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RESEARCH ARTICLE

Durability Properties of Five Years Aged Lightweight Concretes Containing Rubber Aggregates

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Abstract
Scrap tyres are one of the most important wastes. They can be used in different ways because of their availability and their non-degradable nature. This paper aims to demonstrate their reuse through durability properties experimental assessment of lightweight concretes aged five years, incorporating rubber aggregates as partial replacement of 5%, 7.5% and 10% of coarse/fine and coarse aggregates. The effect of the rubber aggregates on the lightweight concretes durability has been analysed. Firstly, the water absorption was evaluated, and then the mass losses were measured through many tests: freeze-thaw, elevated temperature and attack by Na_2SO_4 and HCl solutions. Wetting-drying cycles were carried out in order to accelerate the aging of the studied lightweight concretes and to reduce the tests duration. It has been observed that the water absorption decreased with small rubber content. The mass losses of the mixes were almost depending on rubber aggregates content and size, and the exposures duration.

Keywords
lightweight concrete, rubber aggregates, durability, mass loss, absorption, elevated temperature, freeze-thaw, sodium sulfate, hydrochloric acid

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1 Introduction

In the building materials field many researchers are interested on the sustainability subject. The environmental conditions, in which cementitious composites will be used, require that certain characteristics must keep their highest performances along time. The winter period or the chemically aggressive environment can cause disorders that may propagate to the totality of the structure. The traditional materials fail to accomplish this mission. So, the additions use or the raw materials replacement by those that can improve these failures is recommended. However, the complexity of the resulting materials properties from this combination has other consequences related to different factors.

In recent years, the world motivation for recycling is fairly clear due to the environmental problems resulting from the accumulation of solid waste, namely used tires, that can occupy during hundreds of years the spaces where they are abandoned or placed. So, they should be eliminated, retreaded or recovered. One of the most promising solutions for the future is the use of this waste in cement composites, being the most produced materials in the world. This can absorb a large amount of this durable material. Up to this date, this solution has been studied by several researchers who are interested in the optimization of the incorporation of this material in order to preserve or improve the cement composites durability.

The tests made to study the properties of rubberized cement composites and the recommendations made on them are different from one study to another. It has been demonstrated that the concretes strength at elevated temperatures is affected by the rubber aggregates incorporation. Guo et al. [1]have studied concretes obtained by replacing 4%, 8%, 14% and 16% of natural sand with crumb rubber (0.85 to 1.4mm) by volume. They observed a reduction in the compressive strength and Young's modulus of concrete with the increase in the rubber content after their exposure to fire at 200, 400 and 600 °C. The addition of a rubber aggregates appropriate amount in concretes showed to be efficacious in reducing micro-cracks due to elevated temperature [2]. Gupta et al. [3] observed that the exposure of rubberized concretes, with content up to 10% of rubber fiber replacing natural sand, to elevated temperature up to 150°C has little effect on their residual properties. It was also observed that at temperatures beyond 150°C and rubber fiber replacement higher than 5%, the decomposition of rubber fiber leads to rapid decline in the rubberized concrete residual properties.

The studies on the assessment of rubberized mixtures freeze-thaw resistance are differently conclusive. Pedro et al. [4] have studied rubberized mortars containing 15% by volume, of rubber aggregates (0 to 2mm) replacing natural sand. These mortars were tested under accelerated aging at 112 days. They found that rubberized mortars are less sensitive to freeze-thaw than control mortars.

The rubber aggregates size and content have always been directly related to all rubberized concretes performance and durability indicators. Crumb rubber has been recognized for its role in reducing the effects of freeze-thaw cycles compared with coarse rubber aggregates [5]. Salamah [6] found that an increase in the rubber content increases the resistance of concrete to freeze-thaw. He has also observed that the elasticity modulus was greater than 60% and all samples overcome the maximum number of cycles (300 cycles).

The results of the studies on the durability of rubberized concretes in chemically aggressive environments have encouraged the use of rubber at various ways and shapes. Azevedo et al. [7] studied the attack of sulfuric acid on concretes incorporating 0%, 5%, 10% and 15%, by weight, of rubber in partial replacement of natural sand. At 56 days, after exposure to this acid for 28 days, they showed that the mass loss increase with the increase of rubber content. Ganesan et al. [8] evaluated the mass loss of concretes containing 15% of crumb rubber (sizes < 4.75mm) exposed to sulfuric acid (H_2SO_4) and sea water for 90 days. They reported that mass loss was less than that of control concrete.

According to several studies, many contradictions have been highlighted regarding the effects of rubber on abrasion resistance [2]. Thomas et al. [9] have studied three factors to test the abrasion resistance of concretes containing rubber as partial replacement of natural sand. The rubberized concretes has improved abrasion resistance compared with the control concrete for W/C of 0.4 and 0.5. Whereas for W/C of 0.45 and 7.5% of rubber aggregates, the abrasion resistance decreased and it was better with a rubber content greater than 5%. Grdic et al. [10] have demonstrated an increase in the abrasion resistance of concrete containing 10% of crumb rubber (0.5 to 4mm) replacing natural sand. According to their results, rubber content should be limited to 20%, since a higher level caused a abrasion resistance reduction of about 30%.

Carbonation is one of the most important durability factors. Generally, the investigations made about this phenomenon focus on aging tests. In the case of rubberized mixtures, little research has been done for studying this phenomenon. According to the available study, it can be concluded that the addition of rubber in the matrix decreased its carbonation resistance [2]. Bravo et al. [11] studied concretes containing rubber crumb partially replacing natural sand by 5%, 10% and 15% of rubber aggregates. They observed a small increase in carbonation depth with the increase of rubber content. They explained this by the quality of the zone between rubber crumb and cement paste.

Few researchers have studied the resistance of rubberized concretes to corrosion. This is, mainly, due to the fact that these mixtures are elaborated to be intended in almost all of these studies for non-structural applications. Among these researchers, Karahan et al. [12] studied concretes containing 10%, by volume, of crumb rubber (size 0.15 to 4.75mm) as a replacement of natural sand. They have concluded that the state of the reinforcement bars used in both cases were almost the same. Other researchers have reported that the concrete with 5% of natural sand replacement of by rubber aggregates (size 0.3 or 0.6mm) improve the corrosion resistance [13].

