Effect of fly ash and synthesized superabsorbent polymer on the self-repair capacity of cement or concrete

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Abstract

Despite the fact that concrete is an essential component of modern infrastructure, its durability is a major problem affecting the life of the construction structures involving the costs of major repairs, this is due to the infiltration of water through the existing porosity in the structure causing chemical reactions that cause cracking and deterioration of the material. In this paper, it have been able to design a very resistant cementitious material with maximum durability in order to reduce maintenance costs and extend its life, incorporating a fly ash (FA) up to 40% from the incineration allows of waste medicines that to reduce this porosity and improving the compressive strength up to 61 MPa and the flexural strength up to 8 MPa and by adding polymers P1 and P2. These were synthesized by esterification reaction, in order to obtain a partially cross-linked resin E1 and E2 swelling in the presence of water, with an absorption rate for E1 of 9000% of its weight, which has the function of increasing the compressive strength and flexural strength of the cement mortar by 15% and 21% respectively compared to the mortar without addition, and especially to close the various remaining porosities thus preventing the penetration of water, the diameter of the resin which gives a homogeneous cementitious material with an optimal quantity was evaluated at 40 µ with 0.41% of E1 compared to the cement.

Keywords: Fly ash, Portland cement, Adhesive, Cross-linked resin, Compressive strength - Crack healing.

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1 Introduction

Cement is a hydraulic binder mainly used to produce concrete, the most versatile and sustainable construction material in the world [1], its consumption remains very important today and is estimated to increase fourfold by 2050 [2]. Cement is a major polluter of CO_2 since it releases into the atmosphere the equivalent of 5 to 8% of the world's CO_2 emissions [3]. The major problem of cement or concrete is its durability over time, leading to its cracking and deterioration due to the various chemical reactions caused by the penetration of water through the porosity of cement or concrete. Waste management plays a vital role in sustainable development since waste can be used as a replacement for raw material [4-5] in various fields. In the era of sustainable development, the common trends are: rationalizing the use of natural resources, energy and labor and the development of new pro-ecological technologies that reduce the level of pollution, minimize the consumption of raw materials and on the other hand, increase the recycling of waste.

In Algeria, waste is dumped in landfills without any revalorization, which represents a loss of raw materials and leads to a large number of environmental problems and pollution [6].

Cement pastes can be made using only Portland cement. However, its hydration releases a large amount of lime as a result of the hydration reaction of C_2S and C_3S (30% of the anhydrous mass of cement) [7] . This lime contributes to the loss of strength of the hydrated cement paste [8]. It can even be responsible for durability problems, since it can be easily leached by water, and this leaching increases the porosity of the cement paste. The partial substitution of a certain amount of cement by ash can be advantageous [9-10], ash is one of the most commonly used pozzolanic additives in the construction industry, it has pozzolanic properties, it contains silicate and aluminate phases capable of forming hydrates of the ternary system CaO-SiO_2- Al_2O_3-H_2O in the presence of lime and water [11]. The pozzolanic effect of ash is related to its ability to fix lime in aqueous medium, with production of new phases with the character of binder. When mixed with Portland cement, the fly ash consumes portlandite (calcium hydroxide), a hydrate of Portland cement, to form a silico-calcic gel ($CaOx$-SiO_2 y -H_2Oz) which constitutes the main phase of hydrated cement [12]. The incorporation of ash into cement has several economic and environmental benefits [13]. Several researchers have incorporated ash from coal combustion in power plants [14], ash from municipal waste incineration [15] and ash from industrial waste [16] into cement or concrete to improve its strength, knowing that the latter may contain heavy metals [17-18].

Part of our work has focused on industrial by-products called ash (FA) from the incineration of waste drugs are used in the field of civil engineering for improving the mechanical properties of cement pastes by reducing the porosity on the surface of cement, on the other hand, their use has the objective of reducing the consumption of cement, contributing in a simple and economic way to solve environmental problems. This experimental study consists of preparing a cement paste with mineral addition by replacing a certain percentage of cement by the pozzolanic addition of these ashes following the physical-chemical and mineralogical study of the FA that gave practically the same properties as the ashes from coal combustion [19-20] and meeting the standard EN 196-1 and EN 196-2 (ash that can be used in cement or concrete) [21].

The other part of this work was interested in the addition of resin that was synthesized in our laboratory from two polymers and incorporated into the cement paste.

Several works have incorporated polymers to improve the mechanical performance of the properties of cement systems [22-53], other superabsorbent polymers SAP have been incorporated into cement, in contact with an aqueous solution, water molecules diffuse into the polymer networks to hydrate the polymer chains, resulting in a water absorption of a few hundred times their own weight or more [23]. Studies of these SAP polymers have been carried out either for crack healing [24-25], or against freeze-thaw deterioration [26-27], or as an impediment to damage from chloride deterioration [28-52],Although several works have focused on the addition of cement with polymers such as polyester resin, epoxy resin and acrylic resin that are used as binders [29-31], the material that we have made is composed of a mixture of Portland cement, fly ash from the waste incinerations of drugs and E1 or E2 resin synthesized in our laboratory. The choice of E1 or E2 will depend on the compromise absorption rate and its ecological respect during its synthesis, this resin will absorb water and swell producing the closing of the pores in order to avoid possible chemical reactions leading to the appearance of cracks damaging the cement material.

