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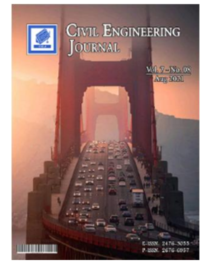

The Early Age Strength Improvement of the High Volume Fly Ash Mortar

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Abstract

In the last decade, the use of Fly ash as replacement to improve the strength and performance of the cement has become a part of mortar and concrete manufacturing. When the used amount of fly ash ranges from 20 to 25%, the proprieties of concrete and mortars such as strength and durability are improved, which also reduce the Portland cement consumption and its impact on environment. For some special applications the High-Volume Fly Ash (HVFA) (up to 50%) is recommended, but the use of HVFA is still limited because of the low early age strength. The aim of this study is to overcome the constraints caused by the use of the High-Volume Fly Ash, by upgrading the mortar using grinding to reduce the particle size, and by the application of an upsetting force to modify the behavior of swelling and to modify the crystal structure of ettringite in order to increase the early age strength of the mortar. The results show an increase in the rupture resistance at 7 days and 28 days by 60% and 30% respectively. Which will make the use of HVFA mortar possible in construction industry and therefore reduce more CO_2 emissions from the cement production.

Keywords: Fly ash; Mortar; Concrete; Swelling; Early Age Strength.

1. Introduction

The concrete and mortar are a widely used in construction industry due to its high strength and its durability. However, the production of the cement is accompanied with a high CO_2 emission. Therefore the use of other materials as replacement for cement has become a necessity [1]. In order to obtain a sustainable concrete and mortar with high strength resistance, different types of artificial pozzolanic materials such as fly ash, silica fume and furnace slag were used [2, 3]. Due to several factors, such as the higher consistency during the fresh stage, the superior durability with respect to chloride resistance and the lower material cost, the Fly ash is widely used in construction industry. The use of high volume Fly ash concrete (HVFA) decreases more the CO2 emission, by reducing the amount of the Portland cement [4]. The HVFA gives better results compared to Portland cement, where the strength resistance at late age as well as the resistance to chloride ion penetration are improved; also, the permeability and the shrinkage are reduced, which make the mortar and concrete have better durability characteristics [4].

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The fly ash influences the properties of the mortar by hydraulic and/or pozzolanic reactions. Strength properties are decreased with the increase of fly ash volume in the cement and not compensated till 90 days. After one year, the resistance of the concrete containing Fly ash generally exceeds that of the control group (100% Portland cement) due to the influence of pozzolanic materials, which favorize the formation of new calcium silicate hydrate (CSH) instead of Calcium Hydroxide (CH) [5, 6].

The spherical particles of Fly ash favorize the hydration kinetics when the specific surface is larger than the one of the cement. Moreover, the hydration kinetics, of all the cementitious materials, forms several types of ettringite by crystallization inhibitors, leading to the expansion and the swelling of the porous structure. With the incorporation of Fly ash as replacement of cement these problems are corrected by the effect of dilution. But, these advantageous characteristics are very limited at early age (3-7 days), especially with high volume Fly ash [7]. The mechanisms that lead to the expansion and the swelling of the material are difficult to interpret.

- As first hypothesis, the Expansion is caused by growth of the formed ettringite in the presence of lime, from anhydrous or hydrated aluminates. According to this hypothesis, the expansion results from the Early C3A hydration in the presence of different kind of calcium sulphate [8]. The diffusion of sulphate and calcium ions through the initially formed layer causes a gradual formation of ettringite in a small volume [9], inducing internal pressure and swelling of the grains.
- As second hypothesis the Expansion is caused by water absorption of the formed ettringite following a process of solubilisation and then crystallization in the presence of lime. This colloidal ettringite has the ability to adsorb a big amount of water molecules. The adsorption of water increase in the distances between ettringite particles, and accelerate the cohesive forces decreasing [10], which increases swelling process [7].
- The third hypothesis assume that the expansion is caused by the pressure of ettringite crystallization, formed by solubilisation-crystallization in a confined space. Based on a thermodynamic explanation [11], the expansion phenomenon can be explained by the pressure of crystallization. The third hypothesis is widely accepted [12].

The aim of this study is to overcome the constraints presented in early age low strength caused by the use of the High volume Fly ash, using the Grinding and the upsetting force in order to reach a sustainable product with less impact on environment.

2. Material and Methods

2.1. Portland Cement

To prepare the mortar samples the Portland cement (CPJ 45) with a minimum Clinker (CaO) content of 65% is used (Table 1). The mortars are made with clean siliceous sand (ES = 75%), where the chloride content is less than 0.2%, which complies with the standard (NF EN 933-8).