The phenomenon of chloride transport in rubberized concretes has also been little studied. Nurazuwa et al. [14] added to the concrete in their research 10% silica fume as cement replacement , by weight, and 0%, 10%, 15% and 20% of crumb rubber as sand replacement. The characteristics of chloride transport have been improved with the increase of the rubber content in concrete with W/C of 0.35. This resistance was 50% higher than that obtained in a concrete with a W/C of 0.50. Gesoglu et al. [15] used crumb rubber (sizes 2 to 4mm) to replace 10% to 20% of natural aggregates in concrete. The permeability coefficient was reduced by 43.75% and 67.46%, respectively, for rubber aggregates of 4mm and by 40.73% and 43.1%, respectively, when the crumb rubber of 2mm was included.

It can be seen from the available researches that the aged rubberized mixes were not studied. In this paper while we refer to our earlier work [16], we tried to explore the possibility of evaluating the durability of rubberized lightweight concretes through a simple indicator which is the mass loss. These lightweight concretes aged of five years incorporate rubber aggregates as coarse/fine and coarse aggregates partial replacement of 5%, 7.5% and 10%. Firstly, water absorption was evaluated, and then the mass losses were measured through: freeze-thaw, elevated temperature and Na_2SO_4 and HCl immersion solutions (5%) tests. Wetting-drying cycles were carried out in order to accelerate the aging of the lightweight concretes studied and to reduce the duration of the tests.

2 Materials and methods

2.1 Experimental program

In this work, we carried out several experiments. The lightweight concretes were manufactured to be used after five years for their durability properties evaluation by measuring the mass losses and examination of the degradations after each exposure. We started by water absorption, then the specimens were tested to: freeze-thaw, elevated temperature, sodium sulfate and hydrochloric acids attacks. The six compositions were compared to a control mix having ordinary composition (without replacement).

2.2 Materials

The materials used in this work were conditioned in the laboratory. Figure 1 shows the grading curves of these materials.

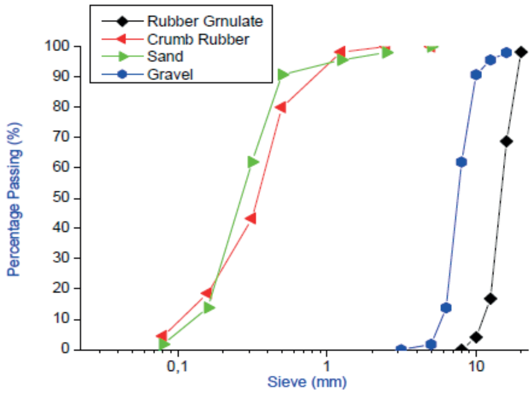

Fig. 1 Grading curves [16]

- Ordinary Portland cement was used for all the mixes, having mechanical performances and physicochemical characteristics according to NA 442, EN 197-1 and NF P 15-301/94, the compressive strength at 28 days is 42.52 MPa. The constituents of this cement are shown in Table 1.

Table 1 Chemical composition of cement

Constituents	C_3S	C_2S	C_3S	C_4AF
(%)	58 to 64	12 to 18	6 to 8	10 to 12

- Natural sand was used as fine aggregate, having granular class of 0/5 and density of about 1600 kg/m^3.
- Gravels were used as coarse aggregates, having continuous grain size distribution and a density of about 2700 kg/m^3.
- Rubber aggregates used as coarse aggregates partial replacement are of 10 to 40 mm, having a density of 1.2. These aggregates were obtained by manual grinding.
- Shredding crumb rubber was obtained by manual grinding and was used as fine aggregates partial replacement having size ranging between 1 and 3mm and a density of 1.3.
- Potable water was used for this study.
- Distilled water was used to prepare solutions (5% sodium sulfate and 5% hydrochloric acid).

2.3 Mix proportions

In this research, water/cement ratio was 0.5. Firstly, three lightweight concretes were prepared with incorporation of 5%, 7.5% and 10%, by weight, of gravels replaced by rubber aggregates, then three others were prepared with incorporation of a combination of rubber aggregates and crumb rubber replacing gravels and sand, respectively with the same replacement levels. The mix proportions are listed in Table 2.

Table 2 Mixture design of 1 m^3 of lightweight concrete [16]

Concrete	Materials (kg/m^3)				
	Cement	Rubber aggregates	Crumb rubber	Sand	Gravel
Cref	380	0	0	858	927
*CRg*5	380	46.4	0	858	884
*CRg*7.5	380	69.5	0	858	851
*CRg*10	380	93	0	858	839
*CRm*5	380	46.4	42.9	815	839
*CRm*7.5	380	69.5	64.35	793.65	851
*CRm*10	380	93	85.8	772.2	884

The letter *C* indicates Concrete, *Cref* indicates referential concrete (no replacement). *R* indicates granules rubber replacing a gravel fraction of aggregate. *Rm* indicates granules rubber and crumb rubber replacing a gravel and sand fraction of aggregate while *n* indicates ratio of substitution [16]. For example, *CRm*7.5 is lightweight concrete incorporating 7.5% of granules rubber and crumb rubber in mixed replacement by weight of gravel and sand.

2.4 Specimens

For each exposure seven (7) specimens of lightweight concretes were manufactured and cured in 2011. These specimens have a prismatic shape (50 × 100 × 100mm) maintained in the laboratory conditions for five years.

2.5 Testing methods

2.5.1 Water absorption

Water absorption was evaluated following ASTM C642 [17], where the immersion temperature was ranging between 20 to 25°C. The specimens were dried until constant weight at a temperature of 105 ± 5°C, for at least 24h, then they are cooled and weighed (control weight). These specimens were immersed in water for 48h, then wiped and weighed again. In using control weight and final weight, the water absorption was evaluated in percentage.

2.5.2 Freeze-thaw test

This test was performed according to ASTM C 666 [18]. 100 freeze-thaw cycles of 24 hours are produced, following procedure B, consisting on carrying out the freeze at –19°C for 12 hours and thawing in water for 12 hours too.

2.5.3 Elevated temperature

Fire resistance was evaluated according ASTM 2032-09 [19]. The specimens were exposed to heating-cooling cycles at different elevated temperatures, 200°C, 400°C, 600°C, 800°C and 1000°C, with a heating rate of 200°C/24 hours and a stabilizing bearing of one hour (1h). An electrical furnace (M110 muffle furnace) was used to carry out heating. The specimens were cooled in the laboratory in natural condition.