This mixture allows to have a double environmental and economic impact, by the reduction of the quantity of cement by the incorporation of an industrial by-product and decreases the porosity of the cementitious material [54] as an added value and the addition of the resin allows its durability in the time by maintaining its resistance in the long term since no degradation is carried out following the closing of the porosity.Several researchers have used different percentages of resins in relation to aggregates, ranging from 10% to 20% [32-56], Our study focuses on using the maximum amount of fly ash wastes with a minimum amount of resin because a large amount of resin will result in an expensive cementitious material. Other researchers have been interested in studying the effect of resin size from a few tens of micrometers to a few millimeters [33-55], but in cementitious material, there have been few studies on this [34],. In this study the diameter and the rate of resin incorporated in the cementitious material was optimized, as this has a very significant influence on the homogeneity and total coverage of the material and on the cost of the latter. Thus, it was shown that the addition of cement to CV at 40% and an E1 resin with a diameter of 40μ requires an amount of resin of 0.41% with respect to the cement and at 20% of CV the same grain size is obtained with an amount of resin of 0.94% with respect to the cement. The mechanical performances of the cementitious material were evaluated by a comparative study of cement without any addition, then with addition at different contents in fly ash (Fa),fly ash-based cement and resin. The self-healing of the material was evaluated by visual observation over several days showing clear closure of the porosity of the cementitious material.

2 Experimental study

2.1 Raw Materials

In this work, a Portland cement CEM I 52.5 R was used which a pure Portland cement (PC) ground to a high fineness and which largely meets to the European cement standard (EN 197-1). Also, the Fly Ash (FA) is used as a cementitious material (Figure 1a). FA obtained from the incineration of medical waste of the incineration plant ECFERAL in ALGERIA and having a high specific surface with 40µm of size (Figure 1b). Several researches mentioned that the finer fractions of fly ash (FA) give a higher pozzolanic activity index than the coarser fractions when used in mortars [35]. Two types of resins were used (E1 and E2) and are synthesized from two polymers polyacrylic acid P1 and polyethylene glycol P2 in solution in the presence and absence of acid catalyst (Figure 1c).

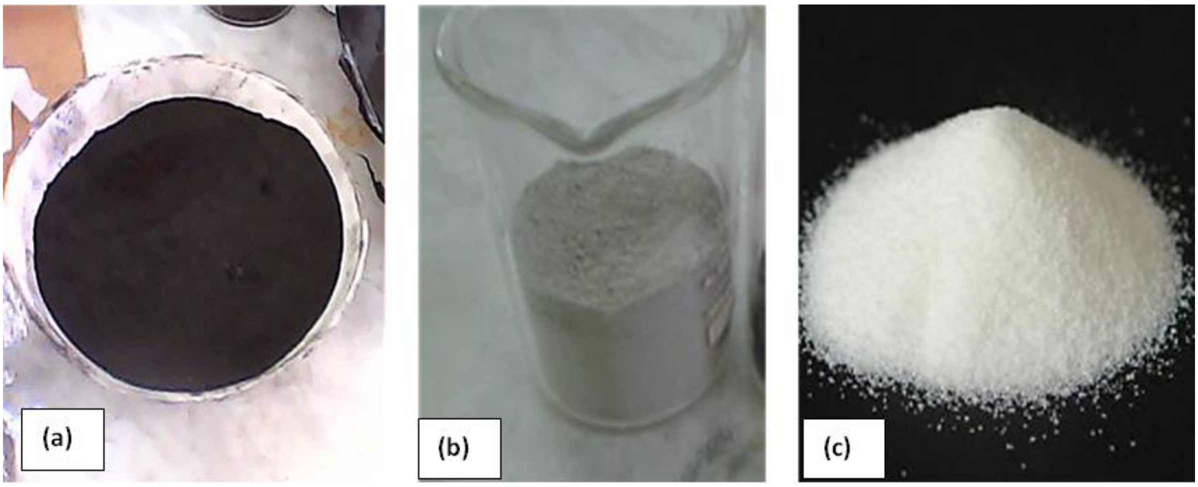

Figure 1 Materials used in this study: a) FA Grinding 40µm b) PC 52.5R (80µm) c) Resin E2

2.2 Characterization of obtained Fly ash (FA)

The black color of the FA indicates the presence of iron pyrite (FeS_2), thus informing us about its crystallinity which must be a glassy solid. The large specific surface of CV means that the material reacts easily with calcium hydroxide. The percentages in organic compounds of CV are in conformity with the standard fixed at 5% (Standard NF EN450 answering the valorization) (table 1).

Characteristics	pH	Color	Humidity %	% en organic	Density	SSA ($cm^{2/}g$)

FA	5.51	Black	10,3	1,75	1, 36	3145

Table 1 Physical and morphological characteristics

The chemical composition of major elements (Table 8 a) shows that the FA is a pozzolanic material consisting mainly of 46.3% SiO_2, 16.6% Al_2O_3 and 11.6% Fe_2O_3 [69] representing 74.5% of the content of the ash, it is a silicoalumina class F2 according to NF P 11-300 [36], andthe concentrations of other major elements are low, except for MgO which has a content of 11% and 16. 6 % in CaO , Studies [37-38], have indicated that fly ashes low in limestone (CaO< 8%) were more effective than highly calcareous fly ashes (CaO> 20%) from the point of view of durability, but that these ashes with a fairly high CaO content (16.6%) prove interesting in our case, because the dissolved Ca^{2+} could bind to the E1 resin and limit the initial swelling effect of E1 during the mixing of the mortar [39-40],.The sulfate and chloride contents are respectively 2.5% and 0.06% (Table 8 b) corresponding to EN 196-1 and EN 196-2 (ash suitable for use in concrete) [41-42],. The loss on ignition which is 1.3% reflects its leveling capacity in concrete according to NF EN 450 (limiting to 5%). Regarding minor elements (table 2) all the elements are weak; most of them are considered as inert and non-dangerous according to the European standard.

