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DRYING SHRINKAGE, SORPTIVITY AND MICRO-STRUCTURAL CHARACTERISTICS OF CELLULAR CONCRETE CONTAINING WASTE MARBLE POWDER AS CEMENTITIOUS MATERIALS

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KEYWORDS: Cellular concrete, Waste marble powder, Drying shrinkage, Sorptivity, Micro-structural, Environment.

ABSTRACT:

Given the escalating shortage of natural resources, the excessive demand for granular materials, and the challenges associated with identifying novel quarry sites, numerous research inquiries have been undertaken to explore the feasibility of reutilizing waste materials and manufacturing by-products in lieu of conventional resources that are progressively diminishing. This research seeks to mitigate construction expenses while simultaneously promoting environmental preservation. The purpose of this experiment study is to examine the impact of waste marble powder used as a cement substitute on the properties of cellular concrete like drying shrinkage, sorptivity and micro-structural using optical microscope (OPM), and Fourier transform infrared spectroscopy (FTIR) so that it can be reused in the production of cellular concrete. More extensive and detailed research is needed. In order to accomplish this goal, three distinct varieties of cellular concrete are produced composed on sea sand, waste marble sand and river sand, with various substitution rates of waste marble powder (WMP) (0%, 10%, 15% and 20%), and different ratios aluminum powder (0 %; 0.25%; 0.50%; 0.75% and 1%). The findings revealed that: (i) The drying shrinkage decreases with increasing WMP dosage, but it increases as a function of the aluminum content. (ii) As the dosage of WMP and the amount of aluminum increase, the sorptivity of the cellular concrete decreases. (iii) Specimens that containing WMP enhanced the cellular concrete matrix's characteristics, as seen by Optical microscope images. (iv) The FTIR results clearly indicate a shift in peaks when WMP and aluminum powder are present, in comparison to the control mix. (v) Overall, the replacement of cement with appropriate WMP has a positive impact on both the manufacturing process of cellular concrete and the environment.

1. INTRODUCTION

Cellular concrete has gained popularity in recent years as an alternative to standard concrete since it minimizes dead loads on structures, facilitates to energy conservation, and decreases both production and labor expenses associated with construction and transportation (Yanbin, Xiuling, Lixin, & Yunpeng, 2020). In addition, there is a need for the development of novel raw materials in the field of cement, foaming agents, and additives for applications involving cellular concrete. Generally defined as a lightweight product (Chica & Alzate, 2019). The key elements influencing the properties of cellular concrete are microstructure and pore characteristics (Chen, Wu, & Zhou, 2013). The current research trend and the major material performance metrics for engineers into new buildings that meet strength, durability, technological, economic efficiency, low-cost and environmental requirements, such as cellular concrete, and the valorization of local materials for the development of these materials (Sherong, Kelei, Chao, Xiaohua, Jiaxin, & Benbo, 2020).

According to the literature, it has been observed that the mechanical qualities of cellular concrete can be enhanced and have the same conclusion as regular concrete by inserting supplemental cementitious materials while preserving the pore characteristics (Lian, Zhuge, & Beecham, 2011; Chen, Wu, & Zhou, 2013). Several recent research papers have examined the influence of incorporating construction and industrial waste materials on the enhancement of cellular concrete's performance characteristics. The impact of the substitution of cement in the production of cellular concrete by integrating both silica fumes (SF) and residual marble powder (WMP), both of which are considered industrial wastes with high ecological pollution and low recycling rates, and increased the durability and mechanical properties of cellular concrete was explored (Sherong, Kelei, Chao, Xiaohua, Jiaxin, & Benbo, 2020; Sarkar, 2014; Abo-El-Enein, El-Kady, El-Sokkary & Gharieb, 2015). The water absorption, the absorption efficiency, and the penetration depth all have a major impact on the durability of cellular concrete (Tikalsky, Pospisil, & Macdonald, 2004).

Some experimental studies have shown that drying shrinkage of foamed concrete typically takes place within the first 20 days of casting period (Govern, 2000), with the usual amount of 0.1% to 0.35% of the overall volume of the concrete's hardened matrix (Roslan, Awang, & Mydin, 2013) due to the loss of adsorbed water from the material. This phenomenon is especially prominent in aerated concrete, which exhibits a high total porosity ranging from 40% to 80% and possesses a specific surface area of pores in the material. The increase in the foam volume results in higher porosity and decreases the shrinkage (Nambiar, & Ramamurthy, 2009). Images in astructural model indicate drying shrinkage, which can be attributed to the compression forces owing to hydraulic removal

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of pore water. According to the capillary tension theory pertaining to the shrinkage of porous construction materials, water that exists in the pores is under tension, causing a force of attraction between the walls of such pores. Aerated concrete made wholly of cement is said to shrink substantially more than aerated concrete made entirely of lime or combination of lime and cement when dried. The least shrinkage is found in lime–cement products (Narayanan & Ramamurthy, 2000). While there is limited information available regarding the influence of cement content on the drying shrinkage of foamed concrete, a few scholars (Chindaprasirt, Rukzon, & Sirivivatnanon, 2008) have observed adverse consequences associated with higher cement content in cellular concrete. These undesirable effects may be mitigated by partially substituting Portland cement with alternative additional materials such as fly ash, silica fume, and lime, due to their reduced heat of hydration.

Sorptivity is kown for having an impact on the durability of cellular concrete because of its porous nature, there is a strong interaction between water, water vapour and the porous system, and multiple mechanisms of moisture transfer exist. Several investigations (Nambiar, & Ramamurthy, 2007) have been conducted to examine the character of sorptivity in cellular concrete. The observation has been made that the movement of water is not a simple function of porosity. Instead, it is influenced by the distribution of pores, which is affected by several factors such as mineral admixtures, the ratio of water to binder, the diameter of pores, their continuity, and their tortuosity. As a result of the increased volume of air voids, the behaviour of cellular concrete is more complex; hence rendering the assessment of moisture transfer influenced by pore size distribution and water content more challenging. The findings revealed that the water absorption of a foamed concrete mixture without mineral admixtures absorbed more water than one with admixtures, such as fly ash, which served as a partial substitute for cement. Furthermore, increased foam volume reduces sorptivity since it is characterised by capillary suction (Nambiar, & Ramamurthy, 2006). The process of water vapour transfer is described using water vapour permeability and moisture diffusion coefficient, whereas water transfer is depicted using capillary aspiration and water permeability. Capillary aspiration in cellular concrete is sluggish in comparison to porous materials. The determination of sorptivity as parameter that characterizes the moisture transport in porous materials through the absorption and capillary transfer of water, is based on the principles of unsaturated flow theory (Narayanan & Ramamurthy, 2000).