2.5.4 Sodium sulfate and hydrochloric immersion tests

The effects of sodium sulfate Na_2SO_4 and hydrochloric acid HCl solutions on lightweight concrete were evaluated performing ASTM C 267-01 [20]. The solutions were prepared with 5% of each substance. To evaluate the acid attack, three specimens of each mix were immersed in each one during 90 days and three others were immersed in potable water as control environment. In order to control the evolution of the *pH* of the solutions containing the different mixtures, one specimen of each one were placed in these solutions separately. This evolution was controlled using paper *pH* indicator. The specimens were first placed in the solutions for 6h for a complete saturation. The drying lasted 18h for each cycle for Na_2SO_4 immersion. In the case of hydrochloric immersion, the drying lasted 20h. The drying temperature was 60°C in both cases.

3 Experimental results and discussion

3.1 Water absorption

The role of water in the process of deterioration of concrete has been studied incessantly. Therefore, the water absorption properties provide the best information about the durability of cementitious composite. In the Figure 2, the curves show the percentage of water absorption of the different studied lightweight concretes. We may see that the rubberized lightweight concretes of the composition (*CRg*) have recorded the highest percentages of water absorption.

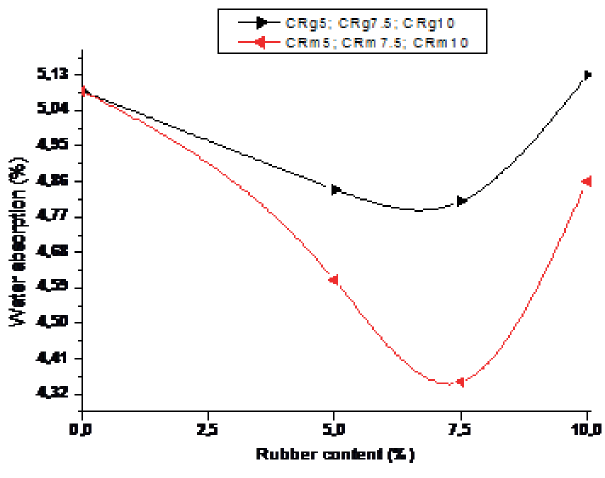

Fig. 2 Evolution of water absorption of lightweight concretes

According to these curves, the biggest percentage of water absorption of about 5.13% and 4.86% were reached when 10% of rubber aggregates were used in both cases of modified mixes *CRg* and *CRm*, respectively, whereas the percentage of water absorption of 5.09% was reached in the case of *Cref.*

The curves also revealed that the smallest percentage of water absorption of 4.81% and 4.35% were obtained by the specimens when 7.5% of rubber aggregates was used in the case of *CRg* and *CRm*, respectively.

At the replacement level of 5 to 7.5%, the water absorption decreased, whereas rubber content higher than 7.5% increased percentage of water absorption. Based on this results many explanations can be given. The light weight concretes water absorption of this study was governed by the heterogeneity of the hardened rubber mixes (random positioning of rubber aggregates) and by the air content introduced into these lightweight concretes as well. This is caused by the nature of these aggregates, trapping this air by their rough surfaces and making them less permeable [21]. The 7.5% addition of rubber aggregates has reduced the percentage of water absorption and has limited the spread of water in the studied mixes which were less sensitive to the water.

According to these results, 7.5% can be considered as substitution threshold. Furthermore, this result is related to those found in the research of Medine et al. [16] who have studied these mixes at their fresh states, whose compatibility test showed that *CRm*7.5had a higher degree of compatibility than *CRm*5 and *CRm*10, therefore a better compactness This shows that the water absorption depends on the compactness. The absorption coefficient of rubber aggregates is negligible [22], indeed the porosity affected by these aggregates is responsible for the increase in water absorption. This was also indicated in the research of Onuaguluchi [23]. The increase of the water absorption percentage was due to improper compaction of rubberized concretes owing to the lower density of rubber [3].

The water absorption of *BCm10* and *BCg10* is almost similar to that of *Cref.* Many researchers have also observed that water penetration in rubber mixes is higher [24] when the replacement level is higher than 12.5% [25] or higher than15% [26]. From this test we may conclude that the percentage of water absorption decreased with smaller rubber content. Andressa et al. [26] have showed that porosity and absorption increased with increasing rubber content [27].

The use of rubber aggregates with a variety of sizes has reduced water absorption (the case of *CRm* composition). Several investigations have revealed that the relationship between the increase in water absorption and the size of these rubber aggregates has been demonstrated. Su et al. [28] have observed that this kind of substitution makes the cementitious composites more compact. The fine rubber aggregates fill the pores produced by the coarse aggregates and the permeability is reduced.

Even if there exist no results corresponding to this age of mixes (5 years), it is possible to affirm that the relationship between water absorption and the addition of rubber depends on the content, size and distribution of this material in the matrix as well as age.

3.2 Freeze-thaw test

Freeze-thaw resistance is an important property of concrete that influences the durability of concrete products and structures [29]. At the beginning of the test, the results shown on figure 3 indicate that the specimen weights of *Cref*, *CRm5* and

CRm7.5 increased up to the 20th freeze- thaw cycles. The gain in mass was ranging between 0.12 and 0.35%. These results confirmed the relationship between freeze-thaw and absorption. Figure 4 illustrates that *Cref* has lost most of its mass compared to the rubberized lightweight concretes. The composition (*CRm*), in particular *CRm10*, was the least affected by this exposure, after 50 freeze-thaw cycles the mass loss of this mix reached 1.78% (Fig.5).

