Hindawi
Advances in Materials Science and Engineering
Volume 2019, Article ID 4609353, 15 pages
https://doi.org/10.1155/2019/4609353

Research Article

Effects of Incorporation of Marble Powder Obtained by Recycling Waste Sludge and Limestone Powder on Rheology, Compressive Strength, and Durability of Self-Compacting Concrete

Rayed Alyousef,[1] **Omrane Benjeddou**,[2,3] **Chokri Soussi**,[1,2]
Mohamed Amine Khadimallah,[1,4] **and Abdeliazim Mustafa Mohamed**[1]

[1]*Prince Sattam bin Abdulaziz University, College of Engineering, Civil Engineering Department, Alkharj, Saudi Arabia*
[2]*Higher Institute of Technological Studies of Sfax, Department of Civil Engineering, Sfax, Tunisia*
[3]*University of Tunis El Manar, National Engineering School of Tunis, Civil Engineering Laboratory, Tunis, Tunisia*
[4]*University of Carthage, Polytechnic School of Tunisia, Laboratory of Systems and Applied Mechanics, Tunis, Tunisia*

Correspondence should be addressed to Omrane Benjeddou; benjeddou.omrane@gmail.com

Received 26 October 2018; Revised 26 January 2019; Accepted 26 February 2019; Published 4 April 2019

Academic Editor: Pietro Russo

Marble has been commonly used as a building material since ancient times. The disposal of waste materials from the marble industry, consisting of sludge that is composed of powder mixed with water, is one of the current worldwide environmental problems. This experimental study aims to valorize marble powder, which is achieved by grinding the sludge as filler added to the cementitious matrix of self-compacting concrete (SCC). The main purpose of this work is to evaluate the marble filler effects on the rheology in the fresh state and on the hardened properties of SCCs compared to those of limestone filler. To this end, two SCCs, SCCM and SCCL, manufactured using marble powder and limestone filler, respectively, were prepared and tested. The fresh properties of the two SCCs' mixtures were determined by slump flow, L-box, V-funnel, sieve stability, bulk density, and air content. Tests on hardened SCCs included compressive strength, homogeneity, and quality in terms of ultrasonic pulse velocity and durability against carbonation and water penetration. In addition, scanning electron microscope (SEM) and X-ray diffraction (XRD) were used to analyze the specimens.

1. Introduction

Self-compacting concrete (SCC) was first developed in Japan in 1988 [1] and is a new concrete that is fully compacted without external energy. SCC provides economic, social, and environmental benefits compared to conventionally vibrated concrete. SCC is made from the same basic constituents as conventional concrete, but with the addition of high levels of superplasticizer (SP) admixtures in order to obtain high workability [1, 2]. The self-compactabilty of SCC is defined as the capability of the concrete to be uniformly filled and compacted in the formwork corners by its self-weight without vibration during placement [3].

Singh et al. [4] and Sharifi et al. [5] indicated that the addition of inert, pozzolanic/hydraulic or waste and recycling materials leads to a significant reduction in the cost of SCC.

There are many types of mineral additions used in SCC composition but the most common is the limestone powder [6–8]. Having a property of physical improvement of the cement paste matrix, limestone filler is one of the materials that have extensively been studied in the literature. The effects of limestone powder on the rheological and mechanical properties of SCC have been widely studied. The results of these studies show that when adding limestone powder to the cementitious matrix of SCC, the workability improved with reduced cement content, the segregation resistance increased, and the pore distribution was modified [6–10]. Another research [11] indicate that limestone powder decreases both plastic viscosity and yield stress. In addition, the incorporation of limestone powder increases the density of the paste, which is particularly important in improving the compressive strength [12]. Marble ranks the largest produced natural stone in the world, and it accounts for 50% of the world's natural stone production [13]. The marble industry produces a lot of waste in the form of powder, sludge, and pieces of irregular size. Marble powder is a waste material generated in considerable amounts in the world [14]. For example, during the cutting process alone, 20–30% of a marble block becomes waste marble powder [14]. It was also reported that about 50% of the marble blocks is wasted during the production process; 90% of the waste particles are below 200 μm [15].

The considerable amounts of marble powder waste cause serious environmental problems [14]. The main solution is to recycle and reuse this inert powder in construction and building materials. This method offers certain advantages, such as protecting natural resources, saving energy, contributing to the economy, decreasing waste materials, and investing for the future [16].

Marble filler materials can be successfully and economically utilized to improve certain fresh and hardened SCC properties [17]. The effects of substituting cement with marble powder on the rheological and mechanical properties of self-compacting mortar (SCM) have been studied [18–25], and the results indicated an improvement in the workability and compressive strength of SCM with the use of marble powder.

Research on SCC has focused on improving both its fresh and hardened properties. Okamura and Ouchi [1] demonstrated that the self-compactability of SCC may be affected by various parameters, such as the material properties, mix proportions, and mix-design method. Furthermore, they indicated that the self-compactability is highly sensitive to the water-filler ratio and coarse and fine aggregate sizes. They demonstrated that the paste in the SCC requires high viscosity as well as high deformability. Moreover, this deformability is affected by the water-filler ratio, which is preferred over the water-cement ratio because most filler materials are not reactive. Furthermore, Singh et al. [4] indicated that the SCC rheology depends on the ambient temperature, mixer type, and mixing efficiency.

Shindoh and Matsuoka [3] indicated that a superior SCC should deform effectively under its own weight, without segregation of ingredients. They also demonstrated that the slump flow test is applied only as an evaluation test for estimating the SCC deformability. In order to achieve this, they developed a new test method for evaluating the SCC deformability and resistance to segregation.

Attachaiyawuth et al. [26] develop a new mixing method that enables entrained air bubbles to be suitable for enhancement in self-compactability. In the same case, Ouchi et al. [27] improved SCC properties in the fresh state with the addition of an antifoaming agent during the mixing step.

The present experimental study aims at valorizing the sludge, which is the waste from the cutting operation of marble blocks and evaluating the marble filler effects on the fresh and hardened properties of the SCCs compared to those of limestone filler. Two SCCs were prepared and tested: SCCM was manufactured using marble filler and SCCL was manufactured using limestone filler. The fresh properties of the two SCCs' mixtures were determined by slump flow, L-box, V-funnel, sieve stability, bulk density, and air content. Tests on hardened SCCs included compressive strength, homogeneity, and quality in terms of ultrasonic pulse velocity and durability against carbonation and water penetration. In addition, the experimental results were analyzed by the scanning electron microscope (SEM) and X-ray diffraction (XRD).

2. Experimental Program

The main purpose of this study is to highlight the possibility of using marble powder, obtained by grinding the sludge, as filler added to the SCC cementitious matrix.

