Feasibility of marble powder and calcined bentonite in SCM as partial substitution of cement for sustainable production

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Abstract
Global cement production has increased dramatically in recent years, and it is now the third largest source of carbon dioxide emissions. The use of binary and ternary cement in production of new binder leads to more debates on the reduction of CO_2 emissions. The main goal of this paper is to investigate the feasibility of marble powder (MP) and calcined bentonite (CB) as partial substitution of cement. In this experimental study, nine mixtures of self-compacting mortar (SCM) were prepared by substituting the cement with MP and CB in ternary blend systems. SCM was tested at fresh state with mini slump flow and mini V-funnel flow time. At hardened state, compressive strength, density, water absorption and ultrasonic pulse velocity (UPV) were measured. The results indicated that the inclusion of MP and CB mixtures improved the flowability of SCM. The use of ternary blended cements based on CB and MP increased the mechanical properties with a content ranging from 15 to 20%. This result has a positive impact on reducing the amount of CO_2 emitted into the atmosphere by combustion cement plants, resulting in a much cleaner environment.
Keywords: self-compacting mortar, calcined bentonite, marble powder, ternary cement
Kulcsszavak: öntömörödő habarcs, kalcinált bentonit, márványpor, háromkomponensű cement

1. Introduction

The cement industry consumes a considerable amount of energy [1] and therefore produces a significant quantity of greenhouse gas [2]. This gas results from the combustion of fuel and the decarbonation of $CaCO_3$, contained in the raw material, during the clinkerization process. Ternary cements are one of the alternative solutions, in which clinker in Portland cement is partially replaced with a combination of two supplementary cementing materials (SCM) in order to preserve or improve the performance of the cement [3]. Limestone Calcined Clay Cement or LC3 is a ternary binder system consisting of limestone and calcined clay used in conjunction to make a composite cement. in these ternary cement systems, a synergetic effect is developed which aluminate from the metakaolin reacts with calcite and enhances the formation of carboaluminate phases [4, 5]. Similarly, marble stone calcined clay cement (MC3) is produced and checked the physical and chemical properties of these cement [6]. LC3 has attracted a great deal of attention within recent years according to the even higher clinker replacement levels and achieve properties comparable to that of the conventional concrete [7]. Ergün [8] reported that the concrete specimens containing 5% of MP and 10% of diatomite as cement replacement had higher compressive strength than that of the control concrete. The results that concrete made using 5% of MP and 10% of Diatomite as cement replacement had highest compressive and flexural strength. Another anterior investigation [9] pointed out that an improvement in the workability of self-compacting concrete (SCC) with the use of natural pozzolan and marble powder, while that the use of 5% MP and 5% of pozzolan, leads to obtain better compressive strength at 28 days. Baoguo et al [10] have noticed that the combination between 10 % of marble powder (MP) and 3% of nano-silica, could partially offset the negative effect caused by each other and obtain acceptable hardened properties of mortars. On the other hand, recent works [11, 12] have allowed to know the usefulness of bentonite clay calcination which offers an excellent pozzolan, considered as judicious solution to minimize the use of cement in concrete.

The main goal of this paper is to investigate experimentally the effect of low carbon cement consists of MP and CB as ternary cement on fresh and hardened properties of SCM for sustainable production.

2. Materials and experimental program

2.1 Materials

For all mortar mixtures, an Ordinary Portland Cement CEM I 42.5 was used. MP is a waste resulting from cutting, shaping and lustrating of the marble stones. CB was obtained by thermal treatment of a local bentonite clay at 800 °C in an electrical furnace for 3 h. The calcination procedure was chosen according to a previous work [12]. The chemical composition and physical properties of raw materials are given in *Table 1*. According to this table, the chemical composition of MP is mainly CaO, which is more than 56%. While Al_2O_3 and SiO_2 are the most dominant compositions in CB (>80%).