Furthermore, as a common concern of innovative cellular concrete and traditional concrete. Cement serves as the principal cementitious component employed in both conventional cellular concrete and standard concrete production. The consideration of excessive cement content is necessary. From the perspectives of both engineering application and sustainable development, it is essential to enhance the material characteristics of the newly manufactured cellular concrete, establishing the groundwork for the promotion and implementation of cellular concrete (Khodabakhshian, Brito, & Ghalehnovi, 2018 ; Evi, 2016 ; Aprianti, Shafigh, Bahri, & Farahani, 2015). In this context, the use of mineral additives in the production process of concrete in Algeria, particularly cellular concrete, allows for the recovery and recycling of materials that would otherwise be disposed of in landfills, as well as a reduction in the use of natural raw materials and CO_2 emissions by lowering the amount of cement (Khodabakhshian, Ghalehnovi, Brito, & Shamsabadi, 2018). The objective was to improve local resources that looked promising from an economic and environmental standpoint (Bouglada, Noui, & Belagraa, 2021).

On the other hand, WMP is an inert material derived from a metamorphic rock characterized by a significant concentration of calcium carbonate ($CaCO_3$) (Gesoglu, Güneyisi, Kocabag, Bayram, & Mermerdas, 2012). It has been extensively studied in common pastes, mortars, and concretes. The economic and environmental benefits and reduced cost of employing these wastes have made it a priority and studied in the composition to improve concrete qualities by several researchers (Rai, Naushad, Abhishek, Rushad, & Duggal, 2011 ; Patel, Raval, & Pitroda, 2013 ; Aliabdo, Abd Elmoaty, & Auda, 2014). Ordinary concretes incorporating MP as a partial replacement of sand offered superior resistance to carbonation, enhancing their durability (Ashish, Verma, Kumar & Sharma, 2016 ; Vardhan, Siddique, & Goyal, 2019). According to (Singh, Choudhary, Srivastava, Sangwan, & Bhunia, 2017; Singh, Srivastava, & Bhunia, 2019), adding 10-15% of marble powder (MP) as a partial replacement of cement has no effect on the setting times of concrete. However, it enhances the workability of the mixture, reduces absorption, and improves the mechanical flexural and split tensile strengths (Ashish, Verma, Kumar, & Sharma, 2016 ; Aruntas, Gürü, Dayi, & Tekin, 2010). Additionally, this substitution lowers concrete production costs and the CO_2 emissions of cement production (Arel, 2016). The capacity of the MP to infiltrate into the granular skeletal porosity of the concrete, so effectively filling voids and reducing porosity explains these gains (Baoguo, Jie, Hongbo, Xiangguo, Lixiong, Yang, & Zhugen, 2019). Mortars with MP have good cohesion (Corinaldesi, Moriconi, & Naik, 2010) and reduce the shrinkage values (Hebhoub, Belachia, & Djebien, 2014). In another study, the macroscopic performance of cement is adversely affected by the presence of MP (Nežerka, Hrbek, Prošek, Somr, Tesárek, & Fládr, 2018). The introduction of MP in cement paste at varied replacement rates of 5, 10, and 15% increases porosity. The authors concluded that 5% MP may produce satisfactory results (Toubal, Mellas, Sadowski, & Żak, 2018), up to 7.5% according to (Soliman, 2013), and up to 20% according to (Mashaly, El-Kaliouby, Shalaby, El–Gohary, & Rashwan, 2016). However, other research has looked at the effects that using WMP as a cement substitute on CC characteristics, particularly shrinkage and sorptivity.

WMP is commonly used as a partial substitute for cement in mortar and concrete manufacturing. This research investigates the feasibility of using WMP as a partial substitute for cement in the composition of cellular concrete may improve its durability by evaluating their influence on some properties such as drying shrinkage, sorptivity and micro-structural. Cellular concrete mixtures were tested at hardened state. Aside from the economic and technical benefits, This study not only provides a valuable insights for the usage of WMP as a potential replacement for cement in cellular concrete mix design, Furthermore, it provides some significant information regarding its application in civil engineering work resulting in green concrete that fits with the needs of sustainable development.

2. MATERIALS

2.1 Sands

Three distinct varieties of sands were utilized in the formulation of cellular concrete. The first type is a waste marble sand (MS) characterized as class 0/3, and the second type is a sea sand (SS) classified as class 0/1, both collected from the region of Skikda, and the third sand is a river sand (RS) sourced from the northwestern region of Constantine classified as class 0/4. The particle size distributions of the three sands are shown in (Fig. 1).

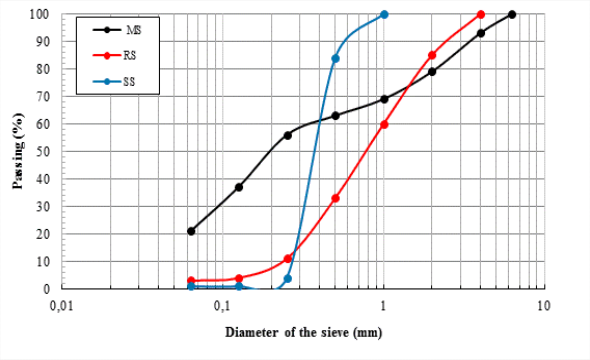


Figure 1. Granulometric curve of the sands

The grains of MS have angular shapes (shown in Fig. 2a), whereas SS and RS grains have rounded shapes (seen in Figs. 2b and 2c). Their physical properties are listed in Table 1. While, the main differences amongst these sands in terms of type, grain form, granularity, and proportion of fine constituents are given in Table 2.



a) MS b) SS c) RS

Figure 2. General aspect of used sands and the grains shape

Properties	Sand type		
	MS	RS	SS
Apparent density (kg/m^3)	1470	1570	1540
Absolute density (kg/m^3)	2600	2610	2580
Absorption (%)	4.38	1.20	2.15
Value of blue methylene (%)	0.6	0.2	0.45
Sand equivalent (%)	65	77	95
Fineness modulus (%)	2.03	3.06	2.1

Table 1. Physical properties of sands used

Sand type	Nature	Grain shape	d/D (mm)	Fineness	% of fine elements	Particle size distribution
MS	Limestone	Angular	0/3	Fine	High (21%)	Continuous spread out
SS	Siliceous	Rounded	0/ 1	Fine	Very low (0.6 %)	Continuous very tight
RS	Siliceous	Rounded	0/4	Coarse	More or less low (3.3 %)	Continuous spread out

Table 2. Comparison between used sands

2.2 Cement

In this study, Ordinary Portland Cement (OPC) of type CEM I 42.5 from Tunisian Artificial Cement company, was used in the preparation of all CC mixtures. Whose physical characteristics and chemical compositions are shown in Table 3 below.