Table 2 Chemical composition of the FA

Elements	Composition (%)	Elements	Composition (ppm)
S_iO_2	46.3	As	20.11
Al_2O_3	16,6	Ni	524.66
CaO	05.2	Cr	263 .82
$N_2 O_3$	16.6	Pb	82.103
$Na_2 O_3$	-	Zn	1115. 31
MgO	11.0	Cu	121.60
$Fe_2 O_3$	11.6	Cd	ND
BaO	-	Sulphates	16,32soit (2,5%)
Ti O_2	02.2	Chlorides	27 soit (0,06%)
SO_3	00.7		
$K_2 O$	01.3		
ZnO	-		
PbO	00.8		

The mineralogical composition (fig 4) shows that the CA ash is predominantly amorphous [43] composed of M (mullite) $Al_6Si_2O_{13}$, Q (Quartz) SiO_2, R (rutile) TiO_2, Ma (maghemite) Fe_3O_4, P (periclase) MgO, E (ettringite) Ca_6Al_{l2} (SO_4) (OH) and 12 A (anatase) TiO_2. These results corroborate several studies of silicoalumina ash [44-45], and give it a strong pozzolanic potential. According to the literature [46-47], the complex mineralogy of ash residue is the result of several processes that occur during the processing and incineration of flue gases, which may include vaporization, melting, crystallization, vitrification, condensation and precipitation, despite the complexity of the mineralogical composition of the residue, the literature indicates a comprehensive analysis, where the main mineralogical phases are identified.

The morphology of the residues provides valuable information on the leaching behavior and thus on their long-term behavior.The elements of Fly Ash (FA) are in the form of more or less hollow balls, the surfaces vitrified, shiny and smooth, composed of a wide range of sizes, with rough or smooth surfaces (Figure 2) due to the process of condensation to the decrease of the gas temperature. The morphology of similar residues can be found in several studies [48]. The residues are spherical particles that consist of polycrystalline and aluminosilicate platelets with a large specific surface area. This settling effect of spherical fly ash particles reduces its permeability.

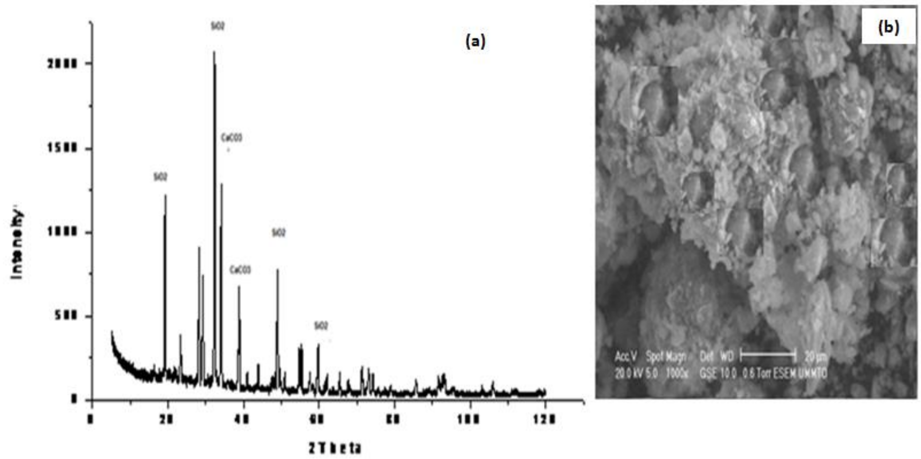

Figure 2 SEM image and XRD of used Fly Ash (FA)

2.3 Synthesis of the absorbent resin

The two partially cross-linked resins E1 and E2 are synthesized from two polymers P1 and P2 by esterification reaction in solution in the presence and absence of an HCl acid at a temperature of 100°C. Table 1 gives the operating conditions synthesis of the used resin.

Table 1 Operating conditions for the synthesis E1and E2

N° experience	P_1 (mol)	P_2 (mol)	Solvant	Catalyst type	Bath T°(°C)	Retention time (mn)
1	0.041	0.090	DMF	-	100	45

2	0.041	0.181	Benzene	HCl	100	65

DMF ;Dimethylformamide

a). Study of the evidence of the synthesis of the resin

The cross-linking of the two polymers in resin was highlighted qualitatively by tests of solubility of the two polymers and the products resulting from the esterification reaction.The study of the solubility of P1, P2, E1 and E2 was carried out with different solvents as shown in table 2, in order to highlight the formation of a new product in the characteristics are completely different from the initial product.

Table 2 Comparative study of the solubility of P1, P2 polymers and E1, E2resins.

Solvents	**P1**	**P2**	**E1**	**E2**
DMF	S	S	I	I
Méthanol	S	S	I	I
Dioxane	S	S	I	I
DMSO	S	S	I	I
Water	S	S	I	I
Distilled water +NaOH	S	S	I	I

S: Soluble;I: Insoluble and DMSO :Diméthyl-sulfoxyde

b). Calculation of the esterification rate of E1 and E2

The esterification rate shows the yield of the reaction which is all the more important as the reaction is carried out under the best operating conditions. The results are given in Table 3.

Table 3 Etherification rate of polymers

	E1	**E2**
Theoretical mass (g)	6 .16	6.4
Experimental mass (g)	3 .1	3.8
Yield (%)	50	59.37

c). Water absorption study of E1 and E2 resins

The study of absorption in water was carried out by gravimetric method according to the following protocol:A quantity of resin is introduced into a beaker, and then water is progressively added under magnetic stirring, until total absorption of the liquid. The addition of water is renewed until the resin is saturated (Table 4).

Table 4 Calculation of the absorption rate

Absorption rate	E1	E2
Water absorption rate / gram of resin (g)	90	24
Water absorption rate % by weight	9000	2400

2.4 Mixtures details and testing of studied Mortars

A normal mortar was prepared according to the standard (EN 196-1), using prismatic specimens 40 x 40 x 160 mm3 for the bending and compression tests. The specimens were prepared from Portland cement CEM I 52.5 R, CECN EN 196-1 standard sand and different percentages of CV (0, 10, 20, 40%) at 40 µm diameter, with different amounts of E1 resin (0, 0.01, 0.02, 0.03g). The mortar is dry mixed for 3 min. Water is then introduced during mixing, over a period of 30 seconds. The mixture is then mixed for 3 min. The different experiments are summarized in Tables 5 and 6.The mechanical tests were performed at the following times: 2 - 14 - 28 - 90 days.