The crystalline phases in MP and CB were analyzed by XRD and shown in *Fig. 1*. It shown that MP is mainly composed of calcite with some traces of the quartz and dolomite. For CB, quartz and illite were accompanied with albite, microcline and high temperature crystalline phases such as mullite in addition to slight presence of anatase. *Fig. 2* illustrates the particle size distribution of both of MP and CB compared to cement.

As fine aggregate, a river sand was used with fineness modulus, specific gravity and water absorption of 2.43, 2.68 and 0.79 respectively.

A polycarboxylate superplasticizer (SP) with a specific gravity of 1.07 and a solid content of 30% was used as a high range water reducer in SCC mixtures to achieve the desired flowability.

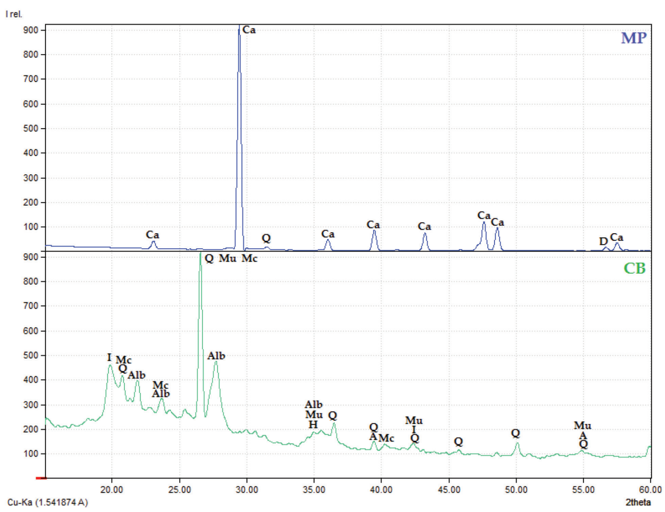

Fig. 1 XRD Analysis of calcined bentonites
(Ca :Calcite, D:Dolomite; Q: quartz; Alb: Albite; Mc: Microcline; Mu: Mullite; I: Illite; H: Hematite; A: Anatase)
1. ábra Kalcinált bentonitok XRD analízise
(Ca: kalcit, D: dolomit; Q: kvarc; Alb: albit; Mc: mikroklin; Mu: mullit; I: illit; H: hematit; A: anatáz)

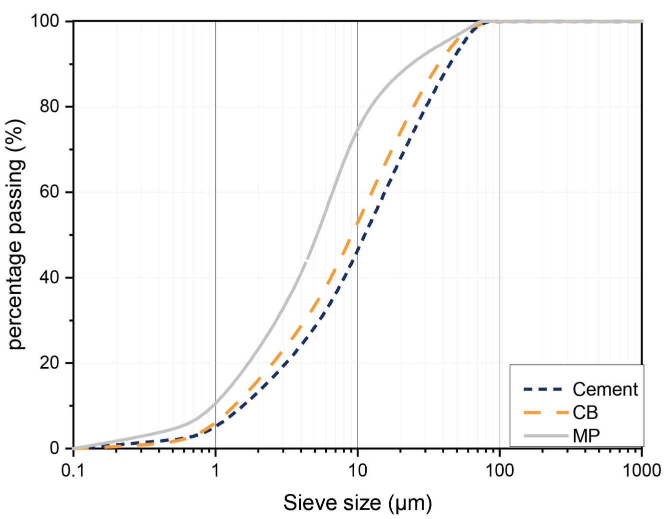

Fig. 2 Particle size distribution of cement, MP and CB
2. ábra A cement, MP és CB szemcseméret eloszlása

Chemical composition	Cement	CB	MP
SiO_2	20.87	63.04	0.51
Al_2O_3	4.17	17.15	0.13
Fe_2O_3	5.65	3.17	0.06
CaO	62.87	1.93	56.24
MgO	1.53	4.12	0.1
K_2O	0.58	1.47	0.01
Na_2O	0.15	2,64	0.45
SO_3	2.29	0.38	0.01
Loss ignition	-	-	42.49
Physical properties			
Specific gravity (g/cm³)	3.08	2.35	2.6
Blaine Fineness (cm²/g)	3344	4371	5243