The experimental program is composed of three main parts: the first part presents a description of the different steps in the preparation of the marble and limestone fillers, obtained by grinding the marble waste sludge and crushing limestone blocks, respectively. The second part presents the results of the identification of the properties of the marble filler, limestone filler, and other used materials.

The third part presents a comparison of the effects of the marble and limestone fillers on the fresh and hardened properties of the SCC. In order to achieve this, two SCCs were prepared and tested: SCCM was manufactured using marble filler and SCCL was manufactured using limestone filler. The order of testing in the fresh state was as follows: 1, slump flow test; 2, V-funnel test; 3, sieve stability test; 4, bulk density test; 5, L-box test and air content measurement [28]. The hardened properties studied included the compressive strength, the evaluation of homogeneity and quality in terms of ultrasonic pulse velocity, and durability against carbonation and water penetration. Finally, microstructural analysis was carried out on the prepared samples.

3. Materials and Experimental Methods

3.1. Materials

3.1.1. Marble and Limestone Powders

(1) Preparation of Powders. In this study, the limestone filler was prepared by crushing limestone blocks, and its characteristics are presented in the relevant section.

The marble sludge, which is a waste product from the marble cutting industry, was collected from local marble cutting plants. Grains of marble sludge clump together into lumps; thus, marble sludge grains were not homogeneously distributed throughout the mixture.

The procedure for preparing the marble powder consisted of the following steps:

(i) The marble sludge was air dried for two days

(ii) The dried sludge was crushed with a hammer to create small blocks

(iii) The marble sludge waste was dried in an oven at 80°C for 24 h to remove moisture

(iv) The dried sludge was ground to obtain a fine powder

(v) Finally, the marble filler was created by passing the powder through the desired sieve to filter the 63 μm particles

Characterization of the marble sludge filler was carried out using a number of experimental techniques in order to confirm its composition.

(2) Chemical Analysis of Fillers. The chemical analysis of the two fillers was performed using atomic absorption spectrometry (AAS), according to the requirements of NF EN ISO 15586 [29]. The chemical analysis results of the marble and limestone powders are presented in Table 1. The results indicate that the two powders are excessively rich in calcite, $CaCO_3 = 94.88\%$ for marble powder and 97.29% for limestone powder. In addition, the results indicate that the two fillers are devoid of all clay and organic matter. This last result was confirmed by measurement of calcium carbonate ($CaCO_3$), according to the requirements of NF P94-048 [30], which indicate that both limestone and marble powder samples contain 95% $CaCO_3$.

Finally, the results of the methylene blue test, performed according to the requirements of NF P94-068 standard [31], demonstrated that the methylene blue value (MBV) of the limestone and marble powders was 0.11 and 0.45, respectively. This indicates that the two powders do not contain any clay.

(3) Particle Size Analysis. Grain size distribution characterization of the two powders was carried out in two steps: the granulometry test by wet sieving, according to the standard NF P94-056 [32], for particles with diameters superior than 80 μm and granulometry test by the sedimentation method, according to the standard NF P94-057 [33], for quantifying fractions of particles with diameters lower than 80 μm.

Figure 1 shows the particle size distribution curves for the limestone and marble powders.

The results indicate that the Hazen coefficients of the marble and limestone powders are 6.33 and 10.6, respectively, and the curvature coefficients of the marble and limestone powders are 0.43 and 2.09, respectively. These results prove that the granular distribution of the two powders are effectively graded and graduated, and their masses consist of different particle size ranges.

(4) Physical Properties. The results presented in Table 2 indicate that the two powders have almost the same absolute density (2.69 g/cm^3 and 2.71 g/cm^3). On the contrary, the results show that the marble powder is excessively light compared to the limestone powder since the bulk density is 0.61 g/cm^3 for the marble powder and 0.90 g/cm^3 for the limestone powder.

The Blaine specific surface (BSS) values of the two powders were determined using the Blaine air permeability apparatus. According to the results presented in Table 2, the BSS values of the marble and limestone powders are 4405 cm^2/g and 9459 cm^2/g.

(4) Reactivity Tests. This section consists of determining whether the two powders have hydraulic properties with water. In order to achieve this, two reactivity tests were performed on two pastes, each prepared by 500 g of a type of powder added to 250 g of water in a thermostatically isolated flask. The reactivity tests consist measuring the temperature evolution of the two pastes.

Figure 2 shows that the temperature of the two pastes should be adjusted very slightly (from 18 to 20°C) for a period of more than 6 h. This result indicates that the two powders exhibit no hydraulic properties in the presence of water; therefore, the marble and limestone powders can be considered as inert components in concrete. In fact, the hydraulic reaction of SCC prepared by one of the two powders will not be modified or influenced.

In addition, the reactivity of the two powders with cement was verified by measure of setting time of cement paste, limestone-cement paste, and marble-cement paste. The setting time measurement test was carried out, using vicat apparatus, according to the standard NF EN 196-3 [36]. The curves depicted in Figure 3 indicate the setting times of the three pastes. The results demonstrate that the addition of the two powders to the concrete has almost no influence on the cement setting time. This confirms the result already demonstrated in the previous section: the marble and limestone powders are inert components in SCC and does not lead to much change in the phase composition of the resultant mix.

There many studies showing that the limestone and marble powder are inert or having low reactivity with the other components of pastes, concrete, or mortars. For example, the microstructure analysis, determined using scanning electron microscopy (SEM) and X-ray diffraction (XRD) on different pastes, of Aliabdo et al. [23] show that marble waste powder does not possess any pozzolanic activity. It will act as an inert and filler material rather than a pozzolanic one. Also, Khodabakhshian et al. [37] and Omar et al. [38] indicate also that marble powder is an inert or quasi-inert material, being noncementitious from hydraulic points of view.

In the same case, Liu and Yan [39] and Chaid et al. [40] indicate that limestone powder has no pozzolanic activity and then it was an inert cementitious addition. But it improves the performance of concrete.

(5) Results Interpretation. Table 3 presents the physical and chemical properties of the fillers according to the standard NF EN 206-1 [41]. Table 3 is reproduced from Benjeddou et al. [2] (under the Creative Commons Attribution License/public domain).

According to the obtained results, the physical and chemical properties of the two analyzed powders correspond to the cited standard criteria. The two tested powders can be added as fillers in SCC formulation.

3.1.2. Cement. The cement used was CEM I 42.5, in conformity with the standard NF EN 197-1 [42]. The physical and mechanical characteristics of this cement are displayed in Table 4.

Table 1: Chemical compositions of marble powder and limestone powder (%).