Table 1. Chemical Composition of cement (%)

Compositions	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	P_2O_5	TiO_2
%	18.14	3.77	3.02	67.58	1.40	4.26	0.29	1.18	0	0.34

2.2. Sand and Fly Ash

Beside the CPJ 45, the mortar contains limestone, Fly ash, and/or pozzolans. The Fly ash has produced in the Jorf Lasfar, based on the burning of coal, which is imported from South Africa (Figure 1). The Fly ash is produced from thermal power plants and come out as a variable material because of several factors such as (Figure 2):

- The type and mineralogical composition of the coal;
- The degree of coal pulverization;
- The type of furnace and oxidation condition and the manner in which the fly ash is collected, handled and stored [13].

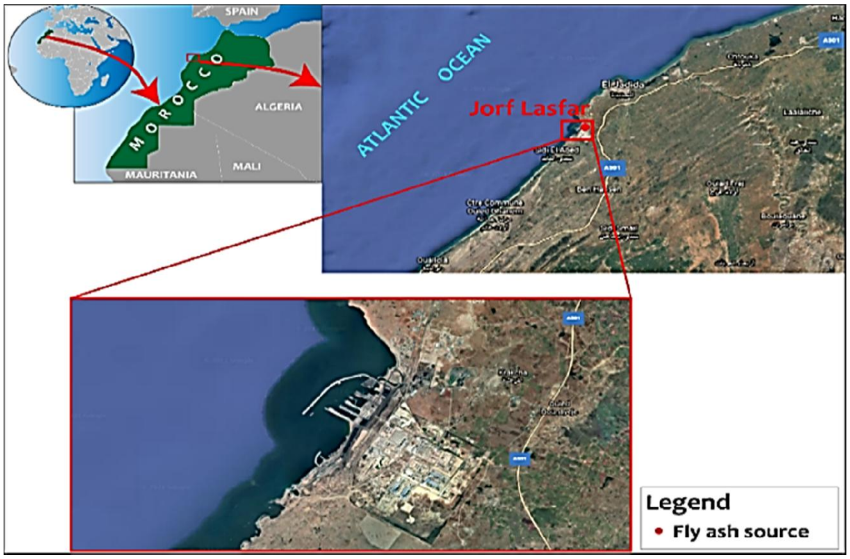

Figure 1. Location map of Fly ash sources

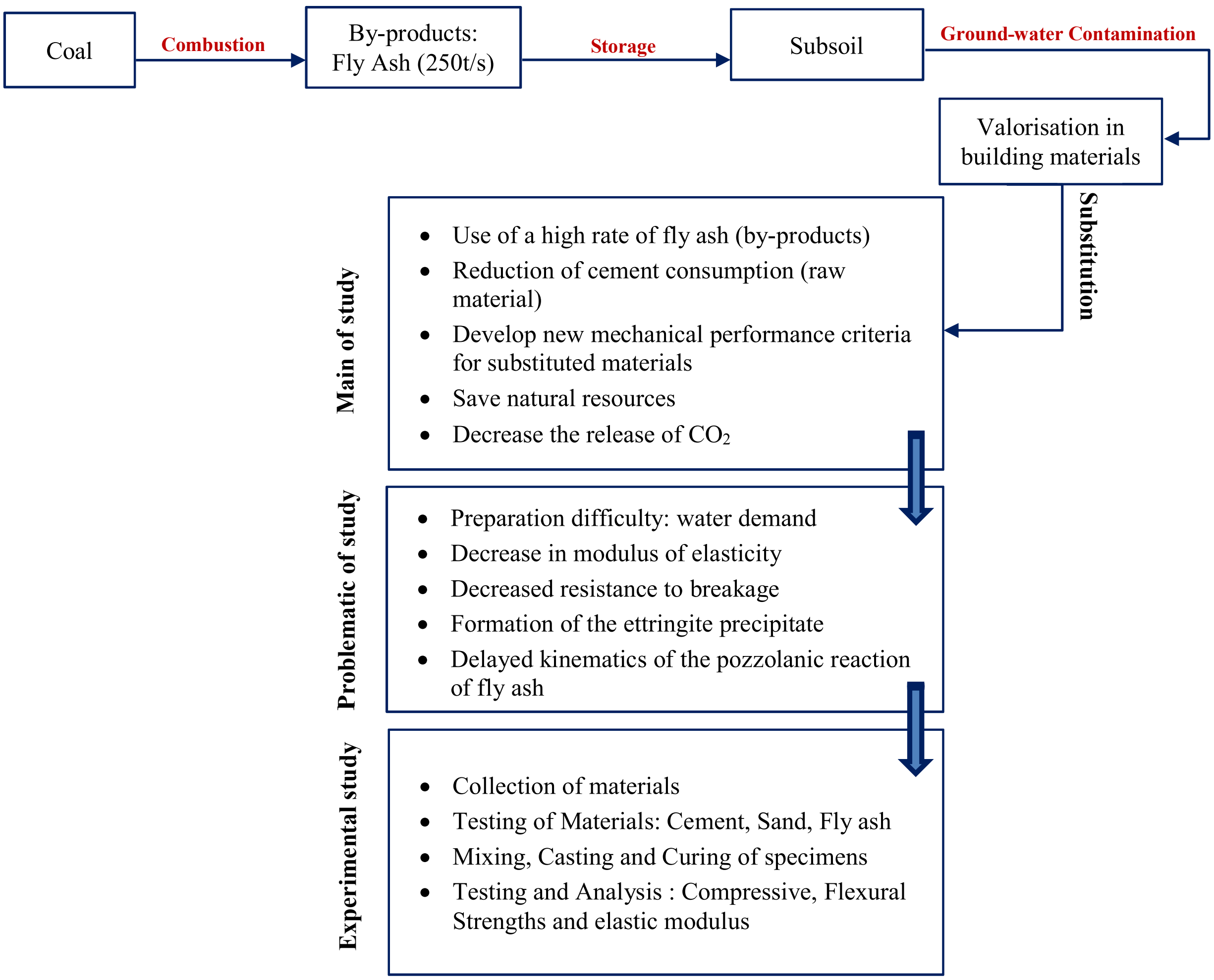

Figure 2. Flowchart of Research Methodology

The chemical composition of the Fly ash (Class F: silico-aluminous) is determined by the X-ray Fluorescence technique (Table 2). The class F fly ash has a lower CaO content and exhibits pozzolanic properties. The F class fly ash consist primarily of an alumino-silicate, which is the determining factor for pozzolanic activity. The crystalline minerals are generally composed of quartz, mullite, magnetite and spinel [14]. The size of the Fly ash particles is 500 µm, where the specific surface of particles is around 2.8 m^2/g.

Table 2. Chemical Composition of Fly ash (%)

Compositions	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	P_2O_5	TiO_2
%	50.05	32.13	5.07	5.06	1.08	0.82	0.69	2.13	1.16	1.82

2.3. Preparation of Mortars

To prepare the control mortar samples (100% Portland cement), the ratios (B/S) = 1/3 and W/B= 0.4) are used (Table 3).

Table 3. Formulations of the different mortar specimens used

Substitution (%)	Water/Binder	Cement CPJ45 (g)	Sand (g)	Water (g)	Fly Ash (g)
0	0.4	1000	3000	400	0
25	0.4	750	3000	400	250
50	0.4	500	3000	400	500

The prismatic samples (4×4×16 cm^3) are stored in a preservation bath until the rupture times 1, 3, 7, and 28 days. Where mortars with the fly ash as replacement of cement 25, and 50% are preserved under the same conditions (Figure 3).