Table 1 Chemical composition and physical properties of cement, CB and MP
1. táblázat A cement, MP és CB kémiai összetétele és fizikai tulajdonságai

2.2 Mix design

A control mix without any substitution, in addition to eight mix designs with two groups of self-compacting mortar made including MP and CB are performed, in the first group the MP content is fixed at level of 10% by weight of cement, while the CB was replaced in proportions of 5, 10, 15 and 20%. In the second group, the BC content is fixed at the rate of 15% by weight of cement, while the MP was replaced in proportions of 5, 10, 15, and 20%.

The self-compacting formulation criteria proposed by the Japanese Okamura and Ouchi [13] was used for all SCM mixtures, with some modifications regarding the quantity of sand in the mortar (the ratio of sand/mortar (S/M) was fixed at 0.5), the water/binder ratio (W/B) (kept constant at 0.4), the dosage of superplasticizers (SP). The SCM mixtures proportions are shown in *Table 2*.

The mixing procedure consisted of mixing the powder and sand for 15 s in dry condition to homogenize the mixture for half a minute before adding 70% the water needed with a regular flow for 1 min, and then adding the remaining 30% of water containing SP for another 1 min. The mixing procedure continued for 5 min.

2.3 Experimental setup

The filling ability of SCM was evaluated by the mini-slump flow test. The apparatus consists of a mould in the form of a frustum of cone, 60 mm high with diameter of 70 mm at the top and 100mm at the base. The cone was filled with mortar and the lifted to allow the mortar to spread, and then subsequent diameter of the mortar is measured. The spread mortar was visually checked for any segregation or bleeding [14].

The passing ability of SCM was assessed by a V-funnel flow test [13], where funnel is filled with 1.1 liters of mortar. The V-funnel flow time is the time between opening the orifice and the first daylight appearing when looking vertically down though the funnel [14].

The average compressive strength of six specimens of 40×40×160mm at ages of 3, 7, 28, 56 and 90 days was determined. The ultrasonic pulse velocity (UPV) is measured in accordance with NF P18-418 standard [15] . The density of

Mix. ID	W/B	Water (kg/m³)	Binder (kg/m³)	Binder			Sand (kg/m³)	SP (%)
				Cement (kg/m³)	MP (kg/m³)	CB (kg/m³)		
Control	0.4	275	690	690	0	0	1325	0.8
10MP5CB				586.5	69	34.5		0.8
10MP10CB				552	69	69		0.8
10MP15CB				517.5	69	103.5		0.8
10MP20CB				483	69	138		0.8
15CB5MP				552	34.5	103.5		0.8
15CB10MP				517.5	69	103.5		0.8
15CB15MP				483	103.5	103.5		0.9
15CB20MP				448.5	138	103.5		1

Table 2 SCM mixture proportions
2. táblázat Az egyes keverékek összetétele

hardened SCM is determined in accordance with NF P18-435 standard [16]. While, the water absorption test was carried out in accordance with ASTM C642-97 [17].

3. Results and discussion

3.1 Fresh properties

The effect of MP and CB on the fresh properties of mortars is given in *Fig. 3*. As can be seen from this figure, the slump flow diameters of all obtained mixtures are acceptable according to the ranges suggested by Domone and JIN [14]. However, it is noticeable that the slump flow of the first group is affected by the CB content, whereas the flowability of mortars decreased as the CB amount increased. This is caused by the fine particles of CB, which have higher surface areas. This result is consistent with several studies [11, 12, 18], which reported the beneficial influence of MP, ensuring a quantity of water destined to the deflocculation and dispersion of cement in one hand. In the other hand CB particles without the need for an additional quantity of water or SP. Khushnood et al [19] concluded that, higher water and superplasticizer demand are required, when CB is used in the self-compacting system. For the second group it is clearly that increasing the quantity of MP lead to an improvement in workability of mixtures. Belaidi et al [9] attributed this gain in workability to the dilution effect generated by marble powder, which proves its effective role in minimizing SP consumption. Ma et al [10] pointed that MP decreased the water requirement and increased the fluidity of mixtures.