Component	LOI	CaO	MgO	SiO_2	Fe_2O_3	Al_2O_3	Na_2O	K_2O	SO_3	P_2O_5	$MgCO_3$	S	Moisture
Marble powder	42.60	52.28	0.50	3.00	0.39	0.14	—	—	—	—	1.04	0.03	0.20
Limestone powder	43.09	54.20	0.69	0.37	0.17	0.11	0.02	0.07	0.06	0.03	—	—	—

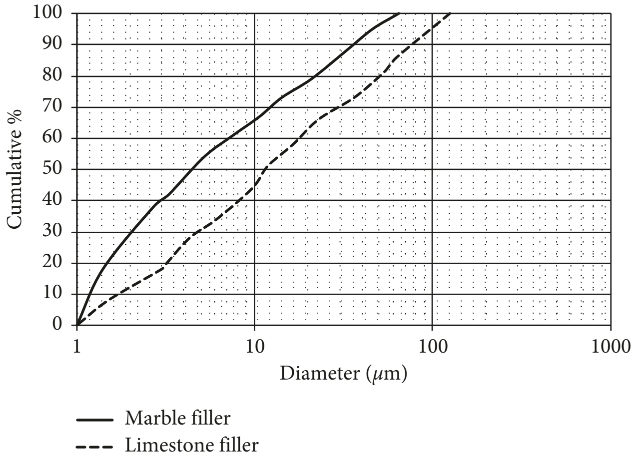

Figure 1: Particle size distribution curves of the limestone and marble fillers.

Table 2: Physical parameters of two powders.

Parameters	Particle size (mm)	Absolute density (g/cm^3)	Bulk density (g/cm^3)	Blaine specific surface (BSS) (cm^2/g)
Standard	NF P94-056 [32]	NF EN 1097-7 [34]		NF EN 196-6 [35]
Limestone filler	0/0.125	2.71	0.90	4405
Marble filler	0/0.063	2.69	0.61	9459

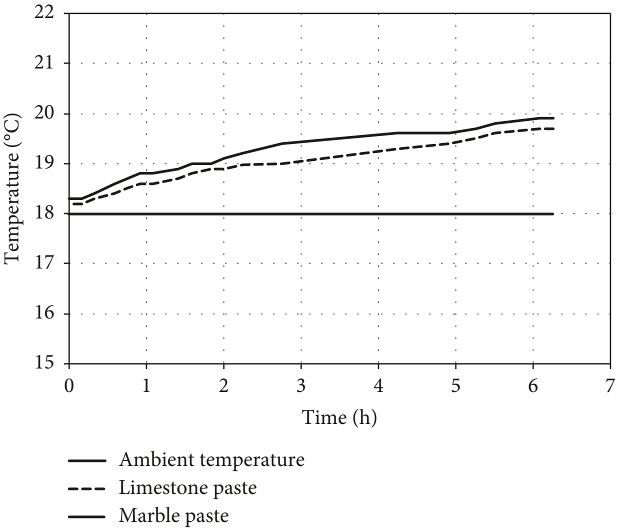

Figure 2: Paste temperature measurement.

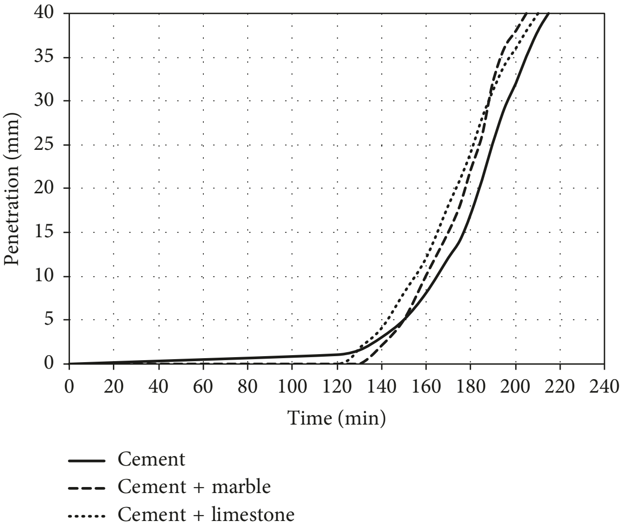

Figure 3: Measurement of setting time of the different pastes.

3.1.3. Aggregates. Natural sand 0/2, crushed sand 2/4, ravel 4/8, and gravel 8/12 were used as aggregates. The physical characteristics of these aggregates are displayed in Table 5.

3.1.4. Admixture. The employed admixture is a superplasticizer (SP); it is a new generation product based on chains of modified polycarboxylate, especially conceived for self-compacting concrete and certified in conformity with standard NF EN 934-1 [46]. The density of this SP is about 1.06, and its pH is between 4.5 and 6.5. In addition, this used SP has a dry extract percentage varying from 28 to 31%. Finally, the Cl^- and Na_2O_{Eq} percentages of this SP do not exceed 0.1 and 1%, respectively.

Table 3: Physical and chemical properties of fillers according to Benjeddou et al. [2].

Properties	BSS	$CaCO_3$ content	Methylene blue value	Sulphate content
Criteria	>2000 cm^2/g	>62%	<1.3 g/100 g	<0.15%

Table 4: Physical and mechanical properties of cement.

Properties	Absolute density (g/cm^3)	Bulk density (g/cm^3)	BSS (cm^2/g)	True class of strength (MPa)
Standard	NF EN 196-1 [43]	NF EN 196-1 [43]	NF EN 196-6 [35]	NF EN 196-1 [43]
Value	3.08	1.03	3100	44.5

Table 5: Physical characteristics of aggregates.

Aggregates	Bulk density (g/cm^3)	Absolute density (g/cm^3)	Absorption (%)	Finesse module	Equivalent of sand (%)
Standard		NF EN 1097-6 [44]		—	NF EN 933-8/IN1 [45]
Gravel 4/8	2.55	1.42	0.32		
Gravel 8/12	2.55	1.40	0.32		
Sand 0/2	2.42	1.47		1.85	91
Crushed sand 2/4	2.43	1.32	0.32	4.8	100

According to the saturation point obtained from the flow time-admixture dosage curve, in accordance with the standard NF P18-507 [47], the optimal SP/cement ratio is equal to 1.2% [48, 49].

3.2. Formulation of a Self-Compacting Concrete. This part of our experimental investigation consists of evaluating the marble filler effects on the SCC properties compared to those of the limestone filler. For this purpose, two SCCs were prepared and tested: SCCM was manufactured using marble filler and SCCL was manufactured using limestone filler.

The two self-compacting concretes, SCCM and SCCL, were formulated by using the excess paste method [50–52] and based on the requirements of the standard NF EN 206-1 [41]. It is an SCC of the SF2 class with a 28-day compressive strength of 30 MPa (SS30). For the slump class SF2, the slump test values must be between 66 and 75 cm. The dosage adopted for manufacturing the SCC30, using limestone or marble filler, is presented in Table 6.