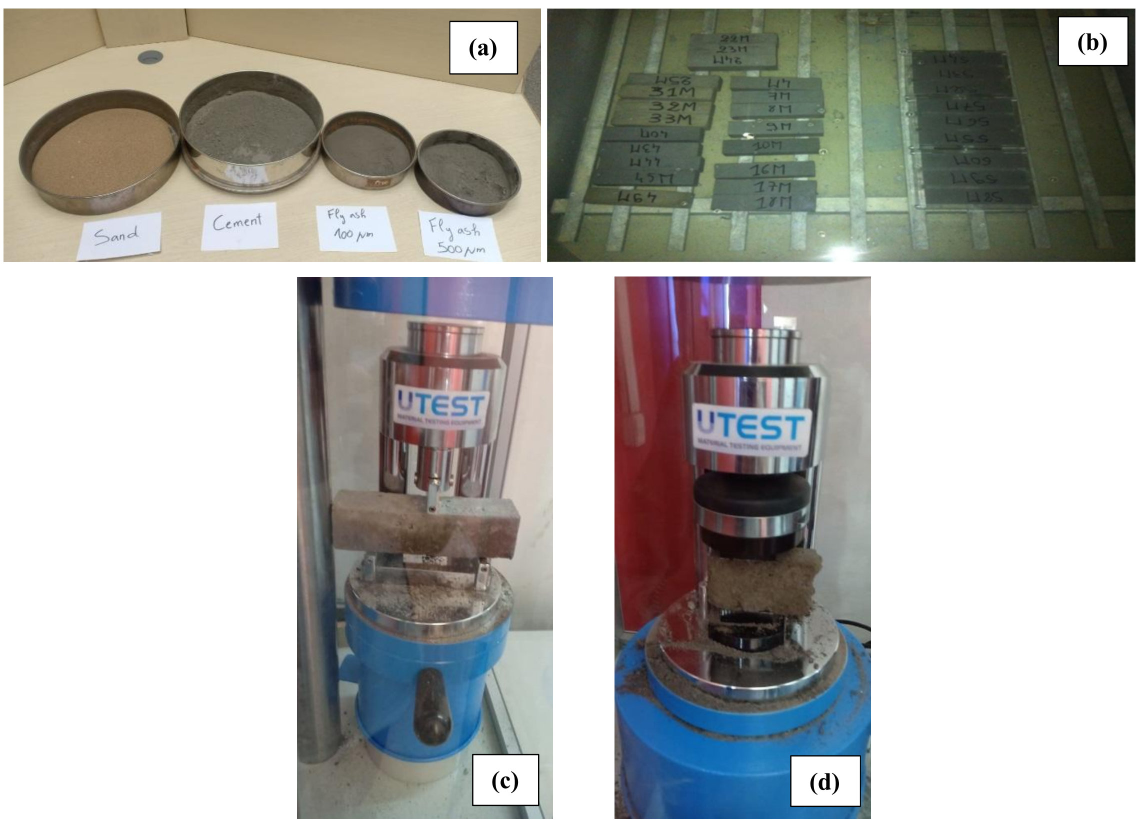

Figure 3. (a) Materials (b) Curing of samples, (c) and (d) Mechanical tests

The *FASTMILL 1.5 by Gabbrielli Technology was used* to grind particles of various sizes, ranging from micrometers to nanometers, with a system of agitation and separation of beads / ground product. The 10-minute grinding of the fly ash (500 µm) increases the specific surface area from 2.8 to 3.4 m^2/g, and decreases the particles size from 500 to 100 µm (Figure 4).

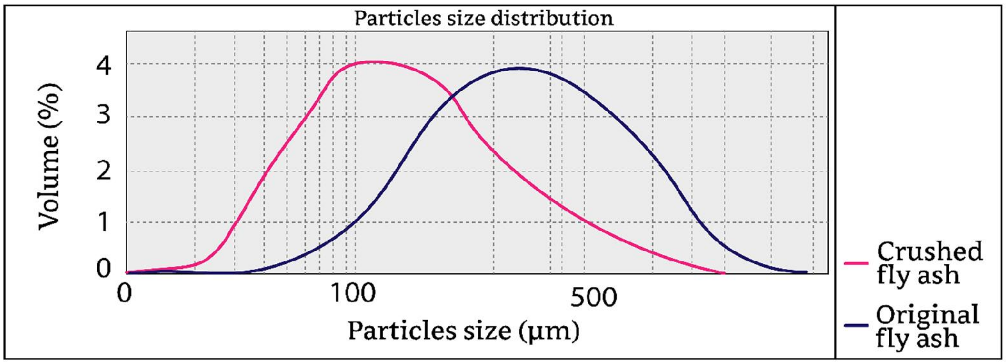

Figure 4. Fly ash particles size distribution

The prepared mortal is compacted in parallelepiped molds, where a metal plate is deposited on the upper face of the sample in order to exert an upsetting force. This force prevents sample expansion along the Z-axis (Figure 5). The mortar samples are kept in distilled water, until the rupture times, for 1, 3, 7, 14, 28, and 90 days. The densities of the samples are measured before each uniaxial compression test.

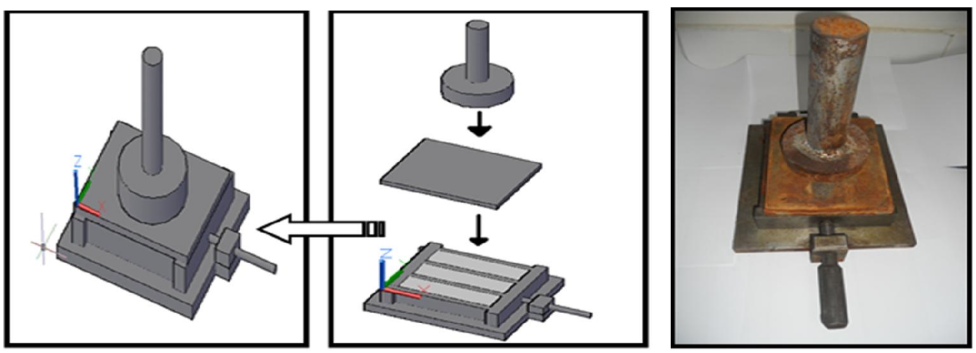

Figure 5. The used device to apply the upsetting force (Design by AutoCAD 2018 software)

3. Results and Discussions

The prismatic samples are prepared to determine the compressive strength. At the ages of 1, 3, 7, 14 and 28 days. The Figures 6 and 7 show the evolution of the resistance for samples with 0, 25, and 50% of Fly ash with various grain sizes. The Incorporation of fly ash in mortar significantly reduces the bleeding and segregation [7]. This result is related to the lubrication effect of the glassy spherical of the fly ash particles and the increased ratio of W/B which decreases the segregation and improves the concrete pumpability.

