parameters of fracture mode I, including crack strength, fracture energy, normalized stresses, and strains, are listed in Table 4.

Fig. 7. a) Adopted test setup and boundary conditions for the simulated prismatic beams; b) Adopted test setup in [77] to impose the distributed flexural loading. Fig. 8. Comparison between the experimental and numerical simulations for: a) PVA0.5; b) PVA1; c) PVA0.5W; d) PVA1W; e) ST0.5; f) ST1; g) ST0.5W; h) ST1W; k) PP0.5; l) PP1; m) PP0.5W; n) PP1W.

Table 4. The unknown parameters obtained using the inverse analysis.

Fig. 9 represents the elastic modulus, tensile strength, and fracture energy obtained from the numerical simulations. According to the results, using a water bath for the reinforced compositions with PVA fibers instead of sealing reduced the elastic modulus and significantly increased the fracture energy (more than 5 and 3 times for the reinforced compositions with 0.5 and 1% PVA fibers, respectively), while the tensile strength was almost constant. Since a hardening deflection was observed for the specimens reinforced with PVA and cured in a water bath, the indicated tensile strength in this case was related to the tensile stress of the initial crack formation; in addition, by using high fracture energy and hardening behavior, this tensile stress can be increased to reach its maximum value. Meanwhile, for the compositions reinforced with PVA fibers and cured using sealing, the tensile stress entered the softening phase after the initial crack formation, and the indicated tensile strength was related to the maximum tensile stress. The increase in fiber content increased the fracture energy and reduced the elastic modulus.

For the compositions reinforced with steel fibers, the fracture energy was almost constant for all mixtures, while the elastic modulus and tensile strength consistently increased with increased steel fiber content and use of a water bath instead of sealing for curing.

Increasing the content of PP fibers also significantly increased the tensile strength and elastic modulus of the mixtures. Additionally, the maximum influence of the curing regime was noticed on the fracture energy of the binders.

Fig. 9. Elastic modulus, tensile strength, and fracture energy obtained from the numerical simulations.

Increasing the span length to a depth ratio equal to or greater than 2.5 results in flexural failure in the beams [55]; therefore, the length of the beams was increased to 800 mm. Moreover, the width of the specimens was increased from 40 mm to 800 mm to acquire a better understanding of the flexural performance of the compositions under the distributed loading. On the other hand, for the next stage of numerical simulation, some reinforced plates (or one-way bending slabs) made with one-part alkali-activated slag and ceramic binders were simulated and subjected to the distributed loading.

4.1. Load carrying capacity under the distributed load

As shown in Fig. 7b, the flexural test setup used to impose the distributed load was adopted based on the one used in [62]. To simulate the plates, 320 elements were employed. The distributed load was imposed to a span length of 760 mm. As with the simulated prismatic beams, similar boundary conditions and the same failure criterion were considered to model the plates (or one-way bending slabs) with a length of 800 mm.

The numerical results in Fig. 10 represent the flexural performance of the slabs under the distributed loading. Similar to the flexural performance of the prismatic beams, the flexural performance of one-way bending slabs showed that it is governed by the fiber type, content, and curing regimes. The deflection control limits of (δ : L/180: 4.2 mm) and (δ : L/360: 2.1 mm) are commonly used in most design codes [67]. It is clear that the permissible service loads based on the defined limits were well below the ultimate capacity, indicating that the developed fiber-reinforced one-part alkali-activated slag and ceramic binders provide high safety factors.

Fig. 11 illustrates the maximum flexural capacity and ultimate deflection corresponding to the maximum flexural capacity of plates under the imposed uniform load. Regarding the results, the maximum load carrying capacity ($\approx 40 \text{ kN/m}^2$) and ultimate deflection ($\approx 17 \text{ mm}$) were observed in the reinforced compositions using steel (with 1%) and PVA (with 0.5%) fibers, respectively.

Due to the use of a water bath instead of sealing, the maximum improvement of the maximum flexural load and ultimate deflection were recorded as about 3.5 and 2.5 times in the compositions reinforced with PP (0.5% fiber) and PVA fibers (0.5% fiber), respectively.