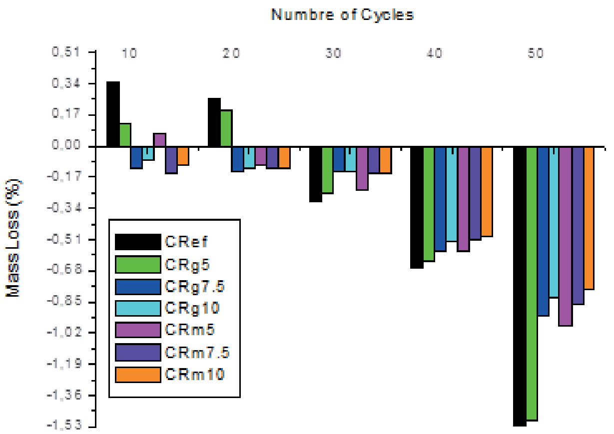

Fig. 3 Mass losses of lightweight concretes due to freeze-thaw up to the 50th cycle

Whereas the highest mass losses were corresponding to *CRm5* and *CRg5*; they reached 2.61% and 2.67% respectively. After 100 freeze-thaw cycles the mass loss in the specimen of *Cref* reach 3.1%

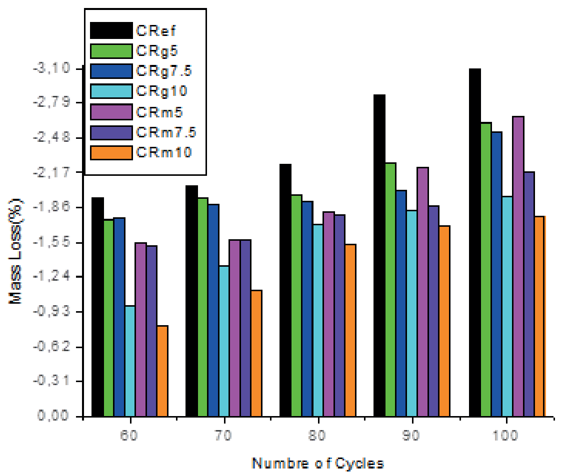

Fig. 4 Mass losses of lightweight concretes due to freeze-thaw up to the 100th cycle

We can conclude that the smallest mass losses were recorded in the case of lightweight containing the biggest amount of rubber aggregates [30]. During this test it was observed that the increase of mass losses was related to the increase of number of freeze-thaw cycles and to the decrease of the size of rubber aggregates. The lightweight concretes containing crumb rubber lost less of their weights. Several researchers, namely, Zhu et al. [5] have proved this in their researches.

Visually (Fig. 5), it has been found that the facets of *Cref* have become rough with appearance of two remarkable forms of pores. The first one existed before the exposure of the specimens to freeze-thaw, their volumes increased because of expansion of the cement paste due to the pressures exerted inside these pores. The second form of pores is that of the voids made by the detachment of the mortar parts and of loosening rubber aggregates from the surface due to the loss of their coating as a result of their expansion. The appearance of micro-cracks at the 100th cycle was also observed.

Fig. 5 Specimens of lightweight concretes after 100 freeze-thaw cycles

These results mean that the rubberized lightweight concretes have resisted to freeze-thaw cycles. The quality of the interface paste/rubber aggregates have reduced the pressures exerted by the expansion due to the freeze-thaw, this explanation was also reported in several research [31]. The rubber aggregates absorbed expansion energy and prevented rupture [32]; they proceeded as an air trainer alleviating hydrostatic pressure. Other researchers have related this to the nature of rubber aggregates and their volume unchangeable during freeze-thaw, reducing pressures and damages in this zone [33].

It should be noted that there is a slight difference between the results obtained in the two compositions (*CRm* and *CRg*). Therefore the choice between them depends on the use of the lightweight concrete and the mechanical strengths required for this use.

3.3 Elevated temperature

Before using any material in the construction, it is necessary to study its resistance to the high temperature and the resulting deterioration processes. The mass changes of rubber aggregates subjected to the elevated temperature were analyzed at different temperatures (Fig.6).

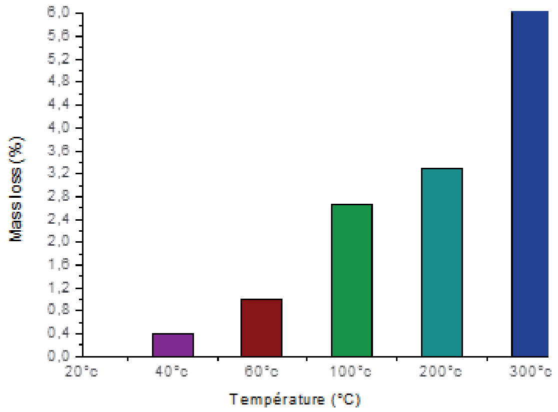

Fig. 6 Evolution of mass loss of rubber aggregates due to elevated temperature

It was observed that the mass losses were almost negligible with a maximum mass loss of about 6.04%. At 100°C the samples became sticky to hands releasing smell of burnt rubber. At 300°C rubber aggregates consistency changed, then inflamed and became oily. Several authors have indicated that the decomposition of rubber aggregates starts rapidly at a temperature of 300°C [34–36]. The same result was demonstrated by thermo gravimetric analysis in the study of Gupta et al. [3]. They showed a rapid decrease in the weight above 300°C and 70% of weight loss was observed when temperature reached 800°C.

Figure 7 illustrates the mass loss variations of rubberized lightweight concretes as a function of temperature. At 200°C, we can observe that rubberized lightweight concretes have lost between 1 and 3% of their masses, whereas *Cref* has lost 1.7%. These small mass losses are due to the beginning of the water evaporation. This occurs at a temperature of 65 to 80°C [37]. At 170°C, the rubber starts to melt, consequently the interstitial pressure induced by the water vapour is reduced and the appearance of cracks is delayed. At this stage of heating the odour of rubber has been felt.

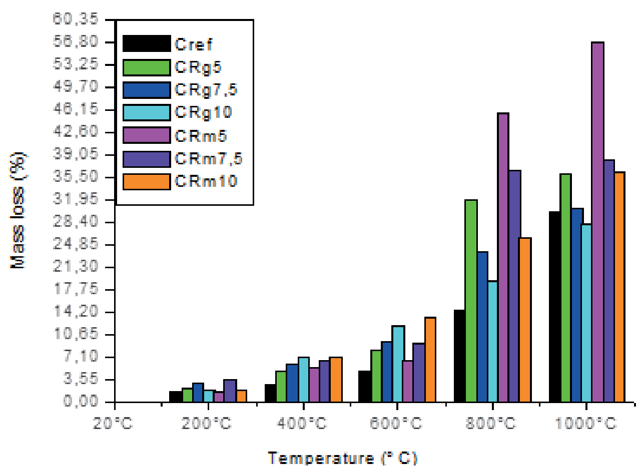

Fig. 7 Evolution of mass losses of lightweight concretes due to elevated temperature

At 400°C, the mass losses increased, these were of about 7.1% for *CRg10* and *CRm10*, whereas it was of about 2.6% for *Cref.* The cracks have appeared due to the accumulation of internal pressures, particularly in the case of the mixes containing a combination of rubber aggregates and rubber crumb (*CRm*). Thus, the differential expansion of different constituents of the studied specimens may explain this. Guelmine et al. [36] studied the performance of recycled crumb rubber mortar exposed to elevated temperature. They indicated that a temperature of 400°C has a strong effect on this mortar.