Physical characteristics		OPC	WMP
	Apparent density (kg/m^3)	996	950
	Absolute density (kg/m^3)	3266	2800
	SSB (cm^2/g)	3869	5804
Chemical composition(%)			
	CaO	62.12	59.54
	Al_2O_3	4.69	1.06
	Fe_2O_3	4.81	0.13
	SiO_2	19.33	1.76
	Mg O	1.27	1.03
	Na_2O	2.13	0.04
	K_2O	0.44	0.03
	SO_3	2.13	0.01
	Cl^-	0.05	-

Table 3. Physical properties and chemical compositions of cement and waste marble powder

2.3 Waste marble powder

The fine employed in this study is waste marble powder (WMP), which was obtained by the process of sifting (with a sieve size of 80 µm) and crushing waste marble sand, its goal was to produce a fine with a specific Blaine surface that was equal to or greater than that of cement. Then, a fine marble powder is acquired, which will be substituted with precise percentages of binder ranging from 0 to 0.20 by the weight of the dry mixture (B = OPC + WMP). Table 3 shows the physical properties and chemical composition of these fines.

2.4 Expansive agent

The expanding agent used was aluminum powder (Al) possessed 99 % purity and 100 µm fineness. This aluminum powder was commercially provided by SIALCHIM Company.

2.5 Water

The water (W) utilized to combine the components of the cellular concrete was straight from the lab's faucet and had not been treated in any way according to NF EN1008 standards.

3. MIXING PROCEDURE

A conventional mixing machine is used to perform a dry knead the solid components of the mixture: sand, fines, and cement (EN 196-1) for duration of two minutes. Without pausing the mixing, water is slowly put into the mixture. The mixing period during the wet phase is set to four minutes: two minutes of low rotational speed mixing, followed by a stop to scrape the walls and, especially, the bottom of the bowl to achieve homogeneity in the mixture. The mixing is then restarted at a high pace for two minutes. The mixing is halted for a second time to incorporate the aluminum powder, then continued for one minute at low speed, then one minute at a rapid tempo (Goual, 2001). Molding process must be done as soon as the mixing is completed to avoid material expansion within the mixing tank.

For every mixture, a series of prisms (40 × 40 × 160 mm^3) were cast in steel molds that had been pre-oiled and stored uncovered in a climatic chamber (T = 20 ± 2 ° C and RH = 95 ± 3%) for approximately 24 hours. Shortly before demolding, the excess material of the expanded portions of the samples, which occurred due to a chemical reaction with aluminum powder, was cleared. Following that, the samples have been removed from their molds. All test specimens are maintained in a climate-controlled room until their age for testing. The experimental findings in the current study are the mean of three measurements obtained from three samples of each combination of several types of sand used to produce cellular concrete. All of the CC combinations' proportions are displayed in Table 4.

Mix Series	Mix ID	B=WMP+OPC		B/(S+B) (%)	S/(S+B) (%)	Al /B(%)	W/B(%)
		WMP/B(%)	OPC /B(%)				
Series 1 : SS, OPC, W/B = 0.50							
SSCR	SSC Al0	0	100	35	65	0	0.50
	SSC Al25	0	100	35	65	0.25	
	SSC Al50	0	100	35	65	0.50	
	SSC Al75	0	100	35	65	0.75	
	SSC Al1	0	100	35	65	1	
SSC M10	SSC M10 Al0	10	90	35	65	0	
	SSC M10 Al25	10	90	35	65	0.25	
	SSC M10 Al50	10	90	35	65	0.50	
	SSC M10 Al75	10	90	35	65	0.75	
	SSC M10 Al1	10	90	35	65	1	
SSC M15	SSC M15 Al0	15	85	35	65	0	
	SSC M15 Al25	15	85	35	65	0.25	
	SSC M15 Al50	15	85	35	65	0.50	
	SSC M15 Al75	15	85	35	65	0.75	
	SSC M15 Al1	15	85	35	65	1	
SSC M20	SSC M20 Al0	20	80	35	65	0	
	SSC M20 Al25	20	80	35	65	0.25	
	SSC M20 Al50	20	80	35	65	0.50	
	SSC M20 Al75	20	80	35	65	0.75	
	SSC M20 Al1	20	80	35	65	1	
Series 2 : RS, OPC, W/B = 0.55							
RSCR	RSC Al0	0	100	35	65	0	0.55
	RSC Al25	0	100	35	65	0.25	
	RSC Al50	0	100	35	65	0.50	
	RSC Al75	0	100	35	65	0.75	
	RSC Al1	0	100	35	65	1	
RSC M10	RSC M10 Al0	10	90	35	65	0	
	RSC M10 Al25	10	90	35	65	0.25	
	RSC M10 Al50	10	90	35	65	0.50	
	RSC M10 Al75	10	90	35	65	0.75	
	RSC M10 Al1	10	90	35	65	1	
RSC M15	RSC M15 Al0	15	85	35	65	0	
	RSC M15 Al25	15	85	35	65	0.25	
	RSC M15 Al50	15	85	35	65	0.50	
	RSC M15 Al75	15	85	35	65	0.75	
	RSC M15 Al1	15	85	35	65	1	
RSC M20	RSC M20 Al0	20	80	35	65	0	
	RSC M20 Al25	20	80	35	65	0.25	
	RSC M20 Al50	20	80	35	65	0.50	
	RSC M20 Al75	20	80	35	65	0.75	
	RSC M20 Al1	20	80	35	65	1	
Series 3: MS, OPC, W/B = 0.60							
MSCR	MSC Al0	0	100	35	65	0	
	MSC Al25	0	100	35	65	0.25	

Table.4. Continued							
	MSC Al50	0	100	35	65	0.50	
	MSC Al75	0	100	35	65	0.75	
	MSC Al1	0	100	35	65	1	
MSC M10	MSC M10 Al0	10	90	35	65	0	0.60
	MSC M10 Al25	10	90	35	65	0.25	
	MSC M10 Al50	10	90	35	65	0.50	
	MSC M10 Al75	10	90	35	65	0.75	
	MSC M10 Al1	10	90	35	65	1	
MSC M15	MSC M15 Al0	15	85	35	65	0	
	MSC M15 Al25	15	85	35	65	0.25	
	MSC M15 Al50	15	85	35	65	0.50	
	MSC M15 Al75	15	85	35	65	0.75	
	MSC M15 Al1	15	85	35	65	1	
MSC M20	MSC M20 Al0	20	80	35	65	0	
	MSC M20 Al25	20	80	35	65	0.25	
	MSC M20 Al50	20	80	35	65	0.50	
	MSC M20 Al75	20	80	35	65	0.75	
	MSC M20 Al1	20	80	35	65	1	

Mix ID (Example): SSC M10 Al25:SSC/ Sea sand cellular concrete – M/Waste Marble powder: 10 % – Al / Aluminum powder: 0.25%

Table 4. Mixture proportions by weight percentage of the different CC mixtures

4. TESTING PROCEDURES

In order to investigate the shrinkage of CCs, specimens of 40×40×160 mm were made for each mixture in accordance with NF P 15-433 standard (Fig. 3). Regarding the evaluation of drying shrinkage, it is important to take certain factors into account. The samples are maintained in a dry environment at a specific temperature of T=25±3°C and a relative humidity of RH=45±5 %. The assessment of length variations was evaluated after 28 days of the drying process. The results that are being given are the means of three samples.