3.3. Test Procedures. Two series of experimental tests were carried out on the two self-compacting concretes SCCM and SCCL: the first series of tests were carried out on the fresh state and the second were carried out in the hardened state.

All tests were performed according to international standards, and each experimental test was carried out in 3 different samples at different times, with different persons, and under somewhat different experimental conditions.

3.3.1. Experimental Tests in the Fresh State. The tests carried out on the fresh state of the two SCCs were the slump flow, L-box test, V-funnel, sieve stability, and bulk density tests as well as air content measurement. These rheological tests were carried out after a nominal interval of 15 min following the first contact of water with cement in the SCCM and SCCL [3].

(1) Slump Flow Test. This test is the simplest and most widely used test method for quantifying the SCC workability [53]. This test, measured according to the standard NF EN 12350-8 [54], indicates the horizontal flow of the SCC, and it is a good indicator of the self-compacting properties of the SCC.

(2) L-Box Test. This test is used to investigate, according to the requirements of NF EN 12350-10 [55], the SCC flow rate and passing ability in confined spaces. It measures the reached height of fresh SCC after passing through the specified steel bar gaps flowing within a defined distance.

(3) V-Funnel Test. This test gives, according to the requirements of NF EN 12350-9 [56], the flow time of SCC which is the period required for a defined volume of SCC to pass through a narrow opening. It can indicate the SCC filing ability, provided that blocking and/or segregation do not take place, and the flow time of the V-funnel test is related to the plastic viscosity to a certain extent.

(4) Sieve Stability Test. This test was carried out according to the requirements of NF EN 12350-11 [57]. A sample of 10 L of concrete was allowed to rest for 15 min; then, 2 L thereof was poured onto a 5 mm sieve from a height of 500 mm, and the sample percentage passing through the sieve was recorded. It has been reported that the variability of such test results is poor, particularly when the segregation is severe [58].

(5) Air Content Test. This test was required for checking air-entrained concrete, the testing method for which is provided in NF EN 12350-7 [59].

3.3.2. Experimental Tests in the Hardened State. The experimental tests carried out on the hardened state of the two SCCs are the compressive strength test, the pulse velocity test in order to evaluate the homogeneity and quality of the two SCCs, the measurement of the carbonation penetration

Table 6: Composition of 1 m^3 of SCCM and SCCL.

Cement (kg)	Filler (kg)	Sand 0/2 (kg)	Sand 2/4 (kg)	Gravel 8/12 (kg)	Gravel 4/8 (kg)	Water (L)	SP (kg)
400	100	720	180	720	180	200	4.4

depth test, the water penetration depth test, and microstructure analysis.

(1) Compressive Strength Measurement. 18 cylindrical specimens of 16×32 cm were prepared for each of the two SCCs, the formulations of which are presented in Table 6. Thereafter, the test pieces were crushed at the ages of 3, 7, 14, and 28 days in order to determine the compressive strengths of the two concretes at these ages.

(2) Pulse Velocity Test. The object of this method is to measure the velocity of the pulses of longitudinal waves passing through concrete in order to evaluate the homogeneity and quality of the tested concrete [60]. This is due to the effect of the concrete properties on the velocity of an ultrasonic pulse. Indeed, the pulse velocity increases or decreases as the concrete matures or deteriorates or changes with time.

The equipment consists essentially of an electrical pulse generator, a pair of transducers, an amplifier, and an electronic timing device for measuring the wave velocity between the transmitting transducer and the receiving transducer.

The ultrasonic pulse velocity measurements have been determined with the direct transmission mode (Figure 4) on three cylindrical specimens of 16×32 cm of SCCM, SCCL, and ordinary concrete at the age of 28 days, in accordance with the requirements of EN 12504-4 [61]. The ordinary concrete specimens were prepared with the same composition presented in Table 6 but without the addition of filler.

(3) Measurement of the Carbonation Penetration Depth. Durability is a major concern for concrete structures exposed to aggressive environments. Many environmental phenomena are known to significantly influence the durability of reinforced concrete structures. Carbonation is one of the major factors to cause structure deterioration.

The carbonation is a chemical phenomenon linked to the emission of carbon dioxide into the atmosphere. Indeed, carbonation is the reaction of carbon dioxide CO_2 in the environment with the calcium hydroxide $Ca(OH)_2$ in the cement paste. This reaction produces calcium carbonate $CaCO_3$ and lowers the pH to around 9 [62]. The carbonation front advances progressively from the facing, and then the protective oxide layer surrounding the reinforcing steel breaks down and corrosion becomes possible [62].

A simple test can be used to determine the depth of carbonation penetration. Phenolphthalein is prepared as a 1% solution in 70% ethyl alcohol, and the solution is sprayed onto the concrete surface which has been cleaned of dust and loose particles, according to requirement of NF EN 14630 [63]. Phenolphthalein turns noncarbonated concrete red and remains colorless in carbonated concrete as shown in Figure 5.

Figure 4: Ultrasonic pulse velocity test method.

The carbonation depth was measured at the age of 90 days on three cylindrical specimens of 16×16 cm of SCCM, SCCL, and ordinary concrete.

(4) Determination of Water Penetration Depth. The measurement of water penetration depth, of the ordinary concrete and the two self-compacting concretes SCCM and SCCL, is carried out according to the requirement of the standard NF EN 12390-8 [64] on three cylindrical specimens of 16×16 cm aged 28 days.

The apparatus used to determine the water penetration depths is presented in Figure 6. Once the specimens were assembled in the test cells, a water pressure of 5 bars was applied for 72 hours. The specimen was then split in half, perpendicularly to the face on which the water pressure was applied. As soon as the freshly exposed surface dried and the water penetration front can be clearly seen, the maximum depth was marked as presented in Figure 5. The maximum depth is called water penetration durability.

(5) Microstructure Tests. Microstructural analysis using SEM and XRD was carried out to compare the SCCM and SCCL properties in the fresh and hardened states [65–69]. Three control pastes, a cement-marble powder paste, and a cement-limestone powder paste were analyzed. The control pastes, denoted CP, MP, and LP, were made from 400 kg of cement, 400 kg of marble powder, and 400 kg of limestone powder, respectively. The other pastes, denoted CMP and CLP, were prepared from 400 kg of cement and 100 kg of marble powder and limestone powder, respectively. The cement-to-water weight ratio was equal to 0.26.

