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## Conversion of Waste Marble Powder into a Binding Material

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#### Abstract

In the marble industry, a lot of marble is wasted in the form of odd blocks of various sizes and slurry consisting of water and micro-fine particles. The slurry on drying converts into powder. Both slurry and powder have adverse effects on the environment. This research is focused on the gainful utilization of waste marble powder (WMP) by converting it into a valuable binding material. For this purpose, WMP and clay were collected, and their physical and chemical properties were determined. A mix of WMP and clay was prepared and burnt at a temperature around 1300 °C. The burnt mix was ground to powder form to get marble cement (MC). The MC was then used in mortar. The compressive and flexural strengths of mortar cubes and prisms were determined. Apart from this, X-ray diffraction (XRD) analysis, thermogravimetric analysis (TGA) and scanning electron microscopic (SEM) analysis were also carried out. The chemical composition showed that the MC has 52.5% di-calcium silicate ( $C_2S$ ) and 3.5% tri-calcium silicate ( $C_3S$ ).The compressive strength of MC mortar after 28 days curing is 6.03 MPa, which is higher than M1 mortar of building code of Pakistan (5 MPa). The compressive strength of MC mortar after one year is 20.67 MPa, which is only 17% less than OPC mortar.

Keywords: Marble Powder; Binding Material; Cement; Mortar; Mechanical Properties.

## 1. Introduction

Marble stone has been used for construction and decoration purposes since a very long time [1]. Its demand has increased exponentially in the last few years and is growing further. Pakistan is one of the largest marble producing countries in the world. It has around 300 billion tons of known reserves of marble, and the actual reserves can be much more [2]. In Pakistan, about one million ton of marble stone is quarried and processed in marble factories annually. During quarrying operations, blasting technique is mostly employed, which results in the wastage of approximately 50 percent of marble stone [3]. The waste generated in quarries is in the form of odd rocks of various sizes [4]. However, there is no proper way to dispose of the waste, and thus the waste remains scattered in the vicinity of the quarries.

The raw marble blocks of large sizes are taken from the quarries to the marble processing units to produce marble tiles and other valuable stones of different dimensions and shapes [5]. Pakistan has numerous marble processing units with a sheer variety of machinery and equipment employed for the processing of these stones. During the cutting and polishing of the raw marble blocks, waste is also produced as a by-product. The waste is in the form of odd blocks of

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different sizes, shapes, and slurry, which contains very fine marble particles. Approximately 20% of these blocks are reduced to the micro-fine particles, which varies with the processing technology [6].

The slurry is usually discarded in the vacant spaces nearby the factories. There is no systematic way to eliminate the slurry; thus, it results in vast mounds of wastes. Consequently, the slurry dries and converts into a fine powder.

During windy seasons, heavy winds can easily carry the fine marble particles and can cause various health issues such as lung cancer, skin and eye irritation [7]. Besides, the heap of slurry remains scattered all around the industrial sector and spoils the aesthetics of the entire area [8]. Moreover, upon mixing with water, the slurry causes water pollution [9]. In addition, it reduces the porosity and permeability of the topsoil, resulting in the waterlogging of the area [10]. The fine marble particles reduce the productiveness of soil by increasing its alkalinity. Further, there is a loss to flora and fauna. i.e., already grown trees and bushes are dried out due to deposition of micro marble particles on leaves of plants and vegetation [11].