Table 5 Composition of the specimens without resin

Fly ash (FA) replacement rate [%]	**0**	**10**	**20**	**40**
Ciment (g)	400	360	320	240
Sand S(g)	1350	1350	1350	1350
FA (g)	0	40	80	160
E/C	0,29	0,32	0,36	0,4
Water (ml)	116	128	144	160

Table 6 Composition of specimens with resin

Fly ash(FA) replacement rate [%]	**0**			**10**			**20**			**40**		
Cement (g)	400			360			320			240		
Sand(g)	1350			1350			1350			1350		
FA (g)	0			40			80			160		
Resin (g)	3	6	10	3	6	10	3	6	10	3	6	10
Water (ml)	116			128			144			160		
E/C	0,29			0,32			0,36			0,4		

3 Result and discussion

3.1 Study of the choice of the resin to be incorporated into the cement paste

Significant water absorption of about 9000% was obtained with the E1 resin, while the E2 resin has an absorption rate of 2400% (Table 2), in line with the resins on the market. The modification reaction was highlighted as a result of the reaction of PAA with PEG, which leads to the formation of interchain or intrachain bonds through ethylene glycol bridges. The development of these bonds would significantly affect the initial solubility of PAA. Indeed the bridging reactions, similar to cross-linking reactions, would limit the initial solubility of the flexible PAA chains, so that at the end the cross-linked network resulting from the esterification is insoluble in the common solvents of PAA. It should be noted that PAA and PEG are soluble in the various solvents whereas the E1 and E2 products resulting from their reactions under different operating conditions remain insoluble (Table 3).

Although the rate of esterification is more important during the synthesis of E2, we chose the E1 resin for incorporation into the cement paste, because its synthesis does not require the use of HCl as a catalyst while having an absorption rate of 9000% and this is very interesting from the environmental point of view.

3.2 Mechanical study of the cementitious material

a). Compressive strength of the cementitious material with the FA addition

The study of the strength of cement mortar without addition and with addition of FA at different percentages 10%, 20% and 40% (figure 3) shows that the strengths of all the specimens increase regularly with age and do not show any drop in strength. However, at young ages these strengths are always lower than those of the control specimen. This decrease in strength at young ages is due to the fact that the pozzolanic reaction is not predominant at young ages, which leads to a less intense hydration of the C_3S (tricalcium silicate) and C_2S (dicalcium silicate) ores at young ages, inducing low strengths. The latter are the two main ores that ensure the development of strengths in the short and medium term.

In the long term, the specimens with 40% FA substitution develop strengths comparable to those of control specimens and sometimes higher. This can be attributed to the slow pozzolanic activity of fixing the portlandite$Ca(OH)_2$ released by the hydration of portland cement to give rise to hydrated calcium silicates. Additional second generation C-S-H occupying a large space in the cement matrix and thus contributing to the development of strength, it occurs mainly after 28 days. J. BARON et al [49,50], have shown that this reaction is practically constant between 2 and 28 days, but after 28 days, pozzolanic activity increases, increasing mechanical strength and durability. This reaction continues its effect by forming additional C-S-H which improves the paste-aggregate interface, giving comparable and sometimes high strengths compared to the control specimen.

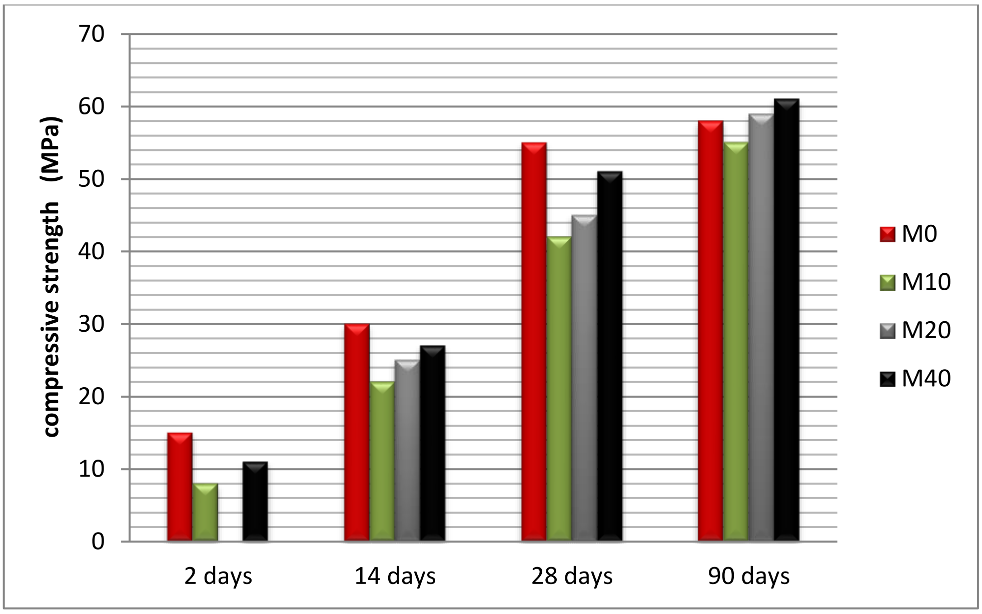

Figure 3 Development of compressive strength of mortar specimens containing different FA content (10, 20 and 40%)

c). Flexural strength of cementitious material with the FA addition

The results of the flexural strength are given in (figure 4) showing, as in the case of the compressive strength, a regular increase in the flexural strengths of all the specimens with age. The specimens containing FA develop their strengths more weakly than the control specimens but in the long term these strengths become more important and exceed the control specimens. The results obtained showed a remarkable improvement in the characteristics, especially the compressive and flexural strengths of cement pastes with different FA substitutions compared to cement mortar without addition.