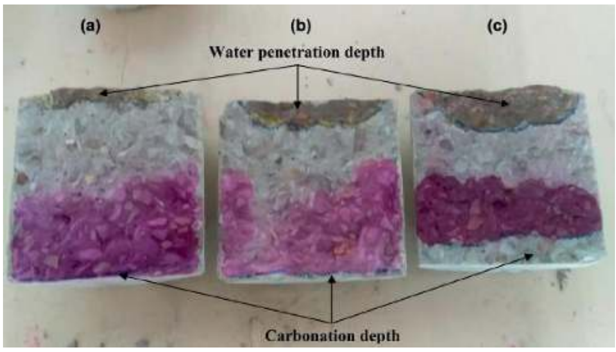

Figure 5: The measurement of carbonation penetration and water penetration depths: (a) SCCM; (b) SCCL; (c) ordinary concrete.

(a) (b)

Figure 6: Apparatus for determination of water penetration depth.

4. Results and Discussion

4.1. Effects of Marble and Limestone Filler on Rheology of SCC

4.1.1. Self-Compacting Properties. All the self-compacting concrete mixes with limestone and marble powder had a satisfactory performance in the fresh state. From the test results, it was concluded that the workability of fresh SCC was not affected for certain percentages of added marble powder. However, the mechanical properties of hardened SCC have degraded.

Previous research assessed the mechanical properties of SCCs where filler materials such as limestone powder, fly ash, and silica fume were used [18]. Literature results are in good agreement with the results obtained here. Additional aspects are addressed in the present study, such as the durability and rheology in the fresh state of the SCC as well as its microstructural characterization.

According to the results presented in Table 7, the slump flow values of the SCCM and SCCL are 55 cm and 67 cm, respectively. Indeed, the SCCM concrete is of flow class SF1, while SCCL is of flow class SF2. SCCL concrete has good self-compacting properties compared to SCCM. The low slump test value for the SCCM concrete is due to the increase in its viscosity.

The difference in the slump test values for the two concretes is a result of two main factors:

Table 7: Properties of two SCCs in fresh state.

Test	Standards value for SF2	SCCL	SCCM
Slump flow (cm)	66 75	67	55
L-box	h2/h1 ≥ 0.8	0.89	0.5
Sieve stability (%)	<15	0.6	2.6
Bulk density (kg/m^3)		2400	2440
V-funnel (s)	8 ≤ time ≤ 14	9.08	2.59
Air content (%)	≤5	2.7	1.2

(i) First, the water absorption ratio of the fine marble grain is higher compared to the limestone grain. It should be noted that the SSB value of the marble filler (9459 cm^3/g) is almost 2.5 times that of the limestone filler (4405 cm^3/g). Moreover, the particle size curves of the two fillers (Figure 1) indicate that the marble filler grain size is less than 63 μm, whereas that of the limestone filler grains are less than 125 μm. The particle size curves also demonstrate that the percentages of the grains with a size of less than 2 μm are 29% for marble grains and 12% for limestone grains. This difference in the two filler grain sizes makes the marble filler absorb more water, reducing the workability and consequently the viscosity of the final concrete. In fact, the slump flow value of the SCCM is less than that of the SCCL.

(ii) The second factor can be explained by the added amount of marble filler (100 kg). This quantity must be reduced, because it is actually necessary to add an equivalent quantity to that of the limestone filler in terms of the fine grain percentage. Finally, it can be concluded that the addition of such amount of marble filler to the SCC cementitious matrix affects the horizontal flow of this type of concrete.

The second test that identified the self-compacting properties of SCCS is the L-box test. According to the results presented in Table 7, the h2/h1 ratio of SCCM and SCCL are 0.5 and 0.89, respectively. According to the requirements of NF EN 12350-10 [55], only SCCL exhibits an acceptable self-compacting ability to be classified as SF2 flow class, since the ratio is higher than 0.80. This is a consequence of the increase in the SCCM viscosity due to its large specific surface. It is also a result of the high density of the SCCM (2440 kg/m^3) compared to that of SCCL (2400 kg/m^3). Indeed, a higher SCC density results in a slower horizontal flow in the L-box and therefore a low h2/h1 ratio.

4.1.2. Self-Leveling Properties. The self-leveling properties of the two SCCs were identified by the V-funnel test. According to the requirements of NF EN 12350-9 [56], the flow time must be between 8 and 14 s. Nonuniform concrete flow from the funnel suggests a lack of segregation resistance.

A lengthy flow time may be a result of high paste viscosity, high interparticle friction, or flow blockage by coarse aggregates. The V-funnel results are related to the concrete viscosity, passing ability, and segregation resistance; therefore, the test results may not identify the true cause of a lengthy flow time [70].

The results displayed in Table 7 indicate that the flow times of the SCCL and SCCM are 9.08 and 2.59 s, respectively.

The rapid flow time of the SCCM is essentially a result of its density. In fact, a higher SCC density leads to a faster vertical flow. The difference in vertical flow between the two concretes can also be explained by the difference in the paste volume. The SCCM has a lower paste volume than the SCCL due to the differences in grain sizes and finesse of the two fillers. Finally, it can be concluded that concrete with a smaller paste volume has a faster flow velocity. Moreover, the addition of marble filler to SCC increases its vertical flow.

On the other hand, the results presented in Table 7 indicate that the two tested concretes exhibit a high resistance to segregation, due to their milt loss percentages of less than 15% (NF EN 12350-11 [59]).

4.1.3. Air Content. Various adjustments are made to provide the true air content, and the air content percentage of an SCC should not exceed 5%.

The results presented in Table 7 indicate that the two concretes, SCCL and SCCM, exhibit air content percentages equal to 2.7% and 1.2%, respectively. These values demonstrate that the two formulated SCCs have maximum compactness, without vibration or clamping.

The low air content percentage of the SCCM is a result of its compactness, which is a consequence of the granulometry of the marble filler used. This filler exhibits a high percentage of fine grains: 29% and 66% of the grains have sizes smaller than 2 μm and 10 μm, respectively. This marble filler fineness leads to the majority of the grains being interposed between the cement grains, and later the compactness increasing, which reduces the air content.

The results show that as the air content reduces, the fresh concrete density increases. This is due to the grain sizes of the two fillers which make the marble filler consume water in terms of absorption and then make the SCC paste denser. Consequently, the workability of SCC was affected by the air content percentage. Thereafter, an increase in density conducts to an increase in viscosity and then to a decrease in workability [71]. Indeed, a higher SCC density results in a lower slump test value and in a slower horizontal flow. In addition, a lower air content percentage with the same water amount gives a lower segregation resistance [70].

4.2. Effects of Marble and Limestone Filler on Compressive Strength of SCC. This part of the study involves comparing the compressive strength of the concrete made with the marble filler to that of the concrete made with the limestone filler. The purpose of this comparison is to evaluate the effects of the marble filler on the SCC hardened properties, and more precisely, its compressive strength.