In order to solve the aforementioned problems, different researchers have used waste marble slurry and powder in various construction materials. Sutcu et al. [12] and Saboya et al. [13] utilized waste marble powder in the production of fired clay bricks. The authors concluded that the weight of clay bricks reduces considerably, while its compressive strength reduces marginally. Gencel et al. [14] used waste marble in the production of concrete paving blocks. They observed that the increase in the amount of marble sludge, results in a decrease of compressive strength; however, this reduction lies within acceptable limits. Moreover, the addition of waste marble increased the durability and freezethaw resistance of the concrete blocks. Rehman et al. [5] incorporated marble slurry in the preparation of masonry bricks. Apart from slurry; cement, plaster of paris, sand, crushed stone and acrylic fibers were also used. They concluded that when waste marble slurry, sand and cement were used in a ratio of 80, 5 and 15 respectively, along with acrylic fibers; a maximum compressive strength of 8.94 MPa was achieved, which is satisfactory for the construction activities where high strength is not required. Moreover, these bricks are low cost as compared to the conventional bricks. Kabeer and Vyas [15] replaced sand in mortar with WMB in various percentages up to a maximum of 100 %. They reported a considerable enhancement in the properties of mortar such as compressive strength, flexural strength, modulus of elasticity and density, considerably improve upon the 20 % replacement of sand with WMP. Sedat et al. [16] investigated the replacement of dolomite with marble powder in adhesive mortars of tiles and insulating boards etc. According to the authors the 28 days compressive strength of bonding mortar containing 100 % marble powder instead of dolomite, was 15 MPa, which is well above the limiting value of 6 MPa as per European standards institute EN-12808-3. Similarly, the 28 days flexural strength of bonding mortar containing 100 % marble powder was 9.5 MPa, which is more than the minimum required value of 0.08 MPa of European standards institute EN- 1348. In addition, the authors carried out cost analysis and concluded that the bonding material with marble powder was 30 % cheaper than the one containing dolomite.

Topcu et al. [17] and Alymac & Ince [18] added waste marble dust as a filler material in self-compacting concrete. They highlighted that waste marble dust did not affect the workability of self-compacting concrete.

Khodabakhshian et al. [19] substituted cement with waste marble powder up to a maximum of 20 percent in concrete. They observed significant increase in the compressive and flexural strengths along with the modulus of elasticity of concrete, when cement is replaced with 5 % WMP. However, mechanical properties decrease with the increase in the replacement level of WMP beyond 10%. Furthermore, the cement was substituted in various percentages with both silica fume (SF) and WMP. When 10% of SF is used; then for any percentage of WMP powder up to a maximum of 20% the mechanical properties of concrete improved. Singh et al. [20] replaced cement with WMP in concrete blocks, in various percentages up to a maximum of 25%. They also varied water to binder ratio for all mixes. The authors recorded an improvement in the mechanical and durability properties of concrete block up to 15% replacement.

Aliabdoet al. [21] highlighted the use waste marble powder/dust in the production of cement as well as concrete. Marble dust was integrated with cement to prepare marble dust blended cement. The marble dust was mixed with cement in various proportions up to a maximum of 15% by weight. The blended cement paste and mortar samples were prepared and tested. It was observed that WMD mixed with cement satisfies the requirements of Egyptian standards. Similarly, the sand in concrete was replaced by waste marble dust in various percentages up to a maximum of 15%. It was concluded that the addition of waste marble dust with a lower water-cement ratio results in concrete having better physical and mechanical properties than the conventional concrete.

Ergun [22] utilized waste marble dust and diatomite as partial replacement of cement in concrete. The samples of concrete were properly cured and tested. The test results indicated that the concrete containing 10% waste marble dust and 5% diatomite or 5% waste marble dust and 10% diatomite as cement replacement has better flexural and compressive strengths as compared to the ordinary concrete.

Ma et al. [23] partially replaced cement by WMP and nano silica (NS) in mortar. According to them the fluidity and setting time of mortar was increased when cement was replaced with WMP only; however, the compressive strength of mortar was significantly reduced when the replacement was more than 10%. The mechanical properties were significantly improved when cement was replaced by a combination of WMP and NS. An optimum replacement of cement with 10% WMP and 3% NS was suggested. Munir et al. [24] partially substituted cement with WMP by 0, 10%, 20%, 30% and 40% in mortar. Workability of mortar with WMP was less as compared to controlled mix. Similarly the compressive strength of mortar with 10% WMP was more than the controlled mix, which decreased with further increase of WMP content.

Kavas and Olgun [25] prepared waste marble dust and crushed bricks blended cement, by partially replacing cement with these materials. Different properties like setting time, volume expansion, flexural strength and compressive strength of both blended cement and mortars containing the blended cement were found by conducting various tests. Scanning electron microscopy (SEM) analysis was carried out to find the microstructure of the mortar. The setting time was delayed with the addition of waste marble dust and crushed bricks in cement. Moreover, the compressive strength of the mortar containing waste marble dust was reduced. However, the inclusion of both waste marble dust and crushed bricks in cement significantly increased the compressive strength of mortar. It was concluded that the partial replacement of cement with waste marble dust and crushed bricks showed very promising results. Aruntas et al. [26] utilized waste marble dust in various percentages in cement to prepare waste marble dust blended cements (WMDCs). The cement clinkers were replaced up to 10% by weight with waste marble dust. Samples of cement mortar were prepared and cured properly. The chemical, mechanical and physical properties of mortar were determined after 7, 28 and 90 days, respectively. It was concluded that WMDCs satisfy EN-197-1 standard, which specifies the 10% replacement of cement clinker with WMD in the manufacturing of blended cement. Khan et al. [3] employed waste marble dust (WMD) in the production of burnt clay bricks. According to the author, the chemical composition of WMD and clay is given in Table 1.