At 600°C, the smallest mass loss was attributed to *CRm5*. The specimens *CRg10* and *CRm10* showed the highest mass loss of about 11.96% and 13.34% respectively, whereas *Cref* has lost 4.8% of its mass. Some rubberized concretes specimens took on the yellowish and reddish colors, the macro-cracks began to be significant, especially in the case of *Cref.* According to Ismail et al. [38] the structures of lightweight concretes modify at temperature above 500°C. The reason of mass loss is the decomposition of constituents of hydration product. Rubber aggregates coating has begun to disappear and more voids were created. The same observation in the study of Gupta et al. [3] who have investigated the properties of rubber fiber concrete exposed to elevated temperature reaching 800°C. They observed that the decomposition of rubber fiber leads to the formation of voids. Therefore, they recommended that the rubber fiber content in concrete should be less than 5% to maintain the stability of their residual compressive strength.

At 800°C, in the case of both compositions, mass losses increased with the decrease in the content of rubber aggregates. For *CRm5* and *CRm10* the mass loss was ranging between 45.59 and 25.85%, respectively, and between 32% and 19.08% for *CRg5* and *CRg10*, respectively. At this temperature, *Cref* has lost 14.28% but its state of cracking was very advanced (Fig.8).

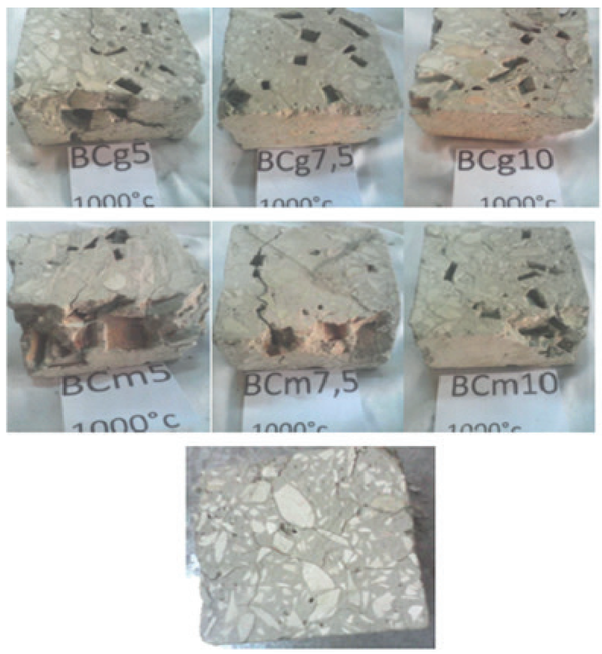

Fig. 8 Effect of elevated temperature on lightweight concretes at 1000°C

At 1000°C the mass losses became considerable. In the case of the composition (*CRg*), the mass losses were similar to those recorded by *Cref* (30%). However, the biggest mass loss in the case of the second composition (*CRm*) was of 56.82% corresponding to *CRm5*. The increase of mass losses was related to the increase of temperature heating

All results were conditioned by the increase of the heating temperature, the size of rubber aggregates, the rubber content and the location of rubber aggregates relatively to the surfaces. Although rubber aggregates have prevented the volumetric expansion leading to early deterioration, the rubberized lightweight concretes are not recommended for applications at temperatures above 600°C because of the flammability of the rubber.

3.4 Attack by acid of sodium sulfate

In this experimental research, the effect of the immersion in the sodium sulfate solution of the lightweight concretes has been also tested. It can be seen from the figure 9 that up to the 16th cycle, all the specimens tested with sodium sulfate have recorded gain in mass, in particularly in the case of the mixes of the composition (*CRg*). Generally, this gain in mass is attributed to the absorption of the solution and to the formation of gypsum and ettringite, following the reaction of the sulfate with the hydrated calcium aluminates to form calcium sulfo-aluminates and the free calcium hydroxides in the cement to form calcium sulfate. From the test results, we may see that the first mass loss has been observed at the 24th cycle of the immersion test.

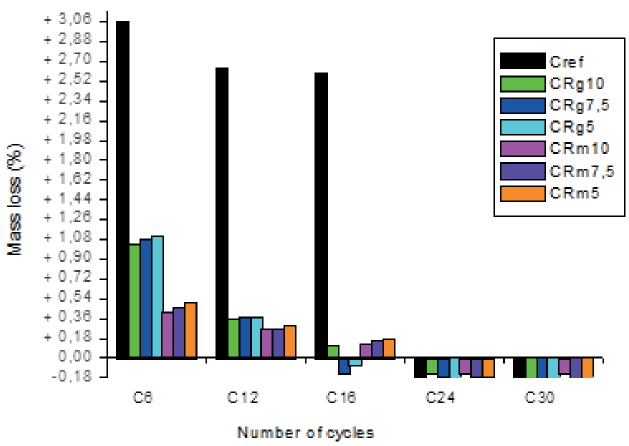

Fig. 9 Mass losses of lightweight concretes up to the 30th cycles due to Na_2SO_4

The mass loss increased slightly with the increase of the immersion duration. The same finding was reported by Yung et al. [13]. At the 30th cycle a mass loss of 0.25% was observed in the control mix (*Cref*). We may also see that the mass loss for any replacement level does not exceed 0.23%. The results shown on the figure 10revealed that the biggest mass losses were observed on the specimens containing 5% of rubber aggregates (*CRg5* and *CRm5*). These results depend most of all on the rate absorption of this mix in the beginning of this test (up to the 16th immersion cycle). Contrariwise,Yunget al. [13] have studied concretes containing rubber aggregates, and have indicated that composites incorporating 5% of rubber aggregates have exhibited the best resistances.