From the results displayed in Table 8, it is noted that the compressive strength values of the SCCM and SCCL are very close at both young age and middle age. The compressive strengths at 28 days are higher than the desired compressive strength of 30 MPa (39 MPa and 41 MPa for SCCL and SCCM, respectively).

Furthermore, from the obtained results, high compressive strength values were observed at a young age for both concrete types (18 MPa and 21 MPa SSCL and SCCM, respectively, at the age of 3 days). This increase is mainly owing to the SP used, which provides the secondary function of hardening acceleration.

Regarding long-term resistance, it is noted that SCCM exhibits a higher compressive strength than the SCCL (41 MPa vs 38 MPa). This can be explained by the results of the tests carried out on the two concretes in the fresh state. The SCCM is less manageable than the SCCL, and therefore its water-cement ratio was reduced, which allowed for its compressive strength to increase.

However, the density value as well as air content percentage in SCCM allows us to conclude that this concrete is more compact than the SCCL, and consequently, its compressive strength is higher.

Likewise, as indicated in the previous section, the granulometry as well as fineness of the marble filler plays a very important role in improving the compactness in the granular and cement matrix.

Finally, the small difference in compressive strengths of the two studied concretes is a result of the differences in the fineness, particle size, and fine grain percentage of the two used fillers.

Table 8: Compressive strengths of four SCCs at different ages.

Concrete type	Compressive strength (MPa)					
	1 day	2 days	3 days	7 days	14 days	28 days
SCC (limestone)	11	16	18	29	34	38
SCC (marble)	12	18	21	30	36	41

4.3. Effects of Marble and Limestone Filler on Homogeneity and Quality of SCC. The homogeneity and quality of the SCCM and SCCL were evaluated by measuring ultrasonic pulse velocity. The velocity of an ultrasonic pulse is influenced by the properties of concrete. Indeed, the pulse velocity increases or decreases as the concrete compaction and the voids in concrete change and thereafter the homogeneity and quality change. The classifying of concretes are excellent, good, doubtful, poor, and very poor for (4500 m/s and above), (3500–4500 m/s), (2500–3500 m/s), (1700–2500 m/s), and (1700 m/s) and below velocity values, respectively [72, 73].

The results show that the ultrasonic pulse velocity values for the ordinary concrete, SCCL, and SCCM are 3820, 4535, and 4746 m/s, respectively. However, based on the pulse velocity values, the ordinary concrete presents a good homogeneity and quality but the two self-compacting concretes SCCL and SCCM present excellent qualities and then excellent homogeneities. This is due to the increase of the velocity value with the decrease of the concrete porosity. This can be deduced from the air content percentages of the tested concrete which is very low for SCCL and SCCM (2.7 and 1.2%, respectively) compared to that of the ordinary concrete which is about 8%. It can be clearly noted the beneficial use of marble powder and limestone powder as fillers in the SCC composition.

On the other hand, the higher velocity value of SCCM compared to then of SCCL is also due to the low air content percentage compared to then of SCCL (Table 7) which led to increase in speed of ultrasonic pulse velocity wave. In addition, the reason may be due to the lower porosity of SCCM due to the higher Blaine specific surface of marble powder, 9459 cm^2/g, compared to that of limestone powder, 4405 cm^2/g.

Higher specific surface of filler resulted in lower porosity hence high compressive strength (Table 7).

4.4. Effects of Marble and Limestone Filler on Durability of SCC

4.4.1. Carbonation Penetration Depth. The results show that the average carbonation penetration depths of the ordinary concrete and the two self-compacting concretes SCCL and SCCM are 12, 4, and 1 mm, respectively. It can be seen that the carbonation penetration depth of ordinary concrete is very higher than of the two SCCs. This is due to the higher volume of cement paste of SCCL and SCCM compared to the ordinary concrete. Indeed, the porosity of concrete decreases when increasing the cement paste volume. It can be concluded that the carbonation depth increases with an increase of concrete porosity. Also, incorporating filler in self-compacting concrete tends to decrease the porosity and volume of capillary pores in concrete and significantly affects the carbonation depth.

In addition, the results show that the carbonation depth of SCCM is lower than the carbonation depth of SCCL. This is can be explained by that when specific surface of filler increases, porosity is decreased which results in decrease of carbonation penetration depth.

Finally, it can be concluded that the porosity affects the carbonation penetration depth of concrete and then its durability will be influenced. Also, the incorporation of mineral filler in self-compacting concrete increases the carbonation resistance of SCC compared to the ordinary concrete and their durability increase [73].

4.4.2. Water Penetration Depth. Figure 5 shows how the penetration depth is taken into the one test specimen of each tested concrete. The results show that the average water penetration depth values of the ordinary concrete, SCCL, and SCCM are 22, 11, and 7 mm.

Based on the obtained water penetration depth values, the ordinary concrete has the highest value of water penetration depth, while the concrete SCCM has the lowest one. This behavior also depends on the porosity of the concrete. Indeed, the water penetration depth increases with the concrete porosity.

Finally, there is a linear relationship between the water penetration depth and the carbonation penetration depth of concretes. Indeed, the two penetration depths increase with the concrete porosity. In addition, it can be concluded that the two self-compacting concretes SCCM and SCCL have a high durability compared to the ordinary concrete [74].

4.5. Microstructure Analysis. Figure 7 presents the scanning electron microscopy (SEM) micrographs and energy-dispersive X-ray spectroscopy (XRD) patterns of cement paste at 28 days. According to the presented results, the hydrating in time of cement paste is composed mainly of hydrated silicates of calcium (CSH) phase accompanied with a hydration products of calcium aluminate (ettringite) and calcium hydroxide (portlandite). The obtained portlandite creates massive, hexagonal crystals.

In addition, researches have shown that CSH is the main component and the most important factor affecting the mechanical strength of hardening cement paste [66, 68].

Figures 8 and 9 present SEM images and XRD patterns of marble powder paste and limestone powder paste, respectively, at 28 days. According to the chemical analysis presented in Table 1, the major component of marble powder and limestone powder is the calcium oxide which demonstrate the carbonate nature of these two powders. XRD patterns of marble and limestone pastes show that as expected calcite is the main crystalline mineral both in marble and in limestone pastes, with significant peaks. In addition, according to Figures 8 and 9, quartz is also identified in very low concentration for the two pastes.

