

# PROPERTIES OF CEMENT AND MORTAR INCORPORATING MARBLE DUST AND CRUSHED BRICK

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*In this study, marble dust (MD) generated by the marble cutting industries and crushed bricks (CB) have been used as a cement replacement materials. The properties of cement and mortar containing MD and CB were investigated. The volume expansion, setting time, compressive strength and flexural strength of the mortar were determined. The microstructure of the mortar was investigated using scanning electron microscopy (SEM). The setting time of the cements were retarded when the waste material replaced a part of the clinker. The inclusion of MD at replacement level of 8 % resulted in a reduction in the strength of the mortar. However, incorporation of the MD and CB in cement resulted in significant increase in the strength of the mortar compared to MD mortar. In conclusion, the use of MD in combination with CB as a replacement material in cement is promising. However, the full evaluation of these materials demands a thorough examination in areas related to their specialized properties, and also extending testing on concrete produced with these materials.*

## INTRODUCTION

Blended cements based on the partial replacement of Portland cement clinker (PC) by wastes have been the subject of many investigations in recent years. The use of the replacement materials offer cost reduction, energy savings, arguably superior products, and fewer hazards in the environment. These materials participate in the hydraulic reactions, contributing significantly to the composition and microstructure of hydrated product [1-7].

The CB is among the waste materials. The raw materials used in the manufacture of bricks are mainly natural clays containing quartz, and feldspar. Bricks are manufactured by the calcination of aluminosilicate clays. Heating destroys the crystal structure of the clays, which results in amorphous or disordered aluminosilicate structure. Amorphous substances react with lime to produce calcium silicate hydrate and/or calcium aluminate hydrate at the brick-lime interface. It has been shown that the calcination temperature of clay has significant effect on the pozzolanic activity depending on the type of the clay mineral [8]. For kaolinite clay, heat treatment converts kaolinite clay (weakly pozzolanic) to metakaolin (highly pozzolanic) at 700-800°C. Although, at temperatures over 800°C, pozzolanic activities of metakaolin are lost due to the formation of high temperatures minerals, some pozzolans such as pulverized-fuel ash (PFA), pozzolanic component,

which is amorphous glass phase, occurs above 1000°C. Studies have shown that a clay brick heated at around 1000°C gives maximum pozzolanic activity when incorporated into cementitious materials [9]. Depending on the type of the CB, it alters the pore size distribution, threshold radius, and compressive strength of the mortars [10]. It also increases the resistance of the mortar to sulfate attack and expansion [11, 12].

Despite growing attempt of using CB as cement constituent, little attention has been paid on the study of MD behavior when it is co-ground with clinker and gypsum to produce blended cement. Large quantities of MD are produced annually in Turkey. The MD is generated as a by-product during the cutting of marble. During the cutting process, about 25 % marble is resulted in dust. The marble cutting industries are dumping the MD in any nearby pit or vacant spaces. This imposes threats to eco-system, and physical, chemical and biological components of the environment. Therefore, utilization of MD in the production of new materials will help to protect environment. Recently the use of MD as replacement materials has been investigated. Agarwal and Gulati [13] demonstrated that the presence of MD in the matrix enhances the early compressive strength of the mortar, and the strength of the mortar decreases with the increasing MD content. According to authors, both of the early and long-term strength of the mortar can be improved by the inclusion of slag and fly ash in the matrix.

Industrial waste management is one of the major environmental problems in Turkey. Therefore, recycling and reuse of industrial wastes play vital role both in solving industrial waste problem and in getting benefit from it. This paper presents a preliminary study on the blended cements produced in laboratory by intergrinding natural pozzolan, crushed brick, marble dust, Portland cement clinker and gypsum. Properties examined include compressive strength, flexural strength of the mortar. X-ray diffraction (XRD) of raw materials and scanning electron microscopy (SEM) hydrated specimens were done and compared with the similar data from a control sample.

EXPERIMENTAL

The cementitious materials used in this study were Portland cement clinker (PC), gypsum (G), natural pozzolan (NP), marble dust (MD), and crushed brick (CB). The PC, and gypsum were obtained from Set cement Plant (Afyon, Turkey). The bricks types used derived from a variety of sources in Turkey, and are referred to as B, D, and L. The MD was taken from deposits of marble factories. The chemical compositions of all materials are given in Table 1.