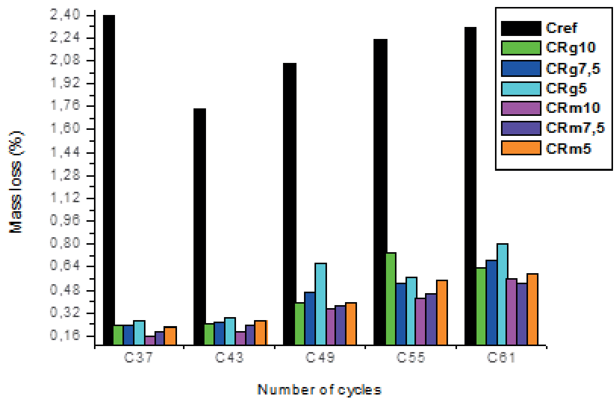

Fig. 10 Mass losses of lightweight concretes up to the 60th cycles due to Na_2SO_4

The figure 10 shows that at the 37th cycle, *Cref* has lost 1.4% of its initial mass. In the other cases of lightweight concretes, the biggest mass loss of 0.26% was also observed on *CRg5* and *CRm5*. The smallest mass loss of about 0.16% corresponded to *CRm10*.

The figure 11 illustrates that the mass loss increased with smaller rubber content. After 90 days of immersion *Cref* was more degraded compared with the lightweight concretes. The mass loss of *Cref* is about 2.62% and it did not exceed 1.97% in the case of the modified mixes.

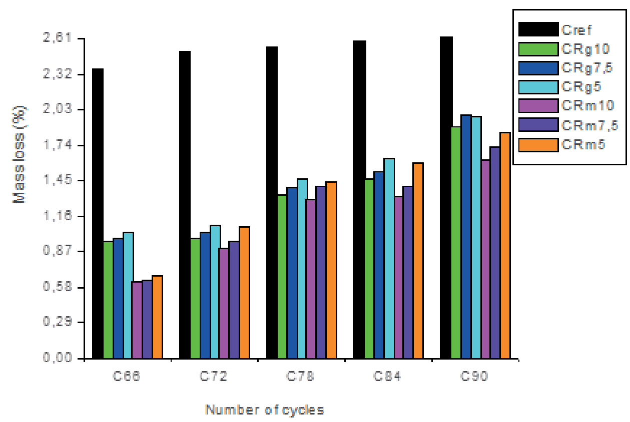

Fig. 11 Mass losses of lightweight concretes up to the 90th cycles due to Na_2SO_4

As shown in figure 12, the surfaces containing less rubber aggregates were significantly influenced by Na_2SO_4 solution. They were rough and covered with slightly white and yellow stains, due to the salts accumulation. The surfaces containing more rubber aggregates were less affected by this solution. They were mended by the presence of these aggregates and the formation of important cracks was prevented.

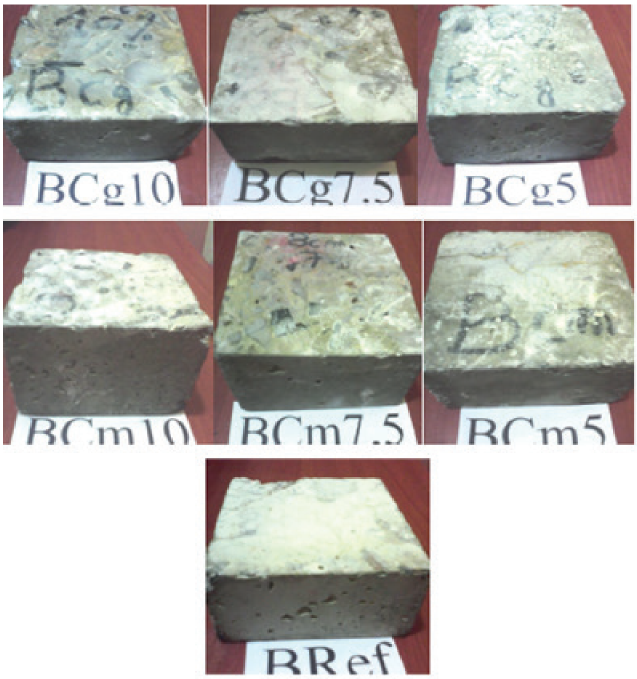

Fig. 12 Effect of (Na_2SO_4) on lightweight concretes at 90 cycles

A similar observation was mentioned by Thomas et al. [25] and Blessen et al. [39] in their research. The visual examination of the tested lightweight concretes leads also to conclude that this solution has no effect on the dimensions of the specimens during all immersion duration.

The cracks were attributed to the reaction between portlandite (*C–H*) after hydration of the cement with the sulfate to form gypsum and ettringite. In the case of this study the wetting-drying cycles have accelerated the mixes aging. During the drying phase, the sulfate concentration increases in surface area, and the crystallization of sodium salts causes damage such as chipping of the surfaces.

In this test, *Cref* was less resistant to Na_2SO_4 solution, whereas the modified concretes have resisted to this attack, *CRm10* in particular. This resistance increased with the increase of rubber content. The results of Jinhua et al. [40] have also showed the same. They indicated that a replacement level of 10% of rubber aggregates improve the resistance of the concrete to Na_2SO_4 solution.

According to several authors, namely Kumar and Brown [41,42], a constant *pH* is considered as one of the desirable criteria for this type of test because it allows to simulate the exposure conditions to be studied, such as sea water. In the case of this research, the tests were done at a variable *pH* to reduce the test duration and simulate the severity of the aggressive medium.

Before immersion the *pH* of Na_2SO_4 was between 5 and 6, the results demonstrate that it was between 6 and 7 three hours after. At the end of the immersion period (90 days), it reached 9 to 10 for the solution containing *Cref*, *CRg10*, *CRm10* and *CRm7.5*, and 8 to 9 for solutions containing *CRg7.5*, *CRg5* and *CRm5*. The pH change reflects the solubility difference of the various elements. This influences the stability of the cementitious matrix [43]. The aggressive agent (SO_4^{-2}) has been partially and progressively neutralized by the cementitious specimens. The alkalinity of the immersion solutions increased over time. This is due to the destabilization of the chemical equilibrium of the hydrates under the effect of the dissolution of the portlandite. Consequently the aggressiveness of the solution decreases.