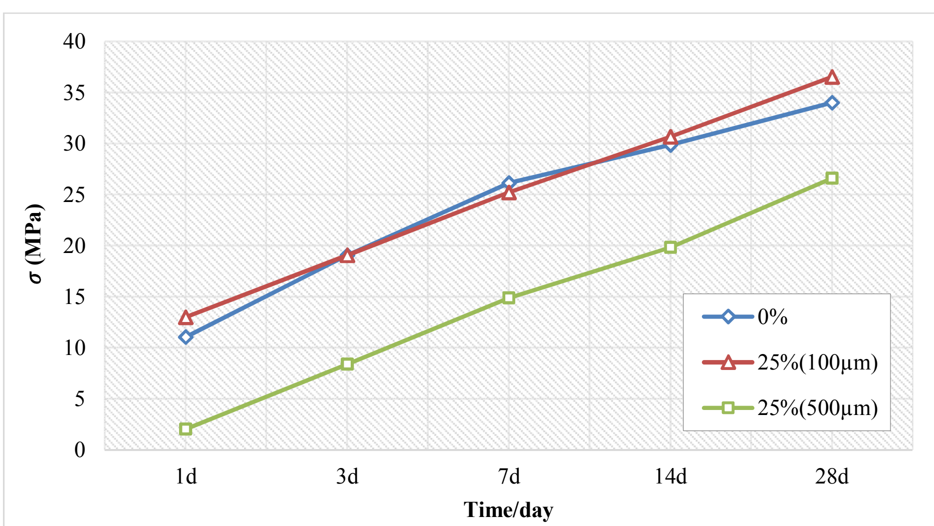

Figure 6. Evolution of compressive strength as a function of time (Replacement up to 25%)

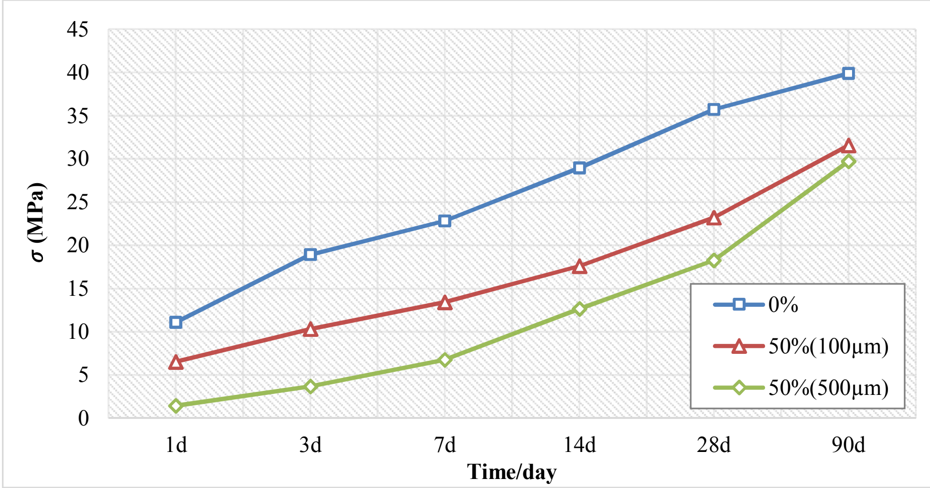

Figure 7. Evolution of compressive strength as a function of time (replacement up to 50%)

The results show that the use of Fly ash particles of 100 µm instead of 500 µm decreases the water requirement and then improves the strength of the mortar. Other studies also conclude that the fineness of the Fly ash is the main factor that affects the compressive strength of the mortar [15, 16]. The use of finer Fly ash (100µm rather than 500µm) decreases the water requirement of the replaced cement, which improves the strength of the mortar [7]. The Grinding of the fly ash increases the fineness, which has a positive effect on the reactivity [17]. According to other studies [18, 19], the use of Fly ash as a pozzolanic material gives performance to the mortal and improves the strength of it. In the other side some studies show that beyond an optimal grinding period (2h), the beneficial effect of this process is insignificant [20], so the fineness of fly ash is related to its pozzolanic activity [21].

As results of this study, the use of Fly ash (100 µm) as replacement of cement (up to 25%) leads to increase the resistance by approximately 80% at an early age which is a significant result (Figure 6). The growth in compressive strength as a function of fly ash content can be explained by the improvement in the stiffness of the product through the formation of new bonds (Table 4). But, despite the improvement in strength after grinding, the values remain low at a young age with HVFA (Figure 7).