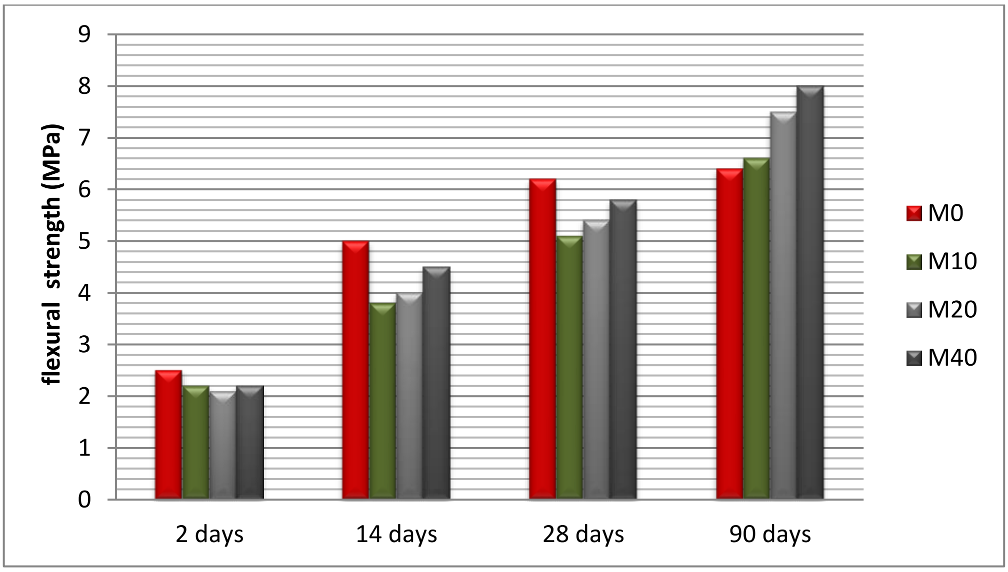

Figure 4 Development of flexural strength of mortar specimens containing different FA content (10, 20 and 40%)

d). Compressive strength of cement mortars modified with addition of E1 resin

A slight improvement of the cement mortar with E1 which is 3% compared to the mortar without any addition, on the other hand an improvement of 15% of the cement material with (40% of FA and E1) compared to the mortar without any addition (figure 3 andfigure 5). Thus, the addition of resin and FA increases the cementitious strength, which confirms several studies [51-52].

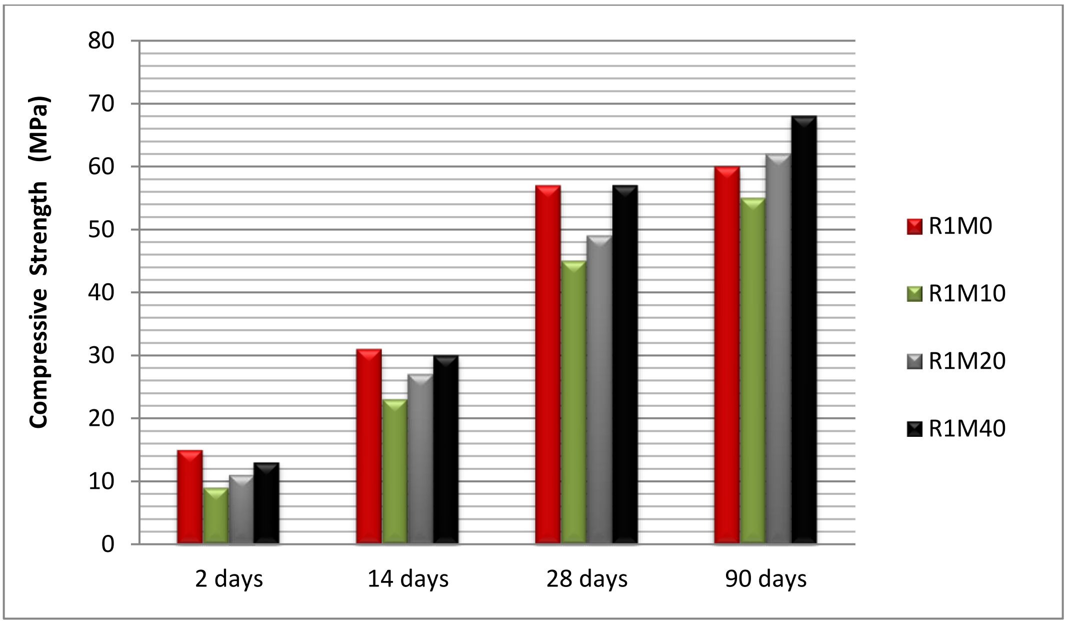

Figure 5 Incorporation effect of resin E1 on the compressive strength development of studied mortars

3.3 Flexural strength of cement mortars modified with addition of E1 resin

The flexural strength of the specimens with the addition of resin are given in figure 6 indicating an improvement in flexural strength of 16% in the cement mortar with E1 and 21% of the cement mortar with (FA to 40% and E1) compared to the cement mortar without addition.