Figure 3. Measurement of drying shrinkage

Porosity is a determining factor in the durability of cellular concrete. The capillary absorption test (Sorptivity) is one of the parameters that control the transport of deleterious materials inside concrete. It is employed to measure the rate of water absorption through capillary suction in cellular concrete, and was performed at 28 days and 90 days of casting. For the test, prismatic specimens 4x4x16 cm^3 were cast. Specimens were placed vertically on their bases measuring 4x4cm^2 into contact with water at a temperature of (20 °C) without hydraulic pressure (Fig. 4). Furthermore, the specimens underwent pre-conditioning in an oven until a constant mass. In order to prevent moisture interaction with the outside environment, a sealed plastic sheet was used to cover side faces and top surface of the specimens, while leaving the bottom surface uncovered. The initial weight of the sample was measured at time equal to 0 min. Then after, the sample was submerged in water at a depth of 5 mm. To determine the water absorption rate by capillary, the weight change of the specimen was measured at regular intervals of a period up to 4 hours (20, 40, 60, 80, 100, 120, 140, 160, 180, 200, 220, 240 min). This allows indirect characterization of capillary porosity. A graph between the square root of time and the increase in mass per unit area over the density of water was plotted. The slope of the best-fit regression line was used as a means to quantify the specimen's sorptivity. The capillary absorption coefficient (Sorptivity) was determined and defined using formula:

$$i(t) = \frac{M(t) - M(0)}{\rho w \cdot Aab} = Sw\sqrt{t}$$

Where: i (t): is the water absorption volume per unit area in ($g/cm^{2)}$; M(t) : is the wet mass of the sample measured at time *t* in (g); M(0): is the initial dried mass of the sample in g; ρ_w: is the density of the liquid water (1 g/cm^3), A_{ab}: is the section area in contact with water (A= 4×4 =16 cm^2) ; and S_w is the sorptivity (capillary absorption coefficient) of the material in ($g/cm^2.min^{-1/2}$).

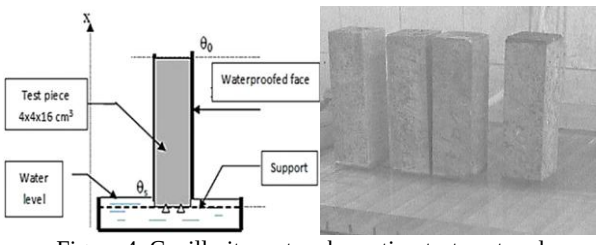


Figure 4. Capillarity water absorption test protocol

In order to examine the micro-structural properties of cellular concrete, the samples are subjected to analysis using an optical microscope at the Research Laboratory on the Physico-Chemistry of Surfaces and Interfaces, located at the University of Skikda (Fig. 5). The optical microscope, as its name suggests, is a technique that works on the principles of light which allows us to obtain a magnified image of objects, and cement materials and to facilite the observation of intricate details that are invisible to the naked human eye.

However, it is important to note that the resolution of these microscopy techniques is limited, as cannot exceed 0.1µm. The photographs taken using OPM are automatically analyzed with the help of an image processing software, which allows us with its separating power to obtain information on the shape and size of the pores present in the material examined, as well as their distribution, according to their varying shade of grey, i.e., indicates its ability to distinguish two points located next to each other.



Figure 5. OPM optical microscope examination

The Fourier Transform Infrared spectrometer (FTIR) model QATR-S from SHIMADZU at the laboratory of growth and characterization of novel semiconductors, which is situated at the University of Sétif (Fig. 6), was used to obtain FTIR analysis of various compositions of cellular concrete. It enables the identification of surface groups of functional materials. An experimental transmission mode was implemented. In order to reduce water interference, the specimens were ground into powder, and 1 mg of the of pulverised samples was combined with 100 mg of KBr that had been oven-dried for an extended period of time. Following this, the mixture is exposed to high pressure to produce a particle that is transparent to visible light. The pellet was affixed to a holder and thereafter analyzed using infrared spectroscopy in the wavelength range of 4000 to 400 cm^{-1}. Each spectrum was obtained by summing 32 scans with a spectral resolution of 4 cm^{-1}. The microstructure of cellular concrete resulting from FTIR analysis was examined using ORIGIN-2018 software to generate the fundamental graphs.



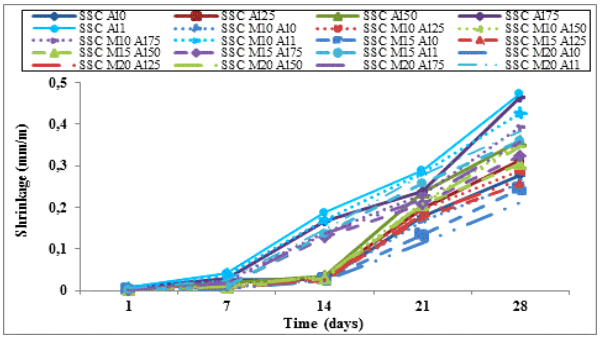
Figure 6. IR spectroscopy scanning instrument