Asgari et al. showed that the flexural capacity of one-way slabs (with similar dimensions) made with cementitious binders and reinforced with glass fibers (1, 1.5, and 2% in Vol.) varied in the range of 12–30 kN/m^2 under the distributed loading [77]. Additionally, mid-span deflection was measured up to 11 mm in different mixtures. Furthermore, the flexural capacity of the reinforced

compositions changed from 10 to 40 kN/m². Comparing the results obtained from this study with the literature [77] revealed that the developed materials discussed in this paper, in addition to producing low CO₂ emissions (compared to OPC [68]) and being easier to prepare and cast (compared to traditional alkali-activated binders containing dangerous solutions [16]), demonstrate appropriate structural performances for use in construction applications.

Fig. 10. Force versus mid-span deflection curves under the distributed loads for the reinforced specimens with: a) PVA fiber; b) steel fibers; c) PP fiber.

Fig. 11. Flexural capacity of the one-way bending slabs under distributed load.

The crack patterns of some simulated slabs at the end of the analysis (i.e., at the end of the last converged load increment) are depicted in Fig. 12. Crack patterns shown in red indicate fully open cracks; patterns in pink indicate that the cracks are reopening; blue patterns indicate cracks that are closing. According to the crack patterns illustrated in Fig. 12, multiple cracks formed under the tensile surface of the slab, which was simulated by using the properties of PVA0.5W. This crack formation demonstrates a deflection hardening behavior for the slabs subjected to the flexural loading that enables the slabs to significantly improve its ultimate load and deflection capacities, as indicated in Fig. 10a. The concentration of the cracks in the fully open status indicates an almost brittle behavior.

Fig. 12. Crack patterns of some simulated one-way bending slabs under distributed load: a) PVA0.5W; b) ST1W; c) PP1W.

5. Conclusions

This paper presented the results of both experimental and numerical investigations of the development of fiber-reinforced one-part alkali-activated slag and ceramic binders. The study aimed to explore the effects of using different fiber types and contents on hardened properties of one-part alkali-activated slag and ceramic binders. Moreover, the effects of two curing regimes (sealing and water bath), both of which are commonly used, were also investigated. In the first stage, the physical and mechanical properties of the compositions were evaluated experimentally. Afterward, the flexural performance of the reinforced compositions under distributed load was evaluated by using numerical simulations. Regarding the results reported herein, the following observations can be made:

- Incorporating fibers and using different curing regimes did not substantially affect water absorption.
- Regardless of fiber type and curing method, increasing the fiber content increased the compressive and flexural strengths.

- Using a water bath significantly enhanced the mechanical characteristics of the reinforced compositions compared to sealing, and this improvement was significantly governed by the fiber type. This improvement could be explained by an increase in the degree of polymerization and a decrease in the porosity.
- Using a water bath led to a greater improvement in the flexural performance of the compositions reinforced with steel and PP fibers, compared to PVA fibers. The maximum increase of the ultimate deflection was measured in the compositions reinforced with PVA fibers due to their hardening behavior.
- In general, using a water bath to cure the specimens had a greater effect on increasing the flexural strength of the reinforced compositions compared to compressive strength.
- Numerical results revealed that using a water bath instead of sealing had the greatest impact on the fracture energy of the reinforced binders with PVA and PP fibers. This is because increasing the elastic modulus and tensile strength in the compositions reinforced with steel and PP fibers is significantly influenced by using a water bath and increasing fiber content.
- Regarding the flexural load carrying capacity presented for the reinforced binders under the distributed loading, it seems that the developed binders have adequate potential for use in construction applications. That said, further investigations into durability performance are needed.

Promisingly, this study showed that fiber-reinforced one-part alkali-activated slag and ceramic binders should be used due to their environmentally friendly (compared to OPC) composition, easy preparation (compared to traditional alkali-activated binders employing impractical alkali activator solutions), and significant fiber-introduced enhancements in terms of mechanical properties.

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Fig. 1. Effect of curing conditions on water absorption of the reinforced compositions containing different fiber contents and types.

Fig. 2. Effect of curing conditions on the compressive strength of the reinforced compositions containing different fiber contents and types.

Fig 3. Effect of curing conditions on the flexural performance of the reinforced compositions: a) containing PVA fiber; b) containing steel fiber; c) containing PP fiber; d) in terms of ductility

Fig. 4. a) flexural strength; b) mid-span deflection corresponding to the flexural strength.

Fig. 5. SEM images from: a) PVA0.5W; b) PP1W; c) PP0.5; d) ST1W.