Four series of mixtures and one reference mixture were prepared according to TS EN 197-1 [14]. Before mixing, gypsum optimization was done for the clinker used and was found to be 5 wt.% of the clinker. Reference mixture was produced by intergrinding PC and gypsum and designated as R. The other series cements were produced by co-grinding of clinker, NP, G, MD,

and CB, and designated as M1(PC+G+NP+MD), M2(PC+G+L+MD), M3(PC+G+B+MD), and M4(PC+G+D+MD). The characteristics of mixtures as well as their chemical compositions are given in Table 2. A laboratory ball-mill was used for the grinding process. The physical tests were carried out following grinding according to TS EN 196-6 [14].

The required water of standard consistency, setting time and volume expansion were examined according to TS-EN 196-3 [14]. The specimen preparation for strength tests was performed at the room temperature. Mortar specimens were produced by mixing one part of the cement with three parts of sand, using a water-to-cement ratio (w/c) of 0.50. The cement-water mixtures were stirred at low speed for 30s, and then, with the addition of sand, the mixtures were stirred for 4 min. The mortars prepared were cast into 40x40x160 mm moulds for strength tests. After 24 h of curing at 20°C, the samples were demolded, and then immediately immersed in a water-curing tank. The temperature of the water was maintained at 20 ±1°C during the curing period. The compressive strength and flexural strength test were carried out at the ages of 2, 7, 28 days according to TS EN 196-1 [14]. The strength value was the average of three specimens.

For SEM studies, selected mortar specimens cured for 28 days were used. A cement prism was cut into cubes approximately 10 mm square, one side of which was ground flat. The hydrated samples were flooded with acetone to stop hydration reactions. After drying and coating with gold and the SEM image of samples were obtained using a JEOL JXA 840A scanning electron microscope. For XRD studies, raw materials were

Table 1. Chemical characteristics of used material.

Oxides	Chemical analysis (wt.%)						
	Clinker	Natural Pozolan	Marble waste	L	B	D	Gypsum
SiO <sub>2</sub>	21.40	74.90	1.38	54.65	54.73	52.02	1.84
Al <sub>2</sub> O <sub>3</sub>	4.99	13.55	0.37	21.60	21.90	21.63	3.43
Fe <sub>2</sub> O <sub>3</sub>	3.55	0.96	0.24	7.46	7.71	7.97	0.50
CaO	64.73	1.00	53.12	5.09	4.42	4.88	35.50
MgO	2.61	-	0.38	2.03	2.11	2.35	0.44
SO <sub>3</sub>	1.25	0.07	0.24	2.58	2.62	3.90	39.12
Na <sub>2</sub> O	0.18	1.32	-	0.41	0.41	0.42	-
K <sub>2</sub> O	0.99	4.39	0.11	4.05	4.19	4.20	0.10
Loss on ignition	0.30	2.90	43.53	2.12	1.87	2.57	19.00

Table 2. Composition of cement containing marble dust and waste brick.

		SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	LOI
M1	(65% PC + 5% G + 22% NP + 8% MD)	30.57	6.42	2.69	48.38	1.75	2.84	1.72	0.40	5.15
M2	(65% PC + 5% G + 22% L + 8% MD)	26.16	8.19	4.05	49.27	2.20	3.38	1.51	0.23	4.98
M3	(65% PC + 5% G + 22% B + 8% MD)	26.17	8.28	4.07	49.10	2.26	3.38	1.60	0.22	4.92
M4	(65% PC + 5% G + 22% D + 8% MD)	25.61	8.21	4.13	49.15	2.25	3.60	1.60	0.25	5.11

ground to a fine powder of < 63 µm. A Rikagu Miniflex X-ray diffractometer using mono-chromatic radiation operating at a voltage of 30 kV and current of 15 mA was used. A scanning speed of 2° 2θ/min and a step size of 0.02° were used to examine the samples in the range of 5-60° 2θ.

RESULTS AND DISCUSSION

The XRD patterns of the three CB and MD are shown in Figures 1 and 2, respectively. The GB samples had very similar diffraction patterns. XRD patterns of all brick showed that they were composed of quartz, being the main component, illite, albite, and hematite. However, the intensities of XRD patterns of bricks are different and suggest that the bricks were manufactured differently by using materials of high clay content. The identified crystalline phases of MD were calcite and dolomite. It contains CaO, SiO<sub>2</sub>, and small amount of MgO, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> (Figure 2). Figure 3 shows the compressive strength of the mortars containing CB types, L, B, D, and marble dust at different curing ages.