The mass losses observed in this test were produced during the 60 days, when the *pH* was < 7, and were reduced as soon as the *pH* reached the value 9. Fettuhi et al. [44] have also concluded that the deterioration of cementitious composites increases when the *pH* of the solution is less than 6.5.

The water with *pH* = 7 containing the test specimens gradually reached *pH* = 12 after three months of immersion. Specimens remain healthy (a small amount of altered mortar was recovered from the bottom of the vat).

The rubberized lightweight concretes have shown increased resistance to Na_2SO_4 solution, due to the particular characteristics of the rubber aggregates. Whereas the mass loss of the *Cref* was more significant compared to them. In conclusion, the best way to introduce rubber aggregates into lightweight concrete for better performance in this medium is to combine the multi-sized rubber granulates with replacement levels ranging between 7.5 and 10% to limit the solution absorption. Finally, these results are not conclusive, because of the diversity in methods, how rubber mixes are made even at identical replacement levels.

3.5 Attack by hydrochloric acid HCl

The results of mass loss of the lightweight concretes after immersion in acid solution (*HCl*) during 90 days are shown in figures 13, 14 and 15. The mass loss recorded in the case of the control mix was ranging between 6.19% and 34.17%.It has increased over time and specially after each renewal of the solution. *CRm10* has recorded the biggest mass losses, ranging from 7.83% in the 1st day to 31.93% in the 90th day. The smallest masses loss between 7.31% and 6.62% on the 1st day and between 30.20% and 29.36% in the 90th day were recorded in the case of *CRg7.5* and *CRg10*, respectively. While the mixes *CRg5*, *CRm5* and *CRm7.5* have almost lost between 31.36% and 32.73% in the 90th day. At the end of the test, it was concluded that the composition (*CRg*), particularly *CRg10*, was the least affected by the solution.

According to the results, it is observed that the mass losses were random except for those obtained on the days of the hydrochloric solution renewal (16, 31, 46, 61 and 76 days) where they have increased significantly. This confirms the relationship between acid concentration and aggressiveness and also explains the high solubility of hydrochloric acid (*HCl*) which reacts rapidly with Portland cement. Following this reaction, the released calcium hydroxide $Ca(OH_2)$ reacts with hydrochloric acid to form calcium chloride $CaCl_2$, accordingly produces the mass losses observed. If the medium of the cementitious composite become highly aggressive with a source of acid renewal, the attack would be very detrimental.

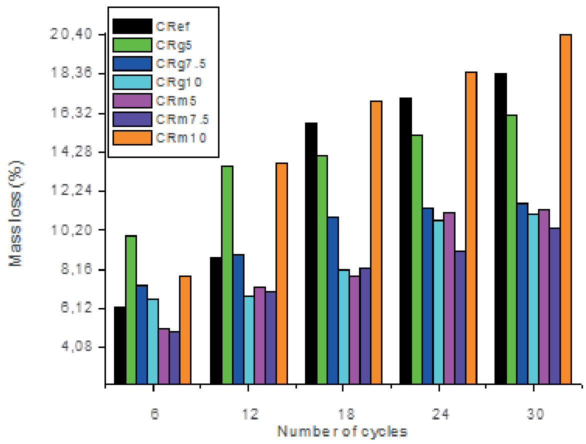

Fig. 13 Mass losses of lightweight concretes up to the 30th cycles of HCl attack

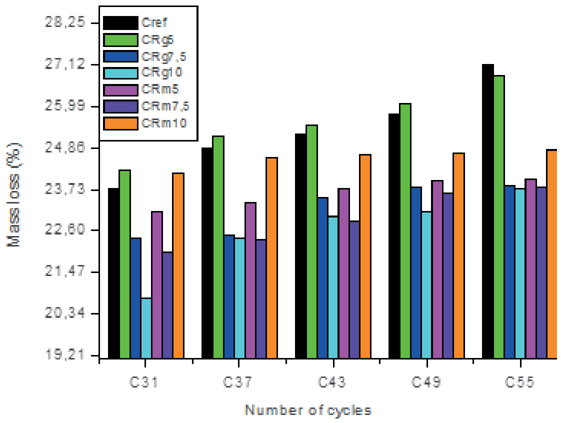

Fig. 14 Mass losses of lightweight concretes up to the 55th cycles of HCl attack

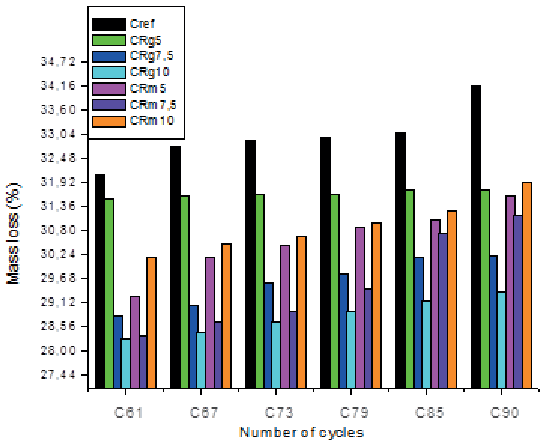

Fig. 15 Mass losses of lightweight concretes up to the 90th cycles of HCl attack

These results indicated that the mass losses of the modified mixes are less significant compared to those recorded in the case of the control mix. This proved that these mixtures resist more to this acid. This improvement was directly related to the size and content of rubber aggregates and the duration of immersion. With their amorphous nature, the rubber aggregates functioned as watertight pores, and have limited the penetration of aggressive agents into the mixtures as well.

It has been noticed during the last 12 days, that the mass loss has evolved slightly. At this stage of testing, a visual examination of the tested specimens has been done and revealed the formation of a soft layer which protected these specimens from effects of the acid solution. This examination also revealed that the texture of rubber aggregates has become rough, the untreated rubber paste/aggregate interface quality and the chemical attack have not affected their adhesion to the cementitious matrix.

It was observed (Fig. 16) an increasing porosity on the facets of the specimens due to the detachment of the natural aggregates in particular and the formation of $CaCl_2$ following the reaction of the HCl with the hydrated cement paste. These facets were yellowish due to the presence of calcium chloride ($CaCl_2$) and iron hydroxide covering the surface. Macias [45] explained this coloring by the formation of ettringite in the degraded depth. Thus, the combined effect of continuous HCl attack and the exposure to wetting-drying cycles have affected the structure of the tested specimens, consequently caused coating losses in the aggregates.