5. RESULTS AND DISCUSSION

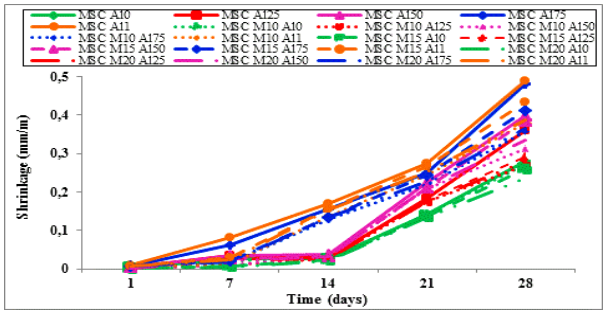
5.1 Drying shrinkage

Figures 7 show the effect of the WMP on the drying shrinkage of CC up to 28 days for different dosages of the aluminum powder. The shrinkage increases with the increase in cement dosage since a larger amount of hydrated cement paste is responsible for shrinkage (Neville, 2000). For all the cases of elaborated concretes, the results show that the process of drying shrinkage develops rapidly during the initial days, but, after the fifteenth day, it begins to evolve slowly with a tendency to stabilize after the twenty-one days. As seen in figures 6, it is clear that the reference concrete composition SSCR, MSCR, and RSCR has higher drying shrinkage values than the other CC mixtures containing WMP. The SSCR, MSCR, and RSCR reached shrinkage values up to 0.474 mm/m, 0.488 mm/m, and 0.455 mm/m respectively with aluminum percentages ranging from 0 to 1% at 28 days. Regarding the substitution of the OPC by the WMP in the composition of CC, the results show that drying shrinkage values were reduced during all drying durations. This decrease is more pronounced in the case of specimens SSCM15, RSCM15, and MSCM10 and the highest value is obtained in the specimens SSCM10, RSCM10, and MSCM15. As can be seen, drying shrinkage decreases with an increase in WMP content (Vardhan, Siddique & Goyal, 2019). The filling effect observed in these fines"WMP" (Baoguo, Jie, Hongbo, Xiangguo, Lixiong, Yang, & Zhugen, 2019; Jiang, Guo, Chen, Zhu, & Jin, 2018; Djebien, Abbas, Bouabaz & Ziada, 2022), which has the ability to infiltrate within the porous structure of CC and fill the capillary gaps. Consequently, this impairs the evaporation of water through the capillary pores, can be used to explain why CC containing WMP exhibits less drying shrinkage. The addition of 10%, 15% and 20% WMP as cement replacement resulted in a reduction of drying shrinkage by 10,07 %, 23,77% and 18,87% for SSCM10, SSCM15, SSCM20 respectively compared to the reference mixture SSCR, the decrease was about 22,49 %, 10,85 % and 20,79 % for MSCM10, MSCM15, MSCM20 respectively, as compared to the reference mixture MSCR, and the decline was about 9,17 %, 25,04 % and 17,03 % for RSCM10, RSCM15, RSCM20 compared to the reference mixture RSCR , when the aluminum content of 1% after a drying period of 28 days. This decrease can be also attributed to the good cohesion of WMP resulting in a denser and more compact concrete structure (Corinaldesi, Moriconi, & Naik, 2010), to the enhancement of the hydration of cement and the creation of C-S-H which lowers drying shrinkage values (Topçu, Bilir, & Uygunoglu, 2009).

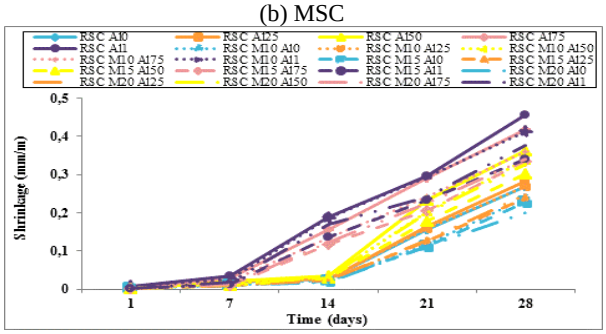
According to experimental findings, the drying shrinkage of CC increases with increasing aluminum content. The increase in drying shrinkage was almost 39,69%, 47,30% and 49,41% for MSCM10, SSCM15, and RSCM15 respectively when the aluminum content in the range of [0–1%]. It is noted that whatever the percentage of expansive agent (Al), the drying shrinkage of compositions containing WMP (WMP + OPC) is lower than that of cellular concrete with only of OPC (without WMP).



(a) SSC



(b) MSC



(c) RSC

Figure 7. Effect of WMP content on the drying shrinkage of CC for various aluminum powder percentages

Figure 8 shows the impact that the type of sand has on the drying shrinkage of different cellular concretes. Based on the results regarding the drying shrinkage of CC, which is composed of siliceous sands, it can be inferred that the mixtures elaborated with RS have a lower drying shrinkage than those elaborated with SS. Neville (2000) pointed out that the drying shrinkage is less wherever the maximum diameter of the aggregate is greater. However, in terms of the cellular concretes made with MS (limestone sand), the findings exhibit significantly greater drying shrinkage compared to the other two types of sands (siliceous sands).

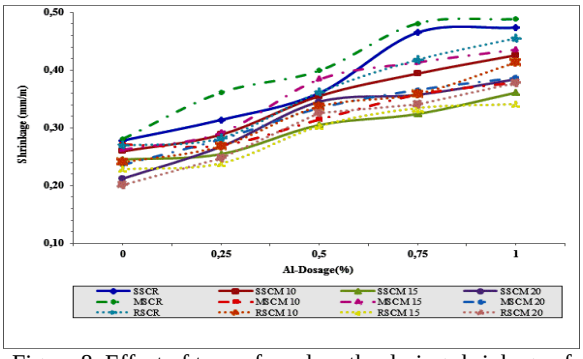


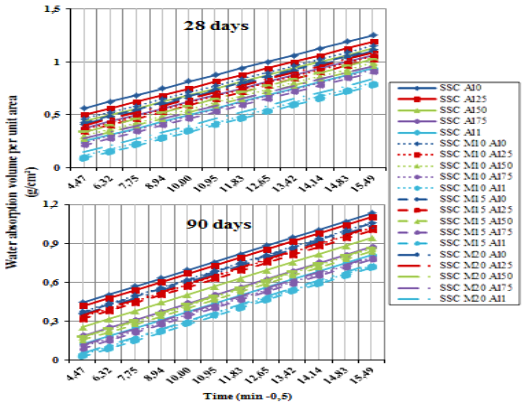
Figure 8. Effect of type of sand on the drying shrinkage of CC

This variation in the performance of the different types of cellular concretes can be attributed primarily to the high amount of fines present in the waste marble sand (21%), which means a greater surface area could potentially necessitate a higher quantity of water during mixing (Water/Binder = 0.60) in order to preserve a consistent fluidity. More water is initially present in the mixture, indicating a more porous structure that facilitates more rapid water loss from the concrete, and, thus, a greater excess of water evaporates and the concrete undergoes volume reduction during the curing process, which results in higher drying shrinkage compared to siliceous sands. Additionally, it is worth noting that the angular shape and surface roughness of waste marble sand have more interlocking between particles, which leads to higher porosity, which might result in more void spaces and increased shrinkage as water evaporates these pores. So, it can be said that the shape and size of sand particles affect the evolution of the drying shrinkage of CC.

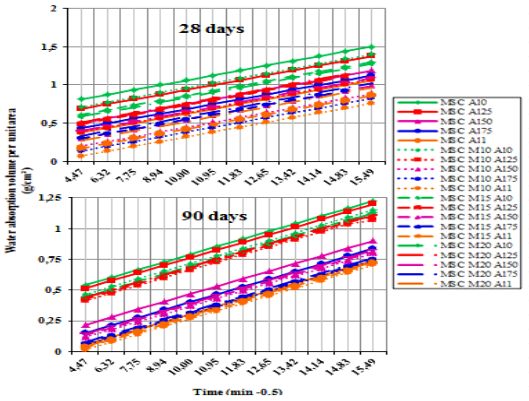
5.2 Durability indicator: Sorptivity (Capillary absorption)

Given the well-established impact of water on the deterioration of porous construction materials (Karagiannis, Karoglou, Bakolas & Moropoulou, 2016), it becomes imperative to gain a comprehensive understanding of moisture migration within concrete. This knowledge is essential for evaluating the longevity of concrete structures and for the purpose of enhancing their overall quality.