In terms of passing ability through the V-funnel, it is clearly that all the values obtained are inside the targeted domain (2 to 10 s). The results obtained show an increase in flow time as the CB content increases for the first group. Contrary to second group, where the incorporation of MP reduces the viscosity of the mixtures, which translates into low V-funnel flow times. Boukhelkhal et al [20] reported similar results in which for the same SP dosage, the use of MP in SCC lead to a reduction in flow time. Laidani et al [12] indicated that incorporating CB increased the flow time of SCM. Khushnood et al [19] pointed out that the increase in V-funnel flow time and T25 of self-compacting pastes containing bentonite, is probably due to the high friction between particles offered during the flow. Belaidi et al [9] concluded that, at a constant W/B ratio and SP content, the use of both pozzolan and MP by substitution to cement has no negative effects on the workability of SCC.

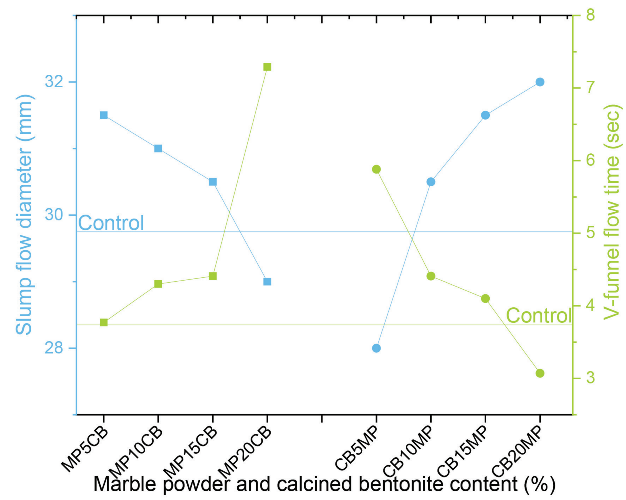

Fig. 3 Slump flow test and V-funnel flow time of SCM made with MP and CB
3. ábra MP és CB alkalmazásával készült cement kiegészítő anyagok roskadásvizsgálati eredményei és V-tölcséres folyási idői

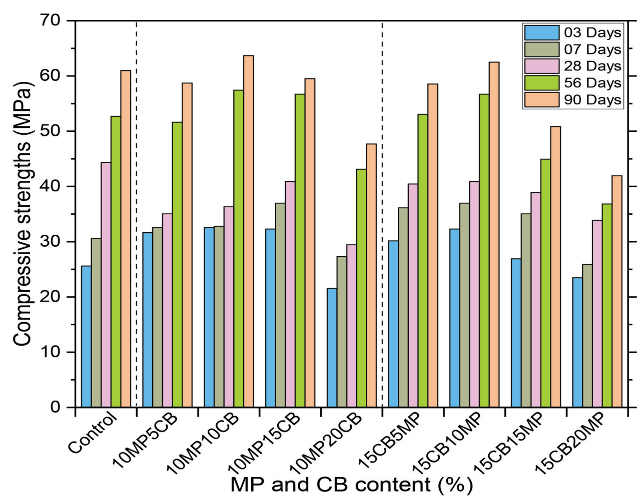

Fig. 4 Compressive strengths of SCM made with MP and CB
4. ábra MP és CB alkalmazásával készült cement kiegészítő anyagok nyomószilárdsági eredményei

3.2 Compressive strength

The compressive strength of all SCM mixes with varying quantities of CB and MP is determined after 3, 7, 28, and 56 and 90 days of water curing and the test results are presented in *Fig. 4*. The figure indicates that the compressive strength increase substantially in the first days of hydration when cement is substituted with MP and CP (3 and 7 days) this is due to the nature of the MP which classified to the family of quasi inert fillers [21]. This filler has a certain physicochemical activity, which contributes to the acceleration of the hydration of C_3S with the component $CaCO_3$ which consequently improves strength at young ages [22].