M	Class	Marble dust		
Major elements (Wt %)	Clay	Buneer	Mohmand Agency	Mix
SiO <sub>2</sub>	57.55	0.77	0.42	0.31
TiO <sub>2</sub>	0.3	0.01	0	0
$Al_2O_3$	15.84	0.24	0.07	0.04
Fe <sub>2</sub> O <sub>3</sub>	3.32	0.05	0	0.03
MnO	0.04	0	0	0
MgO	2.35	0.94	0.24	0.3
CaO	6.77	54.08	55.65	55.07
Na <sub>2</sub> O	1.39	0.39	0.36	0.29
$K_2O$	1.59	0.02	0.01	0.01
$P_2O_5$	0.12	0.01	0.03	0.02
LOI	10.73	43.49	43.232	43.93

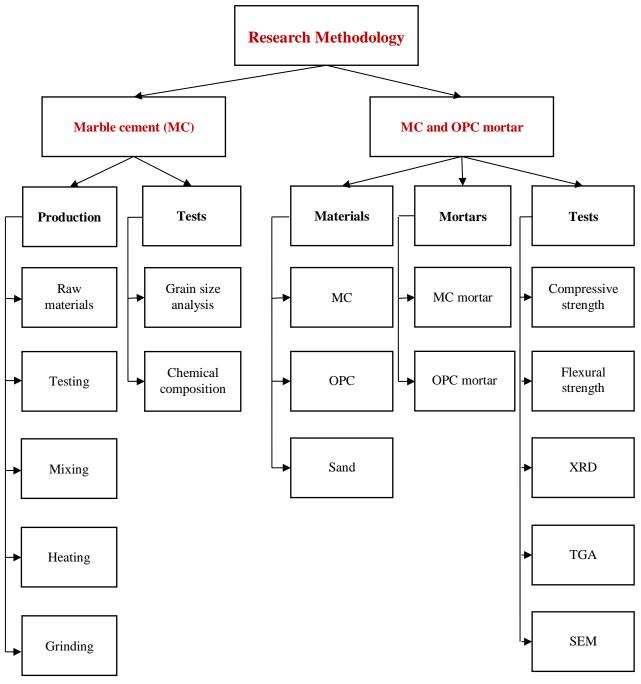
Table 1. Chemical composition of WMD and clay [3]

According to Neville and Brooks [27], the Ordinary Portland cement (OPC) contains approximately 60 to 65 percent calcium oxide (CaO), 20 to 25 percent silica (SiO<sub>2</sub>) and 4 to 8 percent alumina (Al<sub>2</sub>O<sub>3</sub>). Furthermore, according to the authors, ' limestone is utilized as the primary source of CaO in the production of cement. The CaO when reacts with SiO<sub>2</sub> at a temperature of 1450 to 1650  $^{\circ}$ C, produce calcium silicates (C-S), which on reaction with water results in calcium silicate hydrate (C-S-H), having excellent binding properties. The chemical composition of limestone and marble is nearly the same, with both containing a large amount of CaO. Therefore, waste marble powder/slurry and a silica-rich material, e.g., clay, can be used in the production of binding material.

Limestone is the primary raw material used in the production of cement. However, large scale cement production requires a massive quantity of limestone. Thus the natural resources of limestone are going to diminish. Creating a binding material from waste material can result in an alternative material for the OPC, and therefore it is an essential step towards the sustainability of natural resources. Apart from this, hazardous waste material can be converted into valuable material. To the best of authors' knowledge no published data is available on the conversion of micro marble powder into a binding material. In this research, an effort has been made to utilize waste marble powder in a beneficial manner. The waste marble powder and clay have been processed to form marble cement to be used in the construction industry. Various techniques i.e., grain size analysis, X-ray diffraction (XRD), thermogravimatric analysis (TGA) and scanning electron microscopy (SEM) were used for the determination of different properties of marble cement (MC) powder and paste. Besides, mechanical properties such as compressive and flexural strengths of marble cement mortar were also evaluated.