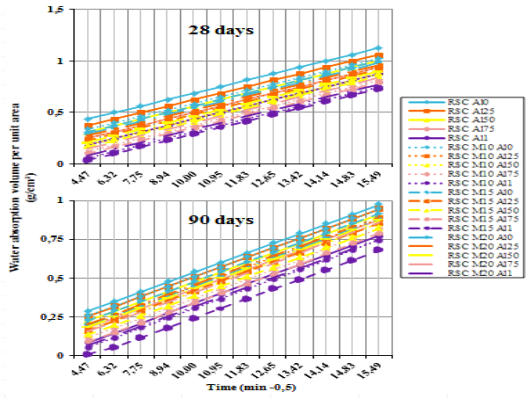
Two popular metrics that are used for assessing the amount of water that is absorbed and transported through capillary action within building materials which are the capillary absorption coefficient and water absorption (Karagiannis, Karoglou, Bakolas & Moropoulou, 2016). In order to better comprehend of how the presence of WMP impacts the sorptivity (capillary absorption) properties of cellular concrete, the sorptivity of CC from different mixes was examined after 28 days and 90 days. The evolution of the initial water absorption volume per unit area of the different compositions of CCs is illustrated in Figures 9. As anticipated, the water absorption declined as the curing period increased, owing to the ongoing hydration process of the cellular concrete. The incorporation of waste marble powder resulted in a reduction in water absorption. Water absorption is typically associated with the structural porosity, which enhances the overall longevity of the mixture. These findings have previously been validated by other studies (Vardhan, Siddique & Goyal, 2019). The mixtures made from siliceous sands containing 15% WMP and the mixture prepared with waste marble sand containing 10% WMP exhibit the lowest water absorption values. The results are consistent with the sorptivity findings.



(a) SSC



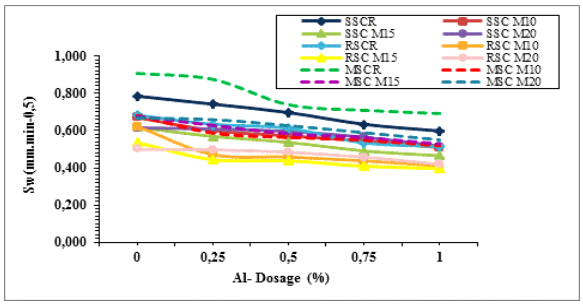
(b) MSC



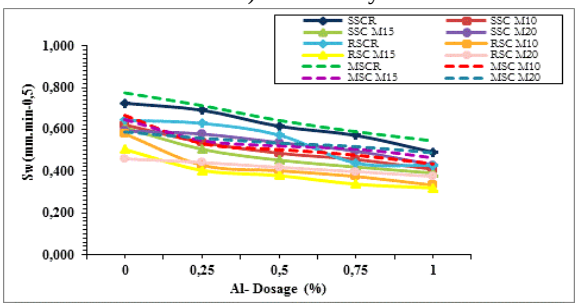
(c) RSC

Figures 9. Evolution of the initial water absorption volume per unit area up to 4 hours, and calculated slope corresponding sorptivities for each compositions

The calculated sorptivity coefficient for various CC compositions was depicted in Figure 10 as the slope of the initial section of the absorption curve per unit area. The reference mixtures SSCR, RSCR, and MSCR demonstrated the highest sorptivity values, as can be observed. Regarding the effect of the WMP when is used as a cement substitute, the sorptivity decreases as the WMP content increases in the mixture. The inter-connection of capillary gaps and the decrease in porosity caused by the addition of WMP explains this tendency (Djebien, Abbas, Bouabaz & Ziada, 2022). The mixtures SSCM15, RSCM15 and MSCM10 exhibited a reduction in sorptivity of 24.75%, 25.89%, 23.90%, respectively ((Figure 10(a)). These results were obtained with aluminum percentages ranging from 0 to 1% at 28 days. Several investigations (Singh, Srivastava & Bhunia, 2019; Gameiro, de Brito & da Silva, 2014) revealed that the sorptivity decreased as the WMP level increased. The inclusion of waste marble may be necessary to enhance the pore structure and microstructure of the concrete matrix as a result of dense mix creation, which produces finer particles than cement. A high WMP content can considerably decrease the capillary absorption of cellular concrete incorporating WMP. Based on Figure 10(b), the capillary absorption of cellular concrete at 90 days is lower than it is at 28 days. For the mixtures of SSCM15, RSCM15, and MSCM10, the decrease in sorptivity was 35.69%, 36.50%, and 34.78%, respectively, with aluminum amounts ranging from 0 to 1%. Capillary absorption of cellular concrete decreases with increasing curing age. This is mostly because as concrete ages, more hydration products are formed, which reduces sorptivity. Concerning the effect of the aluminum content, it can be shown that in all mixtures, sorptivity decreases as the expanding agent content increases due to a decrease in pores capillaries when an expansive agent is introduced into the concrete matrix.



a) At 28 days



b) At 90 days

Figures 10. Effect of type of sand on the evolution of sorptivity of CC as a function of WMP content with aluminum percentages ranging from 0 to 1%

As a result of the diverse sand types, Figure 10 illustrates how sand type influences the capillary absorption of various cellular concretes. The results indicate that the capillary absorption of formulations made with MS (limestone sand) is greater than that of those made with SS and RS (siliceous sands). This expected result can be attributed to the higher absorption coefficient (4.38%) and angular geometry of waste marble sand with rough surfaces, which indicate greater porosity and facilitate the formation of an open structure with interconnected voids in the interface between the matrix and sand of the waste marble sand (Hebhoub, Kherraf, Abdelouahed, & Belachia, 2020). Additionally, the significant volume of capillary pores enhances the water movement freely through the concrete, resulting in accelerated absorption and a higher sorptivity, on the one hand.In addition, the higher proportion of fines (21%) found in the waste marble sand indicates that the compactness of the sand-binder paste is more significant, resulting in increased expansion and the presence of more enclosed pores in the matrix (Ferhat, Goual, Damene, & Quéneudec-t'Kint, 2023), on the other side which increases the sorptivity. Conversely, in the case of mixtures of CC containing siliceous sands, the increase in sorptivity is greater for cellular concrete made with SS (finer grains) than for cellular concrete made with RS (coarser grains). This is a result of the small size of the SS grains, which makes the pores very fine and therefore promotes the rise of the water by capillarity.