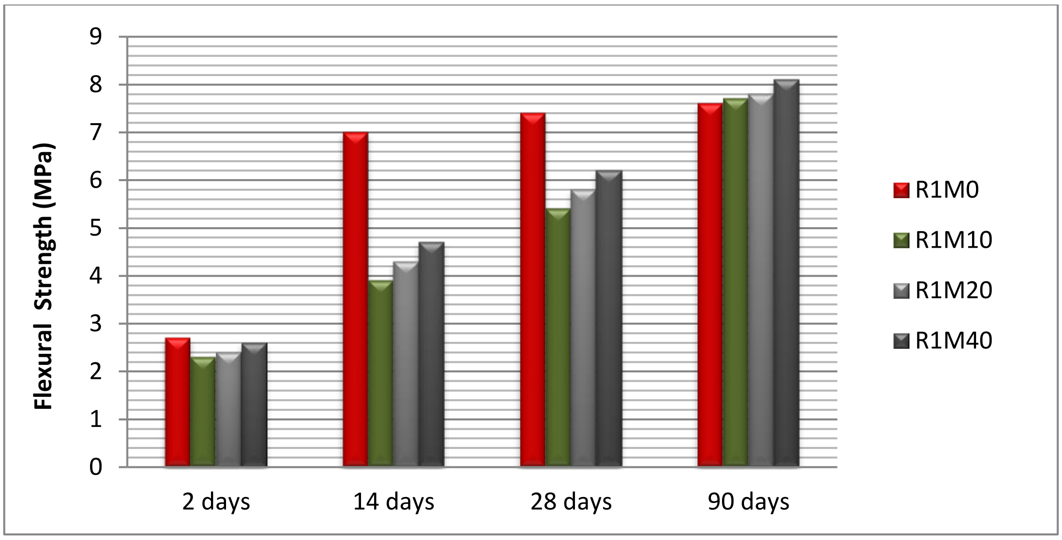

Figure 6 Incorporation effect of resin E1 on the compressive strength development of studied mortars

3.4 Hydration process in cement paste with resin

The mechanism of closure of the pores of the cement (figure 7) shows that the particle of resin which is inside the pores of the cement will be hydrated, following this hydration, this one will swell, this swelling will have consequently the closure of the pores and thus water will not be able to infiltrate in the cement paste causing modifications of the structure of the cement following chemical reactions inducing thus to its cracking.

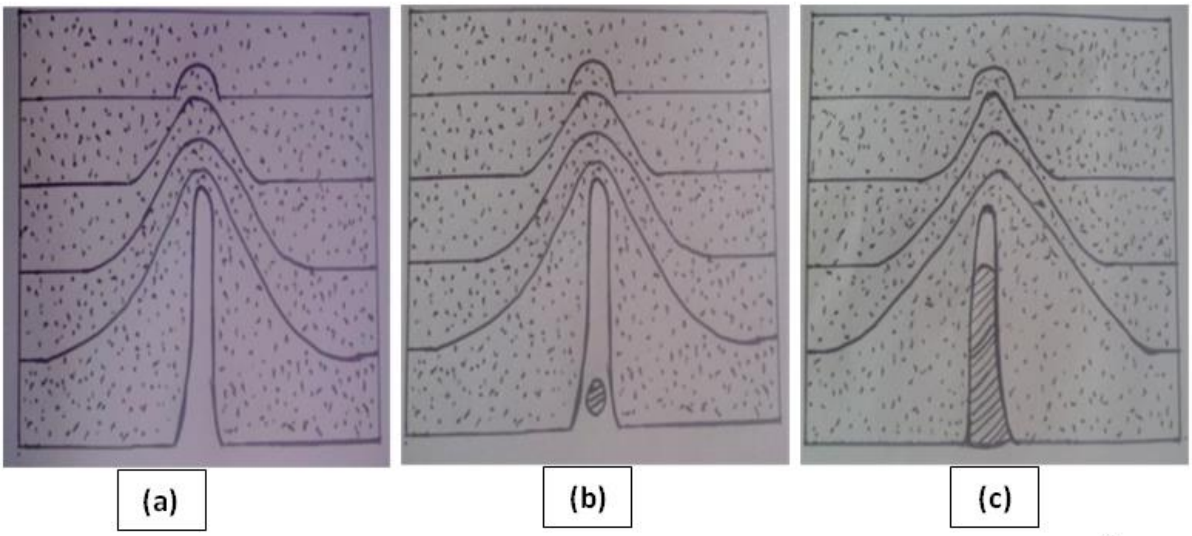

Figure 7 Pore closure mechanism; (a) Pore starter (b) Resin before hydration and (c) Resin after hydration

3.5 Influence of the addition of FA and E1 resin to cement

The cement paste without any addition (figure 8a) presents an important porosity. This is explained by the quantity of portlandite in the paste. The incorporation of E1 in the cement paste (figure 8b) and following the immersion of this last one in water, allowed the closing of the pores by the swelling of the resin, which proves to be interesting since it allowed the closing of the pores but that requires an important consumption of resin when the porosity is very important, while knowing that the resin has a cost in the civil engineering works.

In order to reduce this quantity of resin, it is judicious to use cement with addition of ash which will decrease the porosity of the pores before adding the resin.

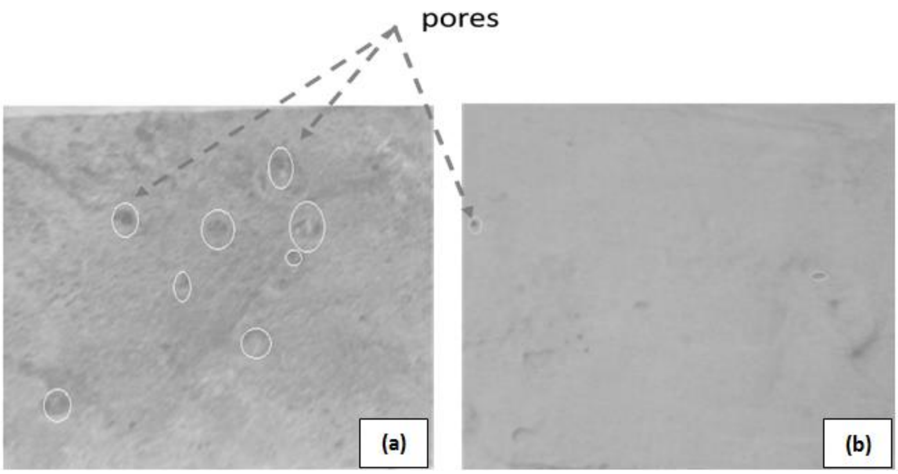

Figure 8 Cement mortar (a) without resin and (b) withresin

The addition of ash in the cement allowed the reduction of porosity compared to the cement without addition (figure 9a), as a result of the shape of the particles of ash that we could decrease this porosity, since the FA is characterized by a spherical shape that decreases the space between the grains of mixture cement - ash, consequently decreases the pores in the paste (cement - ash) as shown in (figure 9b).This decrease in pores allowed to minimize the addition of resin in the paste (figure 9c).