Table 4. Density´s Evolution of Materials (g/cm^3) with different % of Fly Ash volume

Density / Days	1 d	3 d	7 d	14 d	28 d
25% FA (100 µm)	2,094	2,097	2,132	2,136	2,15
25% FA (500 µm)	1,602	1,605	1,608	1,91	2,126
ρ (%) (25%)	23%	23%	25%	10%	1%
50% FA% (100 µm)	2,009	2,098	2,073	2,1	2,191
50% FA% (500 µm)	1,527	1,53	1,535	1,637	2,043
ρ (%) (50%)	24%	27%	26%	22%	7%

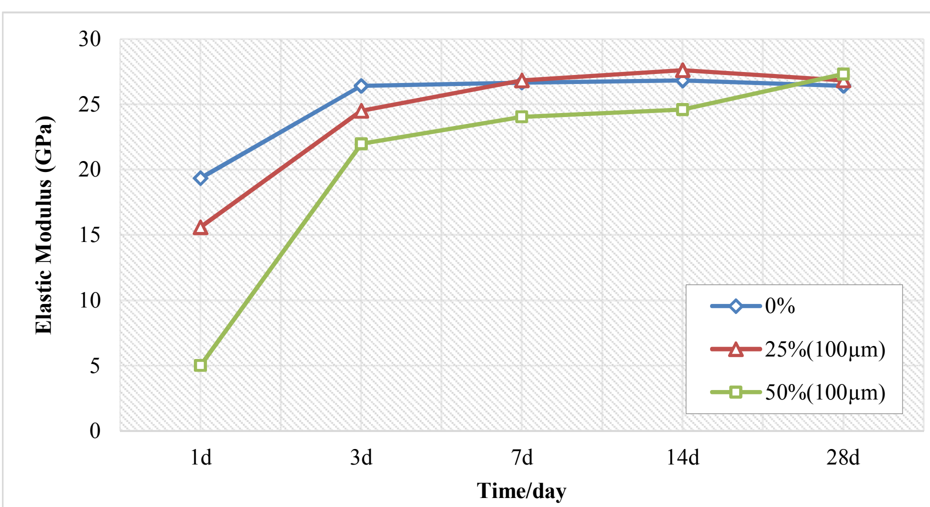

Figure 8. Evolution of elastic modulus according to Fly ash replacement

The formation of new links increases the compactness, which causes the shrinkage of concrete and mortar. In order to validate this hypothesis, the elastic modulus of various samples is measured (Figure 8). The external C-H and C-S-H are intensively formed, directed into empty and waterlogged spaces, which leads to increase the density of the 3D network, and results in massive Portlandite crystals formation in empty spaces, where the size could reach a hundred of µm. As the hydration progresses, reaction products grow in size and mass, but new hydrates continue to form and settle in the voids initially occupied by the ettringite needles. The use of Fly ash as replacement of cement promotes the formation of C-S-H instead the C-H. The C-S-H stores the reagents and influences the response of materials with respect to the pressures developed during the crystallization of ettringite.

The formation of bonds with the increased compactness are factors that increase the rigidity and decrease the pore volume. The combination of these two factors allows the material to resist swelling due to secondary ettringite formation reactions. Therefore, the number of bonds formed between the hydrates of the cements due to the dilution effect is increased. While the pozzolanic reactions is activated, the crushing process of the cenosphere from the Fly ash increases the density and fineness. As result, the pozzolanic reactivity is gradually induced [22]. Because of its fineness as well as pozzolanic reactivity, the fly ash in concrete significantly improves the density of cement paste and the micro-structure of the transition zone between the binder matrix and the aggregate [23]. As result of the continual process of pore refinement, the strength development is achieved.

The mechanical grinding process leads to reach an optimal result with a Fly Ash volume of 25%. Whereas, with a high Fly ash content (up to 50%), the results are insufficient at early age. in order to oppose the expansion and bring the constituent particles together and create new links at an early age, an upsetting force has been imposed. The application of the upsetting force results in the increase of the compressive strength from 15% to 25% at early age (Figure 9), and then reduce the gap with the control sample.