5.3 Fourier transforms infrared spectroscopy (FTIR)

For this kind of examination, various compositions were selected. The infrared spectra of all samples were carried out after 9 months of conservation in the laboratory environment, focusing on the distinctive wave numbers ranging from 400 to 4000 cm^{-1}. The functional groups included in the mixtures were also identified. Figure 11 displays the infrared spectra of specimens with constant aluminum content and varying percentages of waste marble powder. It clearly illustrates that all absorption peaks have shifted towards lower wave numbers. The stretching vibrations of the O-H bond associated with the hydroxyl groups are reflected by the absorption bands at 3640.89 cm^{-1}, 3638.03 cm^{-1}, and 3647.10 cm^{-1}, respectively. These vibrations indicate the presence of calcium hydroxide $Ca(OH)^2$, also known as portlandite.

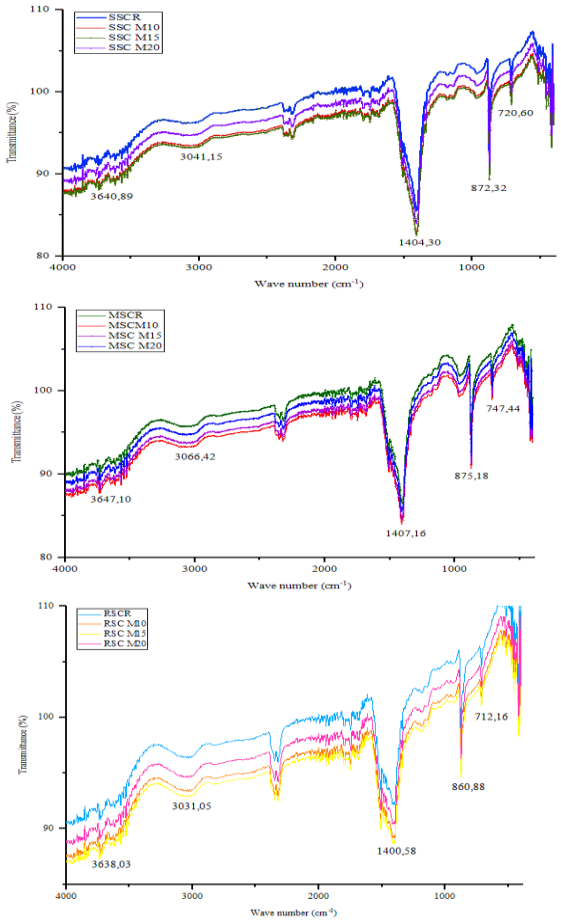


Figure 11. Infrared spectra of CC compositions with a constant aluminum content and varying percentages of WMP

A greater reduction in the intensity of these bands has been noted in compositions that incorporate substitutions of WMP (10%, 15%, or 20%) as compared to the control (which does not contain WMP). The absorption bands at 3041.15 cm^{-1}, 3066.42 cm^{-1}, and 3031.05 cm^{-1} indicate the potential presence of hydroxyl groups in the aluminates or silicates found in cement. The intensities of the bands comprising [10-15%] of WMP are nearly identical across all concrete samples, with the exception of the bands containing 20% of WMP, which are marginally larger and can be noticed. The most intense absorption zone occurs between 1300 cm^{-1} and 1600 cm^{-1}, showing a high level of infrared reactivity for the vibration. The absorption bands at 1404.30 cm^{-1}, 1407.16 cm^{-1}, and 1400.58 cm^{-1} correspond to the out-of-plane bending vibration of the C–O bond, which is caused by the calcium carbonate $CaCO_3$ related to the planar $(CO_3)^{-2}$ ions. The absorption band observed at 872.32 cm^{-1}, 875.18 cm^{-1}, and 860.88 cm^{-1} correspond to the asymmetric stretching vibration of CO_3^{-2}. The absorption band observed at 720.60 cm^{-1}, 747.44 cm^{-1}, and 712.16 cm^{-1} corresponds to the split in-plane bending vibrations of CO_3^{-2} (Damene, Goual. Houessou, Dheilly, Goullieux, & Quéneudec, 2018). Based on the specific calcium carbonate polymorph, $CaCO_3$ is hypothesized to originate from the waste marble powder composition itself, given that it is predominantly constituted of calcium carbonate, and potentially from the carbonization of CC. The characteristic peaks observed at 986cm^{-1}, 1049cm^{-1}, and 1078cm^{-1} for RSC, SSC, and MSC, respectively; indicate the formation of additional C-S-H bonds linked to Si-O bonds. This contributes to the durability of cellular concrete.

Similarly, Figures 12 represents the specimens with a fixed dosage of WMP at 10% for mixtures made with waste marble sand and 15% for mixtures prepared with siliceous sands, while the aluminum content varies. A near-identical spectrum has been observed.The incorporation of aluminium powder into the mixture decreases the intensity of the fine peaks to 3633.04 cm^{-1}, 3630 cm^{-1}, and 3642.25 cm^{-1}, which corresponds to a decrease in the surrounding environment's portlandite concentration. The observed outcome may be ascribed to the aluminum powder present, which impedes the precipitation-induced development of portlandite (Damene, Goual. Houessou, Dheilly, Goullieux, & Quéneudec, 2018). Moreover, the utilization of aluminum powder promotes the accumulation of calcite within the surrounding environment. The distinct peaks observed at 872.32 cm^{-1}, 875.18 cm^{-1}, and 860.88 cm^{-1} provide evidence for this, as they correspond to the deformation vibrations of the C-O bond. Additionally, the existence of this substance induces a slight increase in C-S-H hydrates, as indicated by the vibrations of 986cm^{-1}, 1049cm^{-1}, and 1078cm^{-1} that are linked to Si-O bonds.