Thereafter, according to the results of Table 1 and Figures 8 and 9, it is clear that marble and limestone powders do not possess any pozzolanic activity. Consequently, marble

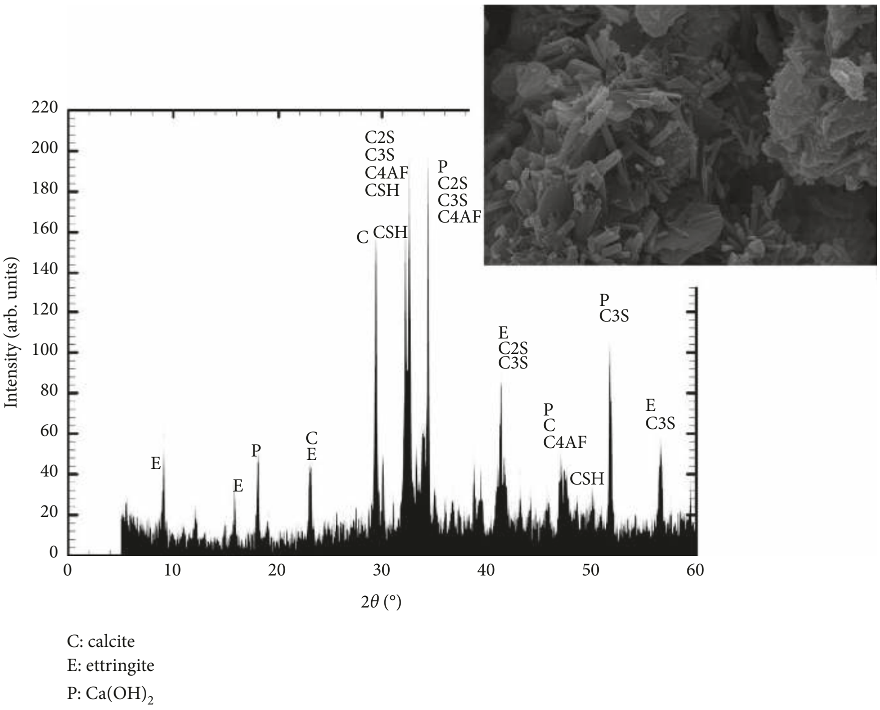

Figure 7: SEM micrographs and XRD pattern of cement paste.

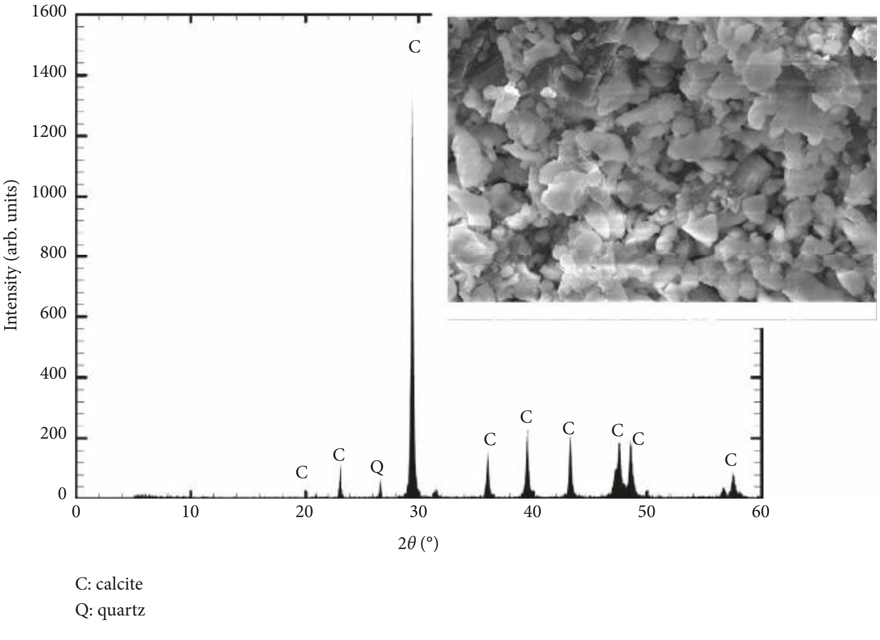

Figure 8: SEM micrographs and XRD patterns of marble powder paste.

and limestone powders can be considered as inert fillers, and so the hydraulic reaction of SCCM and SCCL will not be modified or influenced. This last result confirms the obtained results presented in Section 3.

SEM micrographs and XRD patterns of cement-marble paste (CMP) and cement-limestone paste (CLP) at 28 days are presented in Figures 10 and 11, respectively.

According to SEM micrograph of cement paste (Figure 7) and SEM micrographs of CMP and CLP (Figures 10 and 11), it was remarked that CMP and CLP specimens are denser and less porous than cement paste specimen. This result indicates the refinement of the pore structure in CMP and CLP compared to the cement paste. In addition, it was remarked that the microstructure of cement paste is a highly porous with irregular micropores in cement matrix.

It was remarked also that CMP paste is more dense and less porous than CLP paste and even the concrete SCCM is more denser and less porous than SCCL concrete.

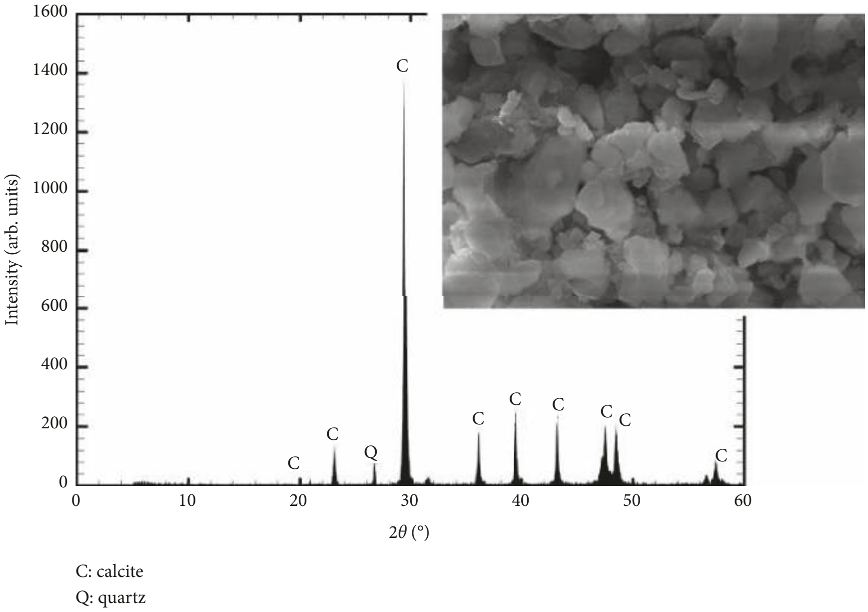

Figure 9: SEM micrographs and XRD patterns of limestone powder paste.