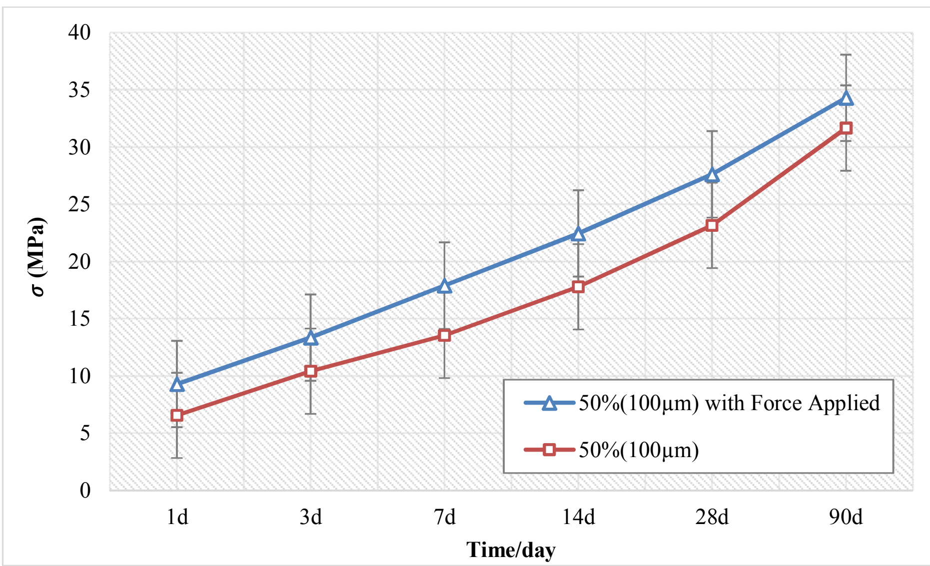

Figure 9. Evolution of compressive strength as a function of time

The application of an upsetting force increases the compactness and therefore makes the mortar withstand the force developed by the expansion of the ettringite, which is the responsible for the weakening of compressive strength at a young age. The imposed stress on the mortar during its hardening and development of the crystallization pressure decreases the demand of water, which leads to reduce the reactive movements [24-26]. The reduction in the initial porosity of the mortar increases the density by 4% (Table 4). In addition, it is necessary to consider two factors at an early age: The evolution of a viscous material towards a solid material and the absorbance of water, which could form micro-cracks that is responsible for the water gradient within the mortar due to the fineness of the Fly ash [27-29].

The modulus of elasticity is proportional to the density, and it is related to the number of links formed. In this study, it is assumed that when an upsetting force is imposed, the constituent particles get closer to each other and therefore, enhances the formation of new bonds which allows an increase in the stiffness of the mortar. This higher rigidity allows the mortar to resist the expansion stresses developed by ettringite. This fact is confirmed by the increase of the elastic modulus values (Figure 10).

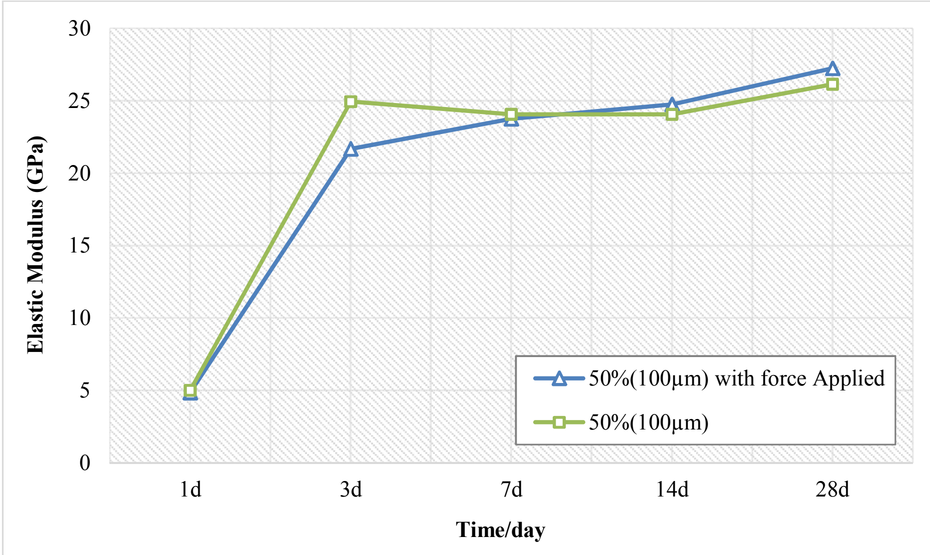

Figure 10. Evolution of elastic modulus as a function of time (50% of fly ash, 100µm)

The considerations formulated within the framework of the crystallization pressure theory indicate that the precipitation of the ettringite, which is formed during the first minutes, and the ettringite formed after 24 hours are not sufficient factors for the development of swelling. Where the ettringite is the responsible for hydration delay, which ensures the workability of the concrete and for the regulation of the cement setting reaction [30, 31]. Nevertheless, the ettringite formed between the 2nd and 7th days, is considered to be responsible for the disorders due to the swelling pressure. This pressure leads to the cracking or to significant variations in the density of the product. After hardening, the substituted mortar forms bonds and gain an appropriate density with less annoying pore. Therefore, the product resists the long-term compressive force, which is justified with the small difference in the compression strength.

The stress in a product resulting from the development of crystallization pressure is a function of the surface fraction of the crystals in contact with the matrix, which is a function of the porosity nature. Thus, the expansions are more important when pores are small and not very connected [32, 33]. Likewise, author authors have noted the importance of crack density, pore distribution and porosity for the development of swelling [34]. The nature of an expansion is related to the volume of cement paste in the material, which is an important factor to take into account during mixing. As result, the application of the upsetting force helps to decrease the effect of these problems.