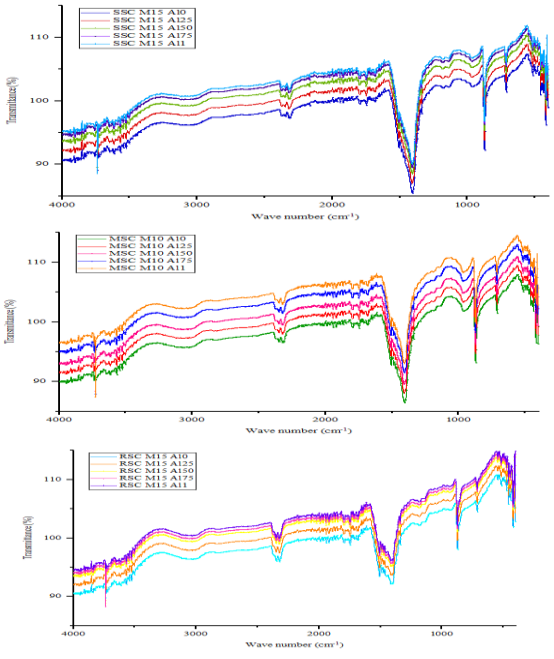
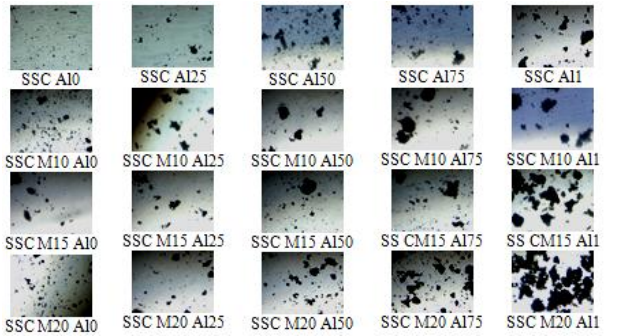


Figure 12. Infrared spectra of CC compositions with a constant WMP content and varying percentage of aluminum

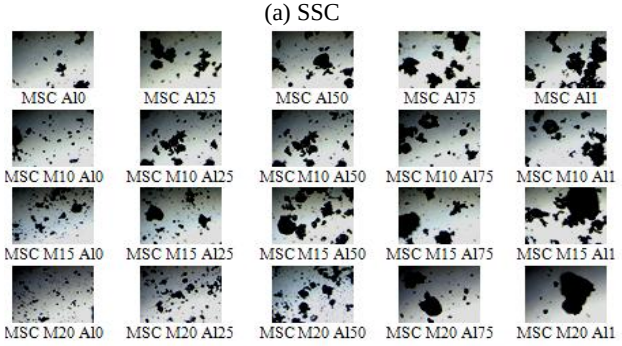
5.4 Optical Microscope (OPM)

In order to enhance comprehension of the impact of WMP on the micromorphological properties for the various mixtures of cellular concrete, Optical Microscope (OPM) images were taken of specimens.

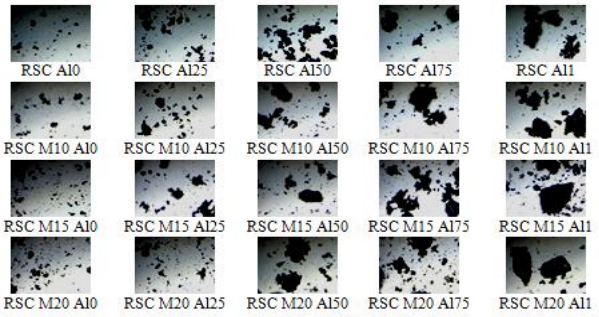
An examination of the visual analysis of Figures 13 reveals a uniform dispersion of sand particles and the binding matrix throughout all compositions. Notably, the cellular concrete incorporating waste marble powder (WMP) exhibits significantly greater compactness and denseness microstructure inside the matrix in comparison to compositions containing only Ordinary Portland Cement (OPC). Furthermore, enhancing the interface transition zone (ITZ) is achieved by the filling action of WMP particles on the qualities of the resulting mixture (Baoguo, Jie, Hongbo, Xiangguo, Lixiong, Yang, & Zhugen, 2019). This augmentation contributes to the improvement of the pore structure within the cellular concrete. In the case of the compositions without aluminium we notice fine pores whose distribution is much more regular and amplified with the increase of the dosage in WMP. All of the images show visible voids when the aluminum powder (Al) was added in various ratios (0.25%; 0.50%; 0.75% and 1%), the pores become more significant and increase in size as the proportion of aluminum in the mixture increases. The images show the existence of sometimes interconnected pores that have an irregular shape (e.g. images of samples with 1% Al). The observed pores are a result from the process of expansion of the paste due to the release of hydrogen gas.



(a) SSC



(b) MSC



(c) RSC

Figure 13. OPM images made on fractions of the different compositions of the developed CC

6. CONCLUSIONS

This study is based on the manufacturing of cellular concrete and the analysis of its properties. The objective of this work was to examine the feasibility of the reuse of WMP as a partial replacement for cement in the production of efficient cellular concrete, as well as to evaluate the impact of the sand type and amount of aluminum used on the shrinkage, sorptivity, and microstructural properties of cellular concrete. The key conclusions of this research are as follows:

1. For the different CCs elaborated with varying WMP and aluminum contents, the results show that the drying shrinkage values decrease with the addition of WMP due to its filling action and increase with the aluminum content.
2. The results of the capillarity absorption (sorptivity) of the elaborated CCs decrease with the addition of WMP and Al content. The test outcomes demonstrate that the incorporation of WMP as a substitute for cement enhances both the quality of the CC matrix and its overall durability, and that the manufactured CCs are poorly absorptive materials. The mixtures elaborated from siliceous sands comprising 15% WMP and the mixture prepared with waste marble sand comprising 10% WMP, respectively, exhibited the lowest sorptivity values.
3. The examination of the optical microscope of the different CCs reveals that the incorporation of WMP enhances the pore structure and degrees of interconnectivity within the cellular concrete. These pores enlarge as the concentration of aluminum powder increases.
4. The study of the microstructure of the CC using FTIR analysis results show that the presence of WMP and aluminum powder causes distinct peak shifts in contrast to the control mixture. The bands associated with C-S-H molecules exhibit also a shift towards lower values when the quantity of WMP increases. Additionally, these bands become more pronounced with higher levels of aluminum powder.
5. The CC mixtures elaborated with waste marble sand exhibited a larger drying shrinkage after the water evaporating present in the samples. They also had higher sorptivity values owing to the capillary pores' high volume percentage than the CC mixtures formulated on siliceous sands.

6. The use of waste marble, whether in sand or powder form, has different functions in cellular concrete, influencing its properties significantly. By using waste marble as sand, it requires a means of protection or additional components to improve both sorptivity and shrinkage from the standpoint of material optimization, thereby expanding the range of potential structural applications for this type of cellular concrete. On the contrary, waste marble as powder (WMP) functions as microfiller that enhances the microstructure of cellular concrete and reduces both sorptivity and shrinkage. This material decreases greenhouse gas emissions significantly and conserves more natural resources, making it advantageous both technologically and ecologically.

7. PERSPECTIVES

- Many factors affect the properties of cellular concrete, therefore, further in-depth microstructural analysis like X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM), or thermogravimetric analysis (TGA), are necessary in order to obtain more comprehensive information and to understand the interaction between the material's constituents and porous systems.
- The applicability of waste marble powder (WMP) in cellular concrete warrants thorough investigation, analysis, and study of the long-term behavior of these concretes in chemically aggressive environments. It is worth noting that the bulk of existing literature predominantly focuses on the feasibility of incorporating WMP into the most common two types, which are conventional or self-compacting concrete.

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