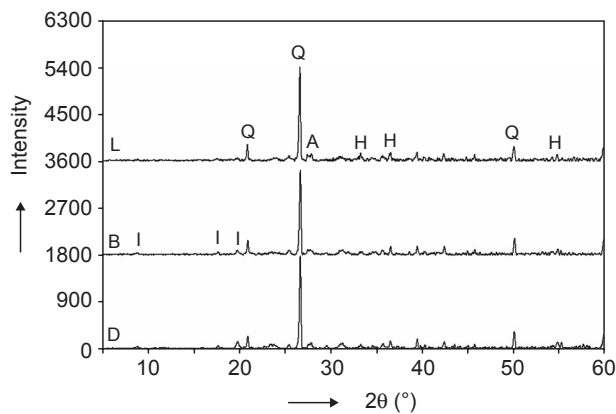


Figure 1. XRD pattern of crushed brick. Q - quartz; I - illite; A - albite; H - hematite.

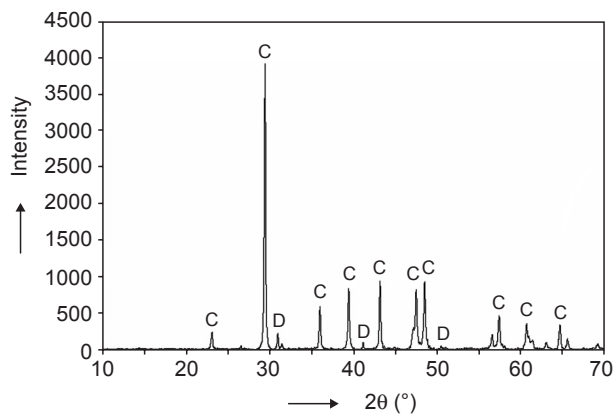


Figure 2. XRD spectrum of the marble dust. C - calcite; D - dolomite.

The partial replacement of PC by CB and MD (irrespective of source) results in a significant decrease in compressive strength of the mortar compared to the control mortar. The reduction in the strength of the mortars may be attributed to the pozzolanic activity and pore structure of the cementitious materials. Since, the replacement of the PC by waste materials reduces clinker content of the cement the amount of cementitious gel formed from pozzolanic reaction decreases. Hence, the strength contribution from this process is lower than that of the control mortar without waste material. It is generally accepted that the strength of mortar is fundamentally a function of the distribution of the void space and porosity in it. In general, the results obtained in this study on the effect of CB are in agreement with those reported by O'Farrell et al. [10]. They showed that the use of CB as a partial Portland cement replacement material increases intruded pore volume, reduces percentage of fine pores and compressive strength of the mortar.

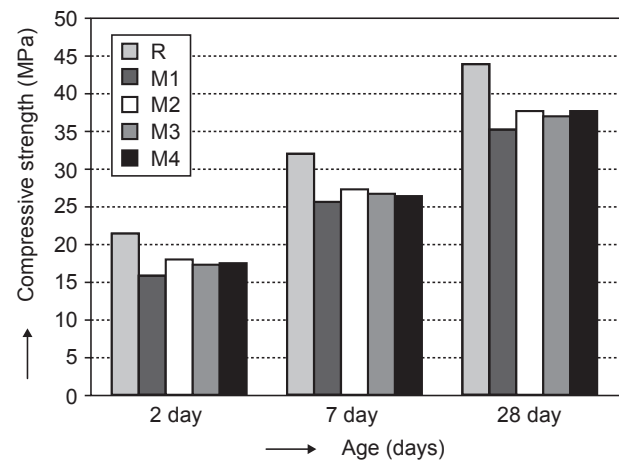


Figure 3. Compressive strength of the mortar containing MD and CB.

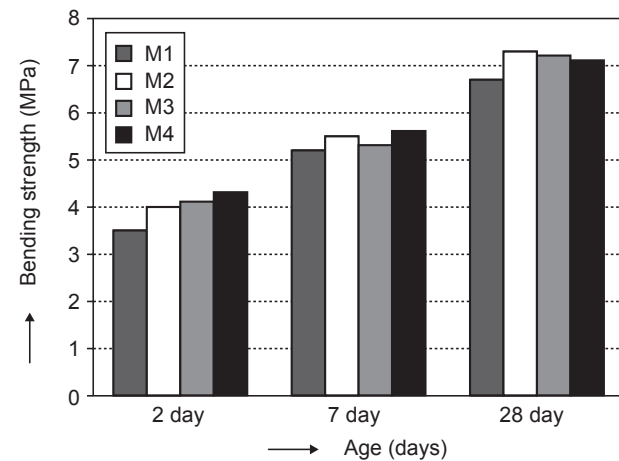


Figure 4. Flexural strength of the mortar containing MD.