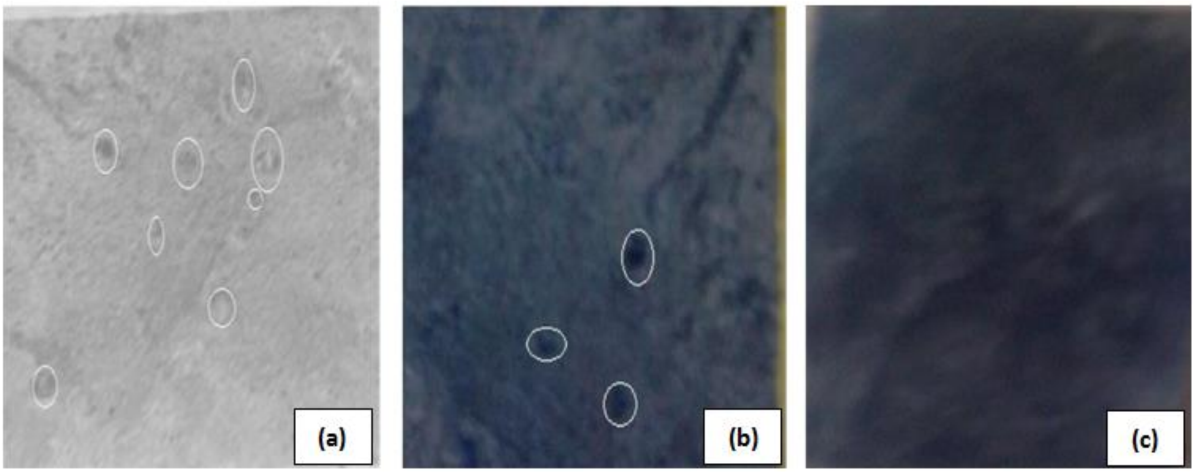

Figure9 Aspect surface of (a) cement mortar (b) Cement mortar with FA and (c) cement mortar with FA modifiedwith resin

3.6 Determination of the resin quantity necessary for pore closure

The quantity of resin that gave the best results concerning a more or less homogeneous distribution on the cement paste and a fairly good coverage of the cement surface is the 20% FA paste with 6g of resin, i.e. 1.875% in relation to the quantity of cement (figure 10a) and the 40% FA with 3g of resin, i.e. 1.25% in relation to the quantity of cement (figure 10b). On the other hand, for the cement paste with 10% ash (Figure 10c), it is necessary to put a larger quantity of resin than 10 g because for a smaller quantity, it is insufficient as shown in the figure.

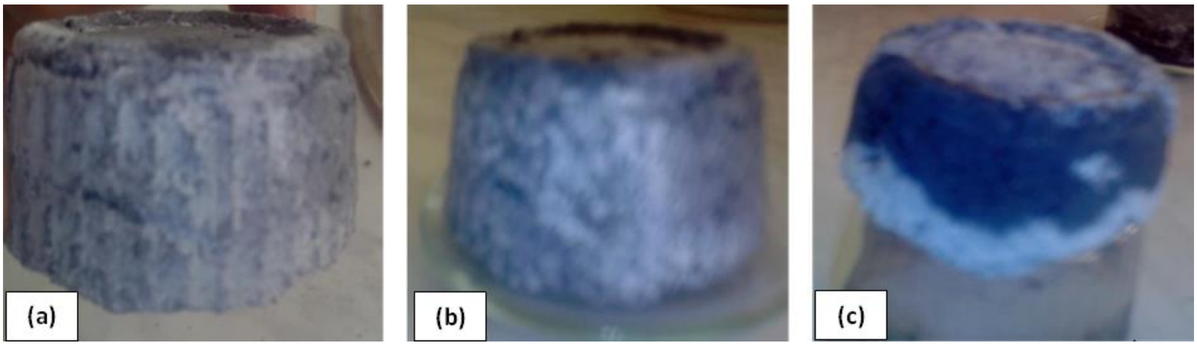

Fig. 15 mortar specimens at (a) 20% FA (b) 40% FA and (c) 10% FA

3.7 Effect of resin particle size

Fig. 18 shows the heterogeneity of the resin on the surface of the cement paste, this could probably be explained by the fact that the resin used with a particle size of 80 µ in diameter. Because of this, it would be interesting to study the effect of the particle size of the resin following its incorporation into the cement.Decreasing the particle size of the resin to 40µ in diameter (Figure 11b) led to homogeneity as well as a good distribution of the resin on the surface of the cement paste.