### 2. Materials and Methods

Flow chart of the methodology is shown in Figure 1.





#### 2.1. Collection and Testing of Raw Materials

In this step, the raw materials were collected, which consists of waste marble powder and clay. The waste marble powder was collected from a marble processing factory at Industrial estate, Hayatabad, Peshawar; while the clay was collected from ring road Peshawar.

After collecting the raw materials, their chemical and physical properties were examined with the help of X-ray fluorescence (XRF) test and grain size analysis.

#### 2.2. Chemical Composition of Raw Materials

Chemical composition of waste marble powder and clay were determined by X-ray fluorescence (XRF) analysis. The results are given in Table 2.

T.	WMP	Clay Weight (%)	
Item —	Weight (%)		
Na <sub>2</sub> O	0.03	4.14	
$Al_2O_3$	0.19	14.2	
MgO	2.73	4.32	
CaO	50	3.46	
SiO <sub>2</sub>	0.4	56.73	
K <sub>2</sub> O	0.01	1.01	
MnO	0	0.05	
$P_2O_5$	0.01	0.12	
Fe <sub>2</sub> O <sub>3</sub>	0	3.23	
TiO <sub>2</sub>	0	0.55	
Loss on ignition	46.63	12.19	

Table 2. Chemical composition of raw materials

The quantity of  $CaCO_3$  in waste marble powder is 96.63%; out of which CaO is 50%, while the loss on ignition, i.e., the amount of  $CO_2$  is 46.63%. The chemical composition of waste marble powder is similar to limestone. Therefore, it has been used as the major raw material in the production of new binding material. Apart from this, clay contains 56.73% SiO<sub>2</sub>, which reacts at very high temperature with CaO to produce alite (tri-calcium silicate) and belite (di-calcium silicate). Hence, the raw materials have enough quantity of CaO and SiO<sub>2</sub>, making it a suitable source of binding material.

#### 2.3. Physical Properties of Raw Materials

Grain size analysis of waste marble powder and clay were carried out by grain size analyzer. The results are shown in Figures 2 and 3.

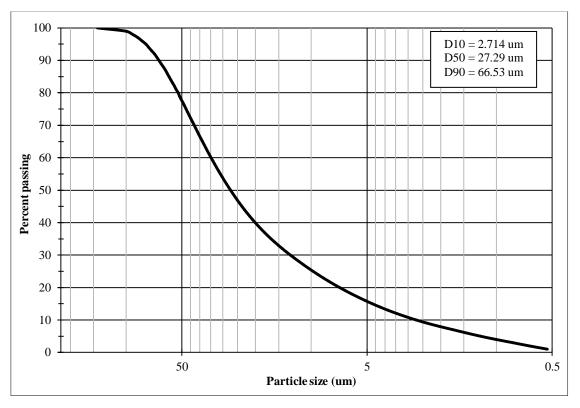


Figure 2. Grain size distribution of waste marble powder

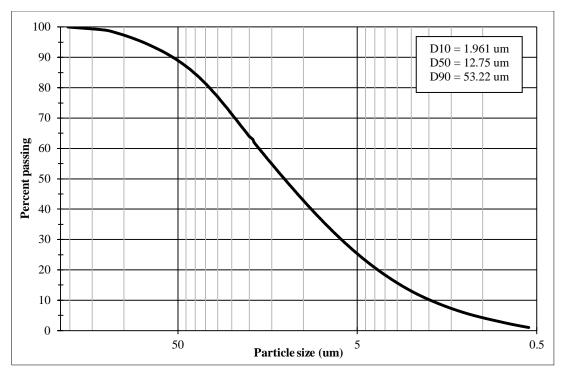


Figure 3. Grain size distribution of clay

D90 of waste marble powder and clay are 66.53  $\mu$ m and 53.22  $\mu$ m, respectively. Usually, D90 of raw mix in the production of OPC is kept below 90  $\mu$ m. As D90 of both the materials is well below 90  $\mu$ m; therefore, no grinding is required.