It is observed that the incorporation of the MD and CB in cement enhance the compressive strength of the mortar compared to the mortar containing MD. The increase in the strength of mortar can be attributed to the glass content of the CB and calcium carbonate content of MD. Studies have shown that the glass content of the ground brick plays an important role in the strength development of ground brick mortars [10]. The additional surface area provided by the calcium carbonate in MD may provide sites for the nucleation and growth of hydration products that leads to further increase in strength [15].

The flexural strength for the specimens with CB and MD is seen in Figure 4. The flexural strength of the MD mortar was lower than that of the mortar containing MD and CB. The incorporation of CB enhances the transition zone strength. Hence, the improvement of flexural strength may be attributed to the nucleation of CH around the fine and well distributed particles of CB additions that replace the large and oriented crystals of calcium hydroxide with numerous, small and less oriented crystal.

The physical properties of the cements are given in Table 3. The blended cements demand significantly more water than the Portland cement. The increase in water demand is mainly attributed to the high fineness

of GB and MD. The blended cements had higher Blaine fineness value compared with corresponding reference Portland cement for equal grinding time. Although, M4 was slightly finer than M1, with respect to particles coarser than 45- $\mu\text{m}$  sieve, its specific surface was higher. The coarse phase in M1 may be attributed to the natural pozzolan component, which is harder to grind compared with CB and MD due to its high content of silicon dioxide. The soundness of cements (expansion according to Le Chatelier process) is satisfactory. From the table, it is clear that the general effect of the waste material is to increase setting time of the cement. The observed retardation in setting times can be mainly attributed to the lower clinker content of the blended cement. For blended cement, which has lower clinker content, the cement particles are expected to be less closely packed. This could decrease extent of interparticle contacts, thus it could slow down the setting. Furthermore, the waste material could also contribute to the change in setting times depending on the rate of pozzolanic reaction.

SEM micrograph of the mortar specimens cured for 28 days are shown in Figures 5-8. It is clearly seen from Figure 5 that an abundance of hydrated phases as well as  $\text{Ca}(\text{OH})_2$  crystal intermixed with calcium silicate hydrate exists in mortar R. However, mortars containing

Table 3. Water content, setting time, and physical characteristics of cementitious mixes.

Symbol	Water (%)	Setting Time (min)		Fineness (wt.%) + 40 $\mu$	Soundness (mm)	Specific surface ( $\text{m}^2/\text{kg}$ )	Density ( $\text{kg}/\text{m}^3$ )
		Initial	Final				
R	24.00	138	234	20.6	1	311.8	3060
M1	31.00	198	294	18.3	1	404.1	2800
M2	29.60	210	294	18.4	1	393.9	2700
M3	29.60	168	300	18.8	2	473.4	2800
M4	29.00	204	264	18.2	1	420.5	2500

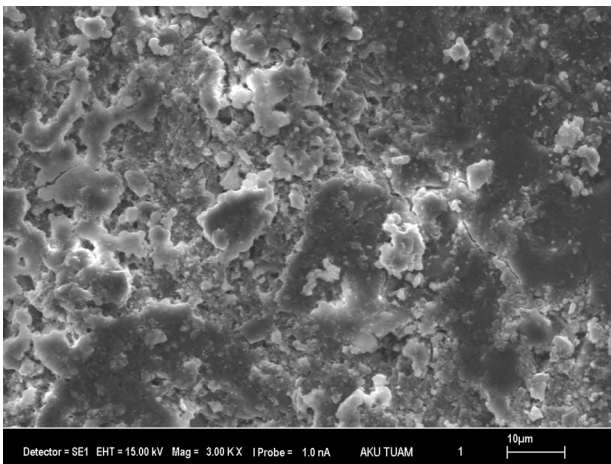


Figure 5. SEM micrographs of fracture surface of mortar after 28 days of hydration (the cement used contains 65% PC + 5% G + 22% NP + 8% MD).

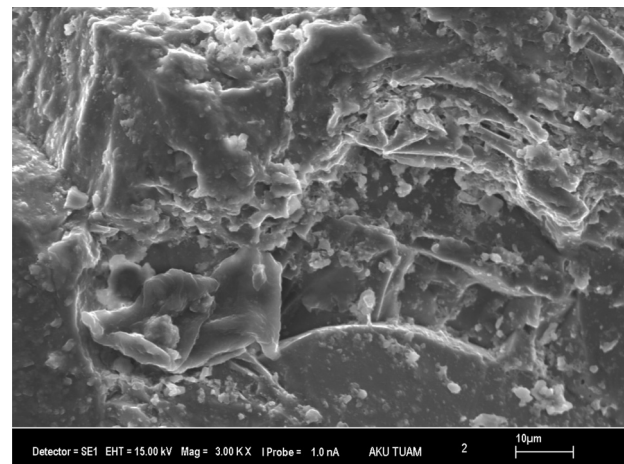


Figure 6. SEM micrographs of fracture surface of mortar after 28 days of hydration (the cement used contains 65% PC + 5% G + 22% L + 8% MD).