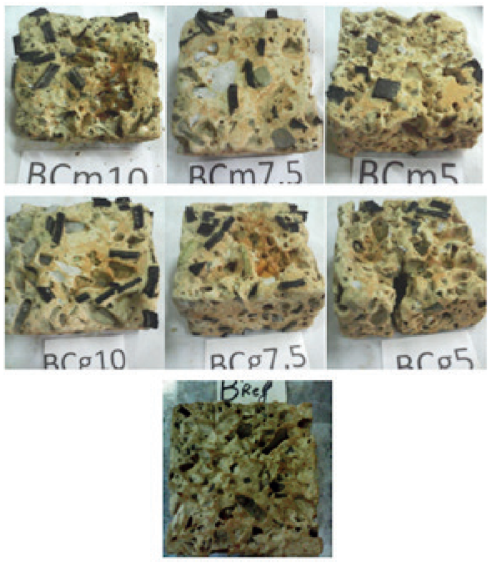

Fig. 16 Effect of hydrochloric acid (*HCl*) on lightweight concretes at 90 cycles

It has also been observed that the depth affected by the acid solution is less important in the case of modified mixes. The addition of rubber aggregates improved the resistance of their exposed parts to chemical attack from the outside. Their stabilized chemical composition did not react with the acid. However, it is concluded that these aggregates increase the chemical resistance of composites in aggressive environment. These results are not different from what has been proved by several researchers who have reported that the incorporation of polymers increases the chemical resistance of cementitious composites in aggressive environment [46].

The reaction of the lightweight concretes and the immersion solution were followed by the measuring of the *pH* of the interstitial solution of these mixes as a function of time. The *pH* evolution of the control solution was slow with a *pH* ranging between 7 and 8 for all the tested lightweight concretes. When the water surrounding the concrete infiltrates inside, it causes the dissolution of Ca^{2+} ions, this releases the OH^{-} ions and generates an increase in the *pH* of the water [47]. At the end of the test, the *pH* values of these solutions were between 10 and 11 for *Cref* and *CRg5* and between 9 and 10 for the other solutions.

After three hours of immersion, the *pH* of the *HCl* acid solutions was between 1 and 2. At 30 days the pH was between 2 and 3, showing that the reaction occurred in this time between the tested specimens and the immersion solution. At 90 days the *pH* of the immersion solution of *Cref* was between 7 and 8, while the other solutions had a *pH* of 5 to 6. It was found that *Cref* was more reactive with the immersion.

The neutralization of the acid by the cementitious composites is important if its content of cement and mineral aggregates are high. The flux of transported ions generates the dissolution of the hydrates and the formation of new compounds and various salts lead to a progressive degradation of these composites. Kumar et al. [48] have reported in their research that Portland cement concrete is acid-resistant and that no hydraulic concrete will be preserved for a long time during its exposure to a solution with a $pH \leq 3$. They have also showed that portlandite starts at a $pH < 12.5$. All of this explains the mass losses especially after the renewal of the immersion solutions.

According to the results of this test, we can conclude that the lightweight concretes formulated by mixing crumb rubber and rubber aggregates exposed to hydrochloric acid (*HCl*) for 90 days are more durable than those formulated by partially replacement of gravel only with coarse rubber aggregates.

4 Conclusions

This paper is an experimental contribution to draw attention to lightweight concretes incorporating rubber aggregates through the evaluation of the durability on freeze-thaw, elevated temperature, Na_2SO_4 immersion, *HCl* immersion, and water absorption. In this work, seven mixes were studied; control mix (no replacement) and six modified mixes containing 5%, 7.5% and 10% of rubber aggregates or rubber aggregates combined with crumb rubber with the same replacement level. From the outcome of our investigation, the following conclusions can be drawn:

- The percentage of water absorption decreased when rubber content was < 7.5%. The biggest percentage of water absorption of 5.13% was recorded in the case of control mix. The highest percentage of water absorption of 4.68% and 4.81% was observed in the case of the mixes containing 10% of rubber aggregates. The percentage of water absorption has decreased when the rubber content was ranging between 5% and 7.5%.This decrease was also depended on heterogeneity, compactness or porosity, and air content of the hardened rubberized mixes;

- The biggest mass loss of 3.1% was recorded in the case of the control mix exposed to freeze-thaw cycles, whereas*CRm5* and *CRg5* have lost 2.61% and 2.67% as the highest mass loss in both cases of modified mixes. It was concluded that the mass loss diminishes with the size decrease of the rubber aggregates and increase with the decrease of rubber content and the duration of freeze-thaw. The results also showed a slight difference between the recorded mass losses in both cases of modified mixes, therefore the choice between them depends on the required properties.

- The exposure of lightweight concretes to elevated temperature of 200°C, 400°C, 600°C, 800°C and 1000°C, has demonstrated that mass loss increased with the decrease of rubber content and the increase of the temperature heating. The mass losses of the modified mixes (*CRg*) were similar to those observed in the case of the control mix (30%).The biggest mass loss of 56.82% was observed in the case of the mix *CRm5*. These results have been also related to the size of rubber aggregates and their location relatively to the surface. Although rubber aggregates have prevented the volumetric expansion leading to important deterioration, especially when temperature exceed 600°C;

- The mass losses due to the Na_2SO_4 immersion increased slightly with the increase of the duration of immersion. The test has also revealed that the biggest mass loss of about 0.26% was observed on the specimens containing 5% of rubber aggregates (*CRg5* and *CRm5*). These results were related to the absorption rate of these mixes in the beginning of the test. It was concluded that the mass losses increased with smaller rubber content. The smallest mass loss of about 0.16% corresponded to *CRm10*. The modified mixes have resisted to Na_2SO_4 solution more than the control mix. This improvement is due to particular characteristics of rubber aggregates, especially when different sizes were mixed at the same mix. The most of the deteriorations were observed when the *pH* of solutions was < 7 and were reduced when $pH \geq 9$;

- The relationship between *HCl* concentration and aggressiveness has been demonstrated. The results have indicated that the mass losses of rubberized mixes are less significant as compared to those recorded in the case of control mix. This proved that these mixtures resist more to this acid. This improvement was directly related to the size and content of rubber aggregates and the immersion duration. At the end of this test it was concluded that the composition *CRg* in particular *CRg10* was least affected by the acid solution.

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