#### 2.4. Proportioning of Raw Materials

In this step, raw materials were mixed in specific proportions, depending on their chemical composition. The ratios were selected based on lime saturation factor. In the OPC production process, lime saturation factor is generally kept between 0.92 and 0.98, to fully convert the calcium oxide (CaO) produced from the calcination of calcium carbonate (CaCO<sub>3</sub>,) into calcium silicates after reaction with SiO<sub>2</sub>. The aim was to keep the final product free from CaO. The target was to achieve a lime saturation factor of 0.92 to 0.98. Based on the chemical composition, 25 Kg of waste marble powder and 7 Kg of clay, were mixed to get a lime saturation factor of 0.97.

#### 2.5. Mixing of Raw Materials

After deciding the proportions, the raw materials were first mixed thoroughly in dry form in a ball mill. Afterwards, they were mixed in a wet form with the help of sugarcane molasses and water. This wet mix was converted into pallets. Further, the pallets were exposed to sunlight for two days for the complete removal of water.

#### 2.6. Heating of Raw Materials and the Crushing of Clinkers Formed

In this step, the pallets were fed into a rotary kiln. Initially, the temperature of the kiln was low. However, the temperature was increased gradually to the maximum available capacity of 1300 °C. The pallets were burnt in the kiln at approximately 1300 °C for two hours.

There was no arrangement to cool down the hot pallets quickly; therefore, these were cooled down gradually by leaving them in the rotary kiln overnight. After cooling, the burnt pallets were removed from the kiln and converted to powder form in a ball mill. The resulting powder is the targeted binding material called marble cement.

#### 2.7. Testing of Binding Material

In this step, the phase composition of marble cement was determined by X-ray diffraction (XRD) test. The phase composition was to confirm whether marble cement had enough calcium silicates, which are necessary for developing binding properties. The fineness of both marble and OPC were determined with the help of grain size analyzer.

The tested marble cement and OPC powders were used in the preparation of mortar. Both OPC and marble cement mortars were prepared by mixing one part of cement with 2.75 parts of sand as specified in ASTM C 109 / C 109 M - 02 and ASTM C 348-14. Water to cement ratio was kept as 0.5 as per ASTM standard. However, the marble cement

mortar had no workability at water to marble cement ratio of 0.5; therefore, the water to marble cement ratio was kept 0.72 to make the mortar workable, as given in Table 3. The mortar was prepared manually as no mechanical mixer was available. To find the compressive strength of mortar, 50 mm  $\times$  50 mm  $\times$  50 mm cubes were prepared from both OPC and marble cement mortars as per ASTM C 109 / C 109 M – 02. Similarly, to determine flexural strength of mortar, 40 mm  $\times$  40 mm  $\times$  160 mm prisms were prepared from both OPC and marble cement mortars as per ASTM C 348-14.

Mixture notation	OPC	Marble cement	sand	w/c ratio	w/(M.C.) ratio
Controlled	1	0	2.75	0.5	0
MCM	0	1	2.75	0	0.72
* Test specimen size: (i) = 50 mm cube (0	Compressive s	strength);			
$(ii) = 40 \times 40 \times 160 \text{ mm}$	n (Flexural st	rength).			

The samples were removed from moulds after 24 hours. The OPC mortar samples were immersed in curing tank, immediately after removal from moulds. Due to the presence of high amount of lime in the marble cement, it dissolves if immersed in water immediately after unmoulding. Therefore, marble cement mortar samples were kept covered in a damp cloth for 14 days. After 14 days, the samples were immersed in curing tank. All the samples were kept immersed in a curing tank till the time of testing.

In order to carry out, X-ray diffraction (XRD) analysis, thermo-gravimetric analysis (TGA) and scanning electron microscopic (SEM) analysis, samples were prepared from marble and OPC pastes. The OPC paste samples were immersed in curing tank after 24 hours, while marble cement paste samples were covered with a damp cloth for 14 days. Afterwards, the samples were immersed in a curing tank.

#### 3. Results and Discussions

#### 3.1. Phase Composition

The phase composition of marble cement and OPC powder were determined by X-ray diffraction analysis. The results are given in Table 4.