Fig. 6. a) Trilinear stress-strain diagram in simulation of the fracture mode I crack propagation ($\sigma_{n,1}^{cr} = f_{ct}$, $\sigma_{n,2}^{cr} = \alpha_1 \sigma_{n,1}^{cr}$, $\sigma_{n,3}^{cr} = \alpha_2 \sigma_{n,1}^{cr}$, $\epsilon_{n,2}^{cr} = \xi_1 \epsilon_{n,u}^{cr}$, $\epsilon_{n,3}^{cr} = \xi_2 \epsilon_{n,u}^{cr}$); b) Relation between crack shear stress and crack shear strain for the incremental and total approaches

Fig. 7. a) Adopted test setup and boundary conditions for the simulated prismatic beams; b) Adopted test setup in [77] to impose the distributed flexural loading.

a)

b)

c)

d)

e)

f)

Fig. 8. Comparison between the experimental and numerical simulations for: a) PVA0.5; b) PVA1; c) PVA0.5W; d) PVA1W; e) ST0.5; f) ST1; g) ST0.5W; h) ST1W; k) PP0.5; l) PP1; m) PP0.5W; n) PP1W.

Fig. 9. a) Elastic modulus; b) tensile strength; c) fracture energy obtained from the numerical simulations.

a)

b)

c)

Fig. 10. Force versus mid-span deflection curves obtained from numerical simulations under the distributed loads for the reinforced specimens with: a) PVA fiber; b) steel fibers; c) PP fiber.

Fig. 11. Flexural capacity of the one-way bending slabs under distributed load.

Fig. 12. Crack patterns of some simulated one-way bending slabs under distributed load: a) PVA0.5W; b) ST1W; c) PP1W.

Table 1. The chemical composition of slag and ceramic waste

Element / oxide, (% in Wt.)	GGBFS	Porcelain waste
Na ₂ O	0.51	2.200
MgO	10.24	0.310
Al ₂ O ₃	9.58	21.010
SiO ₂	32.33	67.060
P ₂ O ₅	0.01	0.080
SO ₃	4.00	0.020
K ₂ O	0.53	2.320
CaO	38.51	1.450
Cr ₂ O ₃	-	0.040
MnO	0.00	0.020
Fe ₂ O ₃	1.23	0.850
NiO	-	0.010
CuO	-	0.006
ZnO	-	0.004
Rb ₂ O	-	0.023
SrO	-	0.122
Cl	0.04	0.041
TiO ₂	2.21	0.352

Table 2. The mechanical properties of industrial steel, PP and PVA fibers

Fiber name	Length/Diameter (mm/mm)	Elastic modulus (GPa)	Tensile strength (MPa)	Elongation at break (%)	Density (g/cm ³)
Steel	10/0.03	200	2200	3	7.88
PP	10/0.012	9.6	910	<12	0.91
PVA	8/0.04	41	1600	6	1.30

Table 3. Molar ratios of the designed mix composition

SiO ₂ /Al ₂ O ₃	Na ₂ O/SiO ₂	Na ₂ O/Al ₂ O ₃	CaO/SiO ₂
6.36	0.15	0.94	0.89

Table 4. The unknown parameters obtained using the inverse analysis

Specimen designation	α	ξ	λ	β	Elastic Modulus (GPa)	Fracture energy (N/mm)	Tensile strength (MPa)	Error (%)
PVA0.5	0.20	0.21	0.40	0.50	35.5	0.25	5.45	2.11
PVA1	0.20	0.21	0.40	0.50	33.0	0.43	5.90	1.38
PVA0.5W	0.11	0.45	0.40	0.80	25.0	1.42	6.00	5.50
PVA1W	0.40	0.50	0.50	0.60	20.0	1.60	5.50	1.20
ST0.5	0.20	0.40	0.21	0.50	33.0	0.10	5.10	1.37
ST1	0.20	0.40	0.21	0.50	38.0	0.10	5.45	2.54
ST0.5W	0.20	0.40	0.21	0.50	50.0	0.10	9.00	3.44
ST1W	0.20	0.40	0.21	0.50	52.0	0.12	9.50	1.46
PP0.5	0.10	0.32	0.70	0.65	10.0	1.60	1.65	5.51
PP1	0.035	0.20	0.90	0.30	25.0	2.20	3.70	6.02
PP0.5W	0.39	0.30	0.40	0.50	48.0	0.10	8.20	1.03
PP1W	0.39	0.30	0.40	0.50	51.0	0.11	9.20	1.16