CB possesses different microstructure as seen in Figures 6-8. This difference may be attributed to the chemical nature of the CB. As mentioned above, pozzolanic reaction of CB depends on the firing temperature of clay [8].

## CONCLUSIONS

The main conclusion derived from this study may be summarized as follows:

1. The general effect of MD and CB is to retard the setting time of the cement.
2. The replacement of PC by MD and CB influences significantly the strength of the mortar. The strength of the mortar containing waste materials was lower than that of the control mortar.
3. Depending of the CB resource, the incorporation of MD and CB results in an enhanced flexural strength compared to the PC mortar containing MD.

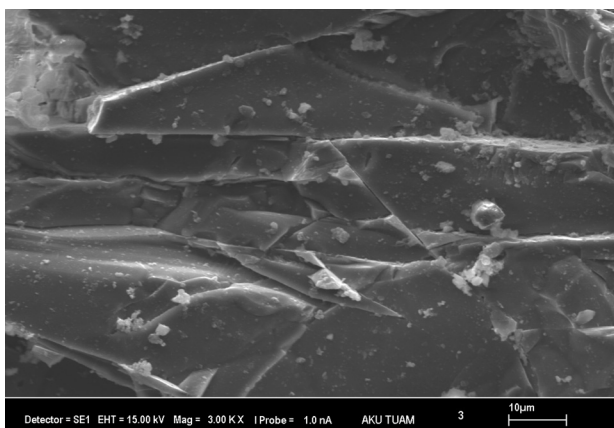


Figure 7. SEM micrographs of fracture surface of mortar after 28 days of hydration (the cement used contains 65% PC + 5% G + 22% B + 8% MD).

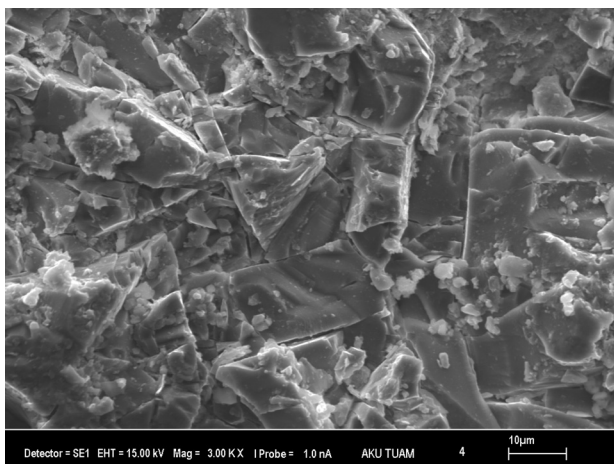


Figure 8. SEM micrographs of fracture surface of mortar after 28 days of hydration (the cement used contains 65% PC + 5% G + 22% D + 8% MD).

4. The cement containing waste material demands higher water content than Portland cement.
5. The production of cement containing MD and CB seems to be very challenging, due to satisfactory properties of the blended cement as well as the low cost and the availability of MD and CB in Turkey.

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## VLASTNOSTI CEMENTU A MALTY PŘI POUŽITÍ MRAMORU

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V této studii byl jako náhražka cementu použit průmyslově vzniklý mramorový prach (MP) a drcené cihly (DC). Byly zkoumány vlastnosti cementu a malty s obsahem MP a DC. U malt se stanovovala objemová roztažnost, doba tuhnutí, pevnost v tlaku a pevnost v ohybu. Pomocí skenovací elektronové mikroskopie (SEM) byla zkoumána mikroskopická struktura malty. V případě náhrady části slinku odpadním materiálem došlo k retardaci doby tuhnutí. Náhrada 8 % slinku MP vedla ke snížení pevnosti malty. Přidavek MP a DC do cementu však vedl ve srovnání s maltou pouze s přidavkem MP k významnému zvýšení pevnosti malty. Závěrem lze konstatovat, že použití mramorového prachu v kombinaci s drcenými cihlami jako náhražky cementu je slibné. Úplné zhodnocení těchto materiálů však vyžaduje důkladný výzkum v oblastech, které souvisejí s jejich speciálními vlastnostmi, a také delší testování betonu vyrobeného s těmito materiály