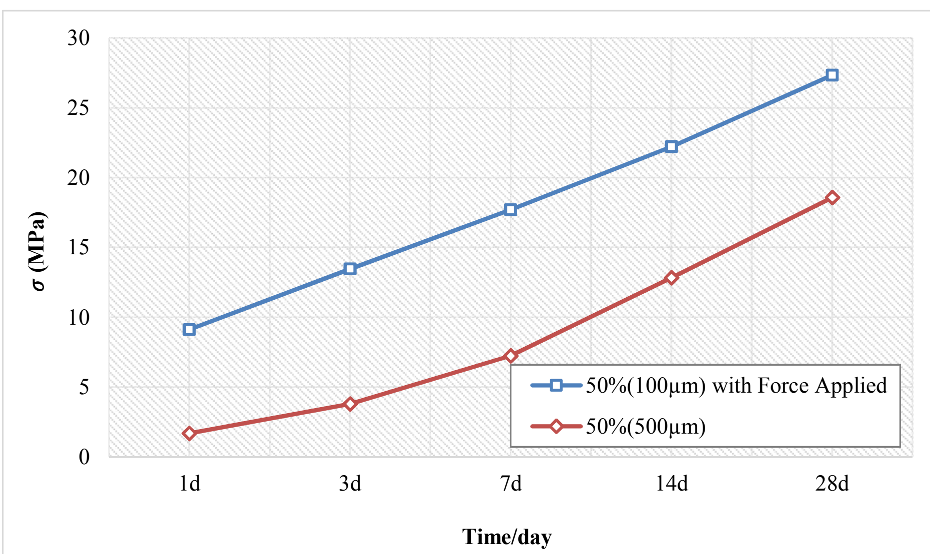

Figure 11. Evolution of compressive strength as a function of age (50% of fly ash, 100 and 500 µm)

The Figure 11 shows a considerable improvement in the compressive strength by the two upgrading methods, compared to the results obtained on the initial mortar with a fly Ash volume of 50% and particles diameter up to 500µm.

Where the breaking strength at the 3th day has increased from 4 MPa to 13 MPa, after grinding and imposing an upsetting force. The pozzolanic reaction of fly ash affect the compressive strength of concrete mixtures by different ways. Azadpour & Maghsoudi and Ogundipe et al. (2019) [19, 22] have identified these as:

- The reaction is slow, so that the rates of both heat liberation and strength development are correspondingly slow,
- The reaction consumes lime rather than producing it,
- The reaction products are efficient in filling up space and subdividing pores. Bazzar et al. (2013 and 2021) [7, 8] studies and recent experimental studies shows, that the rate of strength development depends upon the following factors:
- Fly ash characteristics such as its chemical and mineralogical composition, fineness, and pozzolanic reactivity,
- Type of cement,
- Replacement level of cement with fly ash,
- Mixture proportions,
- Ambient temperature,
- Curing environment reference.

4. Conclusion

It is the pore structure of the cement matrix that determines its porosity and permeability and therefore its sensitivity to transport and diffusion processes, the High Volume Fly Ash (HVFA) as replacement of cement reduces the bleeding and segregation, which reduces the water demand. In addition to their chemical contribution, the fly ash often has the effect of densifying the structure of the cement paste, which leads to limit the possibilities of ion exchange that is responsible for the ettringite precipitation. For this reason, the initial porous structure of an ash cement is initially more open than that of a conventional benchmark concrete, which results in lower mechanical performance at young ages. When the mechanical grinding process gives no sufficient results as it does in the case of low volume fly ash (up to 25%), an upsetting force is imposed, which allows to modify the behavior of swelling/porosity, and to orient the directions of crystallization of ettringite which improve the mechanical properties. Where the compressive strength at young age 7 days and long term 28 days are increased by 60 and 30% respectively. The findings of this study overcome the low early age strength caused by the use of HVFA and make the application of high-volume fly ash mortar and concrete possible in construction industry, which will reduce more CO_2 emissions from the cement production, these "ecological materials" are not only weak emitters; they also, prove to be quite efficient for the quality of the constructions and development the new formulation of concretes.

5. Declarations

5.1. Author Contributions

Conceptualization, software, formal analysis, investigation, resources, writing—original draft preparation: K.B.; Methodology, investigation, validation, supervision: A.H.A.; Author contributed to the final version of the manuscript and took the lead in revising the manuscript: F.Z.H. All authors have read and agreed to the published version of the manuscript.

5.2. Data Availability Statement

Data sharing is not applicable to this article.

5.3. Funding

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5.5. Conflicts of Interest

The authors declare no conflict of interest.

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