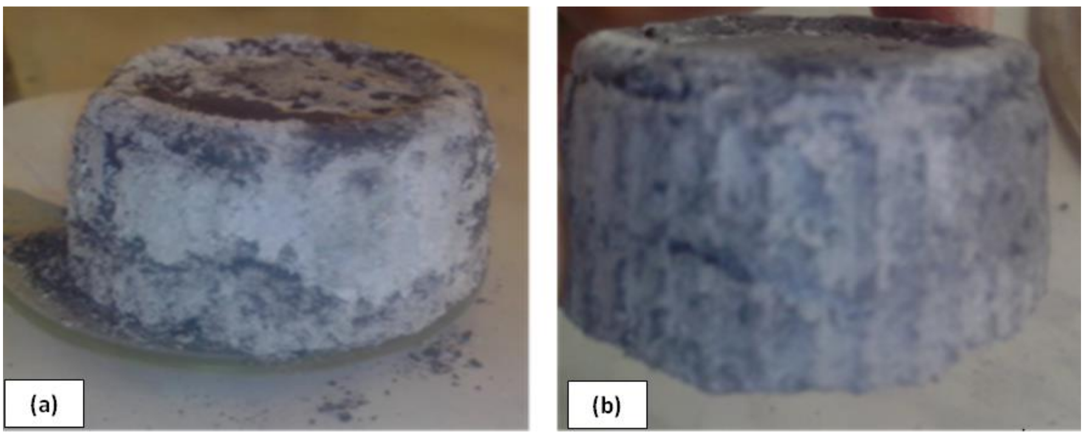

Figure 11Degradation of mortar specimens; (a) M40%FA with Resin E1 at 80µm (b) M40%FA with Resin E1 at 40µm

Figure 12 shows that the amount of resin at different diameters in the cement paste with addition at 40% FA decreases with decreasing grain size until a practically fixed value of 40 µm with an amount of resin of 1g or 0.41% with respect to the cement giving coverage and homogeneity over the whole cement paste. The amount of resin below 0.41% remains insufficient to cover the entire cement paste surface in a homogeneous way, which is why the grain size is fixed at 40µ.

This same curve shows that the amount of resin at different diameters in the cement paste with the addition of 20% FA decreases with the decrease of the granulometry until a fixed value of 40 µ with an amount of resin of 3 g or 0.94% with respect to the cement giving a coverage and homogeneity on the entire surface of the cement paste.

The granulometric study of the resin makes it possible to minimize the quantity incorporated into the cement since it is noticed that the smaller the diameter of the grain of the resin, the quantity of resin added to the cement will be lower, this is explained by the fact that the grain of the resin with small diameter will be dispersed in a homogeneous way on all the surface of the cement paste and during the hydration, there will be swelling of this last one on the level of the pores thus causing their closings. Therefore, the surface can be covered homogeneously for the two specimens of 40% and 20% FA with quantities of resin of 0.41% and 0.94% respectively in relation to the cement, but with a different particle size.

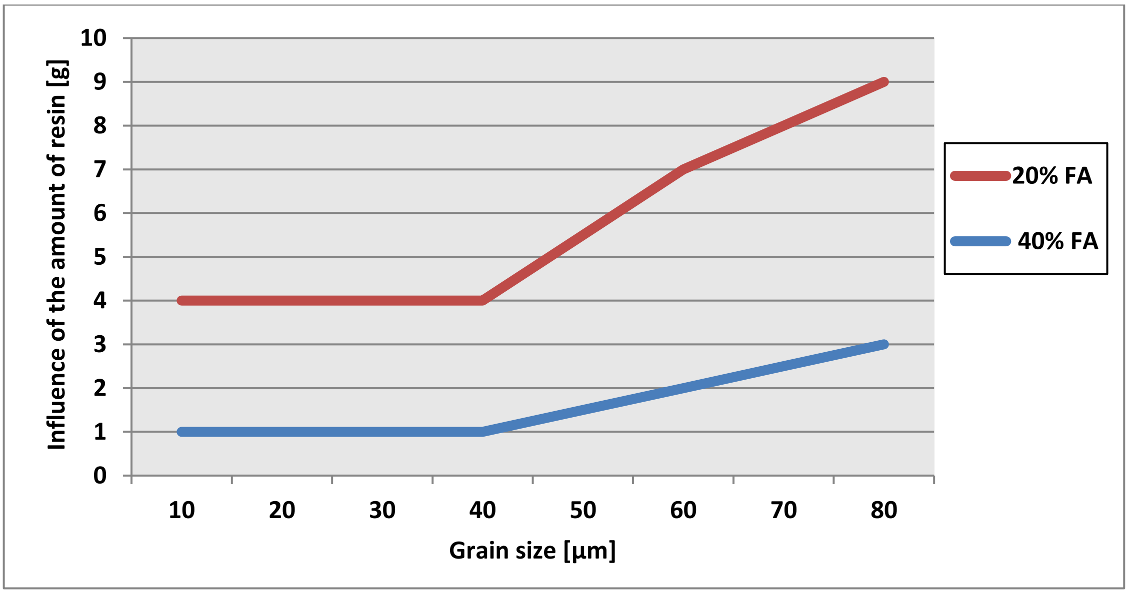

Figure 12 Influence of the amount of incorporated resin in cement mortar at 20% and 40% of FA as a function of grain size

4 Conclusion

The effect of FA fly ash and the synthesized superabsorbent E1 polymer on the self-repair capacity of cement was investigated. The results presented in this paper led to the following conclusions:

E1 resin was synthesized with an absorption rate of up to 9000%, mechanical compression and flexural tests indicated that fly ash (FA) cement from drug waste at different percentages continues to gain strength after 28 days, allowing it to achieve high levels of strength and prolongs its durability. The addition of FA and E1 significantly increases this compressive and flexural strength compared to the cementitious material without any addition, since an increase of 15% in compressive strength and 21% in flexural strength of the cementitious material with (40% FA and E1) is achieved compared to the cementitious material without any addition.

The homogeneity of the cementitious material was studied following the effect of the resin particle size since with a practically fixed value of (40 μ diameter and 0.41%) of E1 compared to the cement; good homogeneity of this cementitious material is obtained.

The incorporation of E1 in the cement swells inside the pores when water penetrates, thus preventing the cracking of the cementitious material ensuring its durability over time.The mixture of ash and resin in the cementitious material increases its durability over time and thus having a material that respects sustainable development.

5 References

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