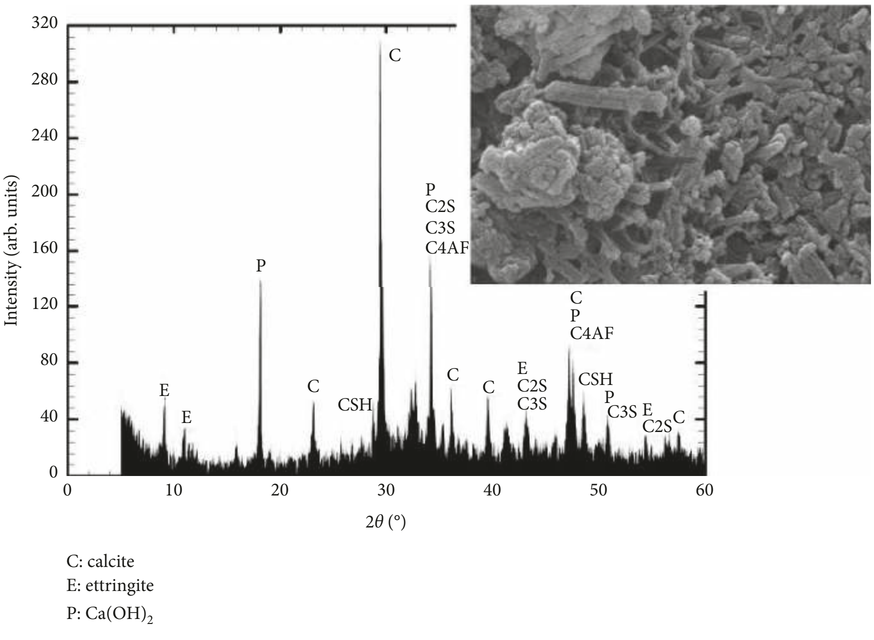

Figure 10: SEM micrographs and XRD patterns of cement-marble powder paste "CMP."

Consequently, the experimental values of the bulk densities and the air content percentages of the two SCCS, presented in Table 7, are confirmed. Indeed, the rheological properties of SCC and more precisely the self-compacting and the self-leveling properties depend on the fineness of the used filler. This confirms the obtained experimental data of slump test, flow time, bulk density, and L-box (Table 7).

In addition, the comparison of XRD patterns of the five pastes (Figures 7–11) indicate that there is no important difference in calcium hydroxide (CH) contents for all pastes. Also, the comparison shows that there is no change in the phase composition qualitatively when incorporating both marble powder or limestone powder in cement paste. As a conclusion, marble and limestone powders are inert materials, and consequently, they do not lead to much change in the phase composition of the resultant mix of SCCM and SCCL.

XRD patterns show also that cement pastes and CMP and CLP pastes have the same portlandite peak. In addition, the very fine needles of ettringite of CMP and CLP are less than those of paste with cement alone. Also, the presence of fine needles in CMP is lesser than for CLP. This is due to the high fineness of marble powder compared to that of limestone powder.

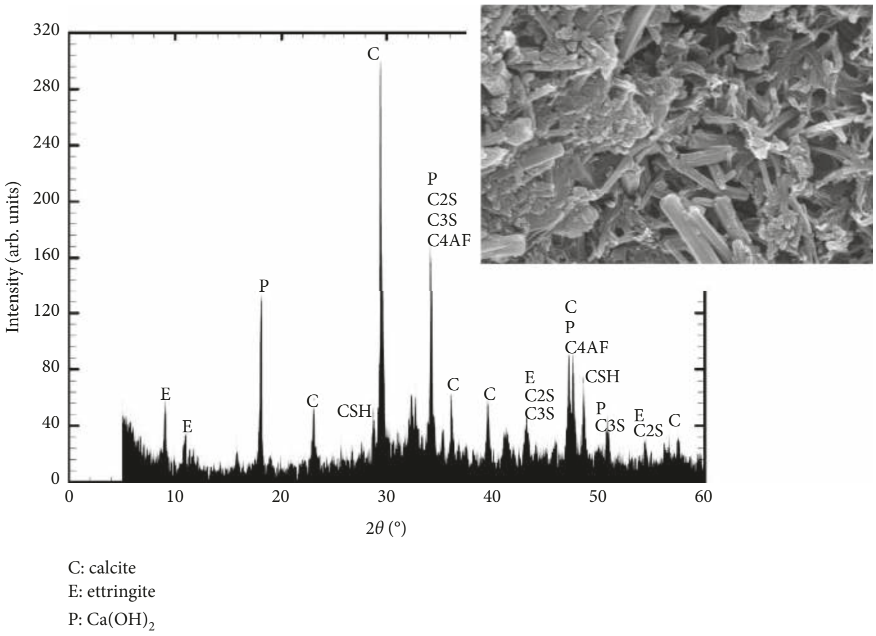

Figure 11: SEM micrographs and XRD patterns of cement-limestone powder paste "CLP."

Finally, the use of marble powder as a filler in the SCC composition increases intruded pore volume, reduces percentage of fine pores, and then increases compressive strength of the SCCM compared to these of SCCL. This is due to the fundamental relation between compressive strength and the void space and porosity distribution of concrete [23]. This last result confirms the experimental compressive strength values, presented in Table 8, of the two concretes SCCM and SCCL at different ages.

5. Conclusion

As a result of this experimental study, the following conclusions can be made:

(i) The powder obtained from the sludge of marble blocks can be used as marble filler added to the SCC cementitious matrix.

(ii) The two concretes, SCCM and SCCL, exhibit satisfactory self-compacting properties in the fresh state, thanks to their adequate filling capabilities and passing abilities.

(iii) The marble filler has a significant effect on fresh concrete properties since it improves the vertical flow and increases the air content.

(iv) The two fillers have significant effects on the compressive strength, which was higher in the SCCM.

(v) A strong correlation was observed between the fresh and hardened SCC properties with the marble filler.

(vi) The speed of the ultrasonic pulse wave is higher in the SCCM due to its low air content, compared to the SCCL.

(vii) Porosity affects the carbonation penetration depth of SCC and its durability. Additionally, the incorporation of mineral filler in SCC increases its carbonation resistance and its durability.

(viii) There is a linear relationship between the water penetration depth and the carbonation penetration depth of concretes. Indeed, the two penetration depths increase with the concrete porosity. In addition, SCCM and SCCL have a high durability compared to the ordinary concrete.

(ix) XRD patterns indicate that marble and limestone powders are inert since they did not lead to a change in the composition of the resultant mix of SCCM and SCCL.

(x) SEM micrographs show that the use of marble powder as a filler in the SCC reduces porosity and thus increases the compressive strength especially in the SCCM compared to SCCL.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

This project was supported by the Deanship of Scientific Research at Prince Sattam bin Abdulaziz University under the research project no. 2017/01/7296.

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