As the hydration reaction progress (56 and 90), the strength of mortar mixes starts increasing with the presence of CB. The 56 days compressive strength of control mortar increases from 52 MPa to 57 for mortar mixes made with 10% of CB and 10% of MP similar increases to 56 MPa, for mortar mixes made with 15% of CB and 10% of MP. Moreover, the 90days compressive strength of respective mortar mixes increases from 61 MPa to 64 MPa and 63 MPa, which are 5%, and 3.3% higher than the control mix, this is explained by the pozzolanic reaction of CB with $Ca(OH)_2$ and the acceleration of the hydration of OPC [11, 23]. However the combination of two additions reduced slightly the compressive strength when the level of 30% of additions of seems ternary mixes, this phenomenon is due to the effect of the high rate of fine particles which absorb the water necessary for the hydration of the cement which improves the compactness of mixes [24].

3.3 UPV and density

The effects of MP and CB on density and ultrasonic pulse velocity are presented in *Fig. 5* and *Fig. 6* respectively. From these figures it can be generally revealed that the trends in density and UPV are similar to those in compressive strength, which confirm the concurrent development and the strong correlation between density, compressive strength and the sound transmission velocity [20, 25]. It can be also seen that the density and UPV values of the first group increased with the increase of MP proportion until a maximum value (corresponding to the rate of 10% of both MP and CB) and then decreased. This can be attributed to the fact that calcite from MP reacts with aluminate from CB and contribute to the formation of carboaluminate that helps in providing more compactness [4]. Moreover, MP fills the void between cement particles owing to its higher fineness. Once the space between cement particles is completely filled, MP then began to occupy the place of cement particles, and therefore the density and the sound transmission velocity decreases [26-28]. It should be noted that the lower specific gravity of MP also contributes to a decrease in UPV and density of hardened SCM [20, 28].

Test results of the second group indicate that SCM mixtures with 15% of CB and 10% of MP show the maximum density and UPV values. Beyond this optimum, the density and UPV decrease. The synergistic effect of CB and MP accelerate the internal structure development by the formation of additional hydrates, which leads to an increase in density and sound transmission velocity. Furthermore, the total porosity decreases because of to the filling effect of CB inside the skeleton of SCM. Beyond a 15% of CB substitution, there was a reduction in density and UPV and that is due to the fact that CB fine grains began to take the place of cement particles, this led to a reduction in compactness at high amounts of substitutions. Moreover, the higher CB replacements could also absorb the water demand destined for hydration process [12].

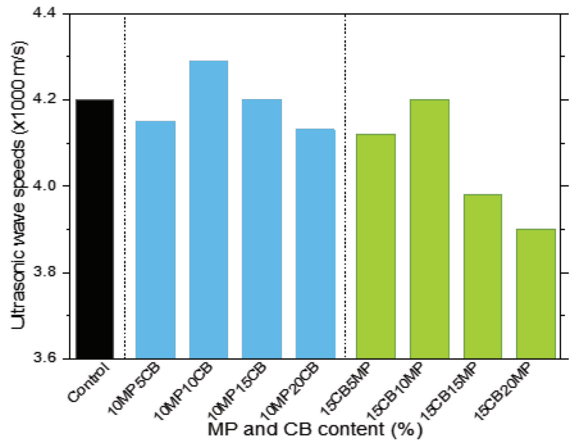

Fig. 5 UPV of SCM made with MP and CB
5. ábra MP és CB alkalmazásával készült cement kiegészítő anyagok ultrahang impulzussebességei

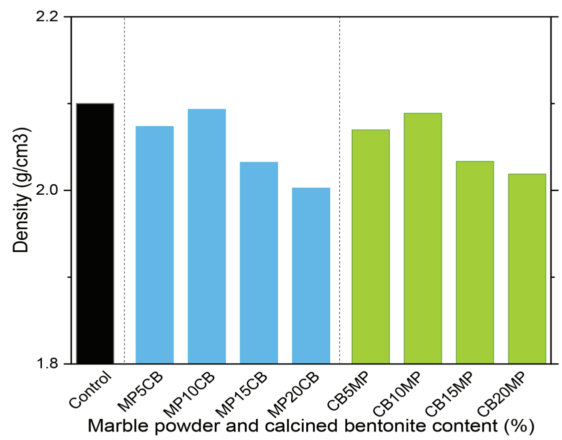

Fig. 6 Density of SCM made with MP and CB
6. ábra MP és CB alkalmazásával készült cement kiegészítő anyagok sűrűségi adatai

3.4 Water absorption

The results of water absorption by total immersion of all mixtures measured at 28 days are illustrated in *Fig. 7*. According to this Figure, its noticeable that the water absorption of both groups are increased by increasing the rate of substitution. In addition, all absorption values are greater than that of control mixture (6.9%).

As optimum, the lowest water absorption value is registered for the mortar 10MP5BC (7.1%), beside that the first group had a lower absorption potential than the second group. This may be due to the filling role of MP, where its grains fill the porous system because they are finer than the cement and CB, in addition to the production of additional C-S-H generated during the pozzolanic reaction between CB and MP. It was noted, that all tested SCM have low water absorption, which is less than 10%.

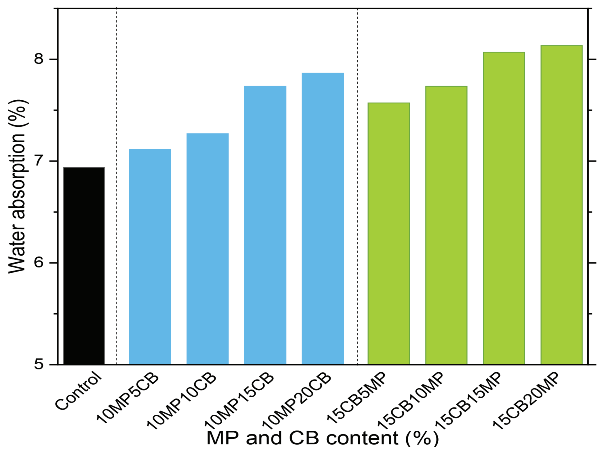

Fig. 7 Water absorption of SCM made with MP and CB
7. ábra MP és CB alkalmazásával készült cement kiegészítő anyagok vízfelvétele

4. Conclusions

Based on the experimental results and the analysis performed, the following conclusions can be drawn:

1. The increase in CB affected negatively the fresh properties and rheological parameters of all mixtures.
2. The use of MP in CB mixtures as ternary cement have shown high fluidity and viscosity of SCM; this increased would have important benefits to improve the fresh quality of SCM production.
3. The compressive strength of all SCM improve with introduction of MP at young age, this increase stabilize beyond 28 days. The substitution of cement with 15% CB as ternary cementitious blends increased the compressive strength of the control SCM mixture by 10% and 5% at 56 and 90 days, respectively.
4. Maximum density and UPV is obtained for mixtures composed of 10% of MP and 10% or 15% of CB, which confirm the presence of synergistic effect between calcined clays and calcium carbonate fillers.
5. The absorption of water increased as the amount of ternary cement (MP+CB) increased.
6. The feasibility use of combination of MP and CB at replacement rate of 15 or 20% to produce a new ternary blended cement in order to reduce the CO_2 emissions for sustainable development.

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