C N-	<b>T</b> 4	Marble cement	OPC	
S.No.	Item –	Weight (%)	Weight (%)	
1	Alite [C <sub>3</sub> S]	3.73	59.51	
2	Belite beta [C <sub>2</sub> S]	52.50	17.14	
3	Ferrite [C <sub>4</sub> AF]	9.26	8.20	
4	Aluminate cubic [C <sub>3</sub> A]	2.48	2.81	
	Aluminate ortho [C <sub>3</sub> A]	3.38	5.59	
5	Free lime [CaO]	23.09	0.00	
6	Portlandite [Ca(OH) <sub>2</sub> ]	1.49	1.50	
7	Periclase [MgO]	3.66	3.10	
8	Acranite [K <sub>2</sub> SO <sub>4</sub> ]	0.00	1.78	
9	Aphthitalite [K <sub>3</sub> Na(SO <sub>4</sub> ) <sub>2</sub> ]	0.41	0.37	
10	Calciolangbeinite [K2Ca2(SO4)3]	0.00	0.00	

Table 4. Phase composition of marble cement and OPC powder

It can be seen from the results that marble cement has 3.73% C<sub>3</sub>S, 52.51% C<sub>2</sub>S and 23.09% free lime. The reason for the low percentage of C<sub>3</sub>S, and high percentages of C<sub>2</sub>S and free lime is the slow cooling process. The marble cement pallets were cooled very slowly. Therefore, C<sub>3</sub>S reconverted to C<sub>2</sub>S and CaO, after reaching 1100 °C. In OPC production the cement clinkers are cooled very rapidly to preserve the C<sub>3</sub>S formed at high temperature. Thus the OPC contains a low percentage of C<sub>2</sub>S and free lime as compared to marble cement, as given in Table 4.

#### 3.1. Grain Size Analysis

The grain size of marble cement and OPC were determined with the help of grain size analyzer. The results are shown in Figures 4 and 5.

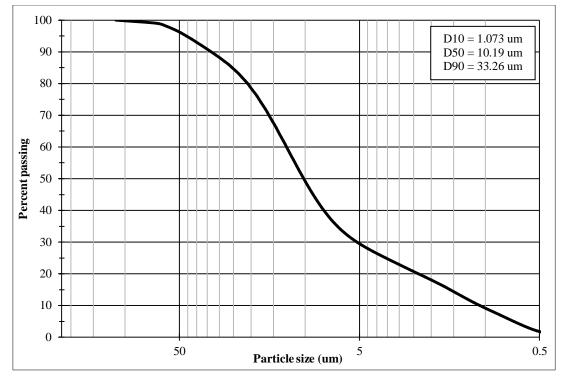


Figure 4. Grain size distribution of marble cement

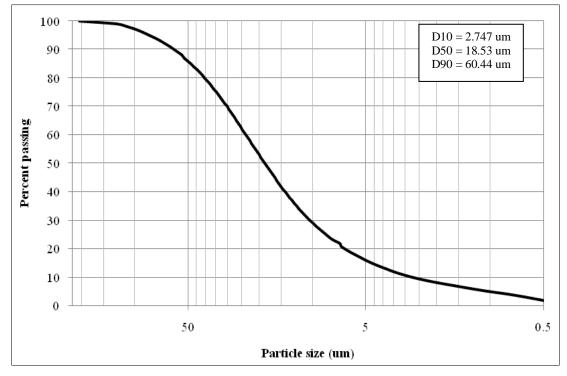


Figure 5. Grain size distribution of OPC

Figures 3 and 4 show that D90 of marble and OPC is  $33.26 \mu m$  and  $60.44 \mu m$ . As the marble cement powder is very fine, therefore it has a larger surface area. Because of large surface area, the water to marble cement ratio is higher than the water to OPC ratio, as shown in Table 3. Moreover, due to large surface area of the marble cement, the hydration starts immediately. Thus, more water is required to keep it workable.

#### 3.2. Compressive Strength of Mortar

In order to find the compressive strength of marble cement and OPC mortars, specimens prepared were removed from curing tank just before testing. The tests were carried out at the age of 7, 14, 28, 56, 91, 182 and 364 days, respectively. The load was applied at a rate of 1.2 KN/sec as per ASTM C109-02 [28]. The results are shown in Table 5 and Figure 6.

Compressive strength (MPa)			
Marble cement mortar	Portland cement mortar		
3.12	8.07		
4.79	10.85		
6.03	12.27		
7.79	13.45		
9.28	14.75		
13.25	18.05		
20.67	24.78		
	Marble cement mortar   3.12   4.79   6.03   7.79   9.28   13.25		

Table 5. Compressive strength of marble cement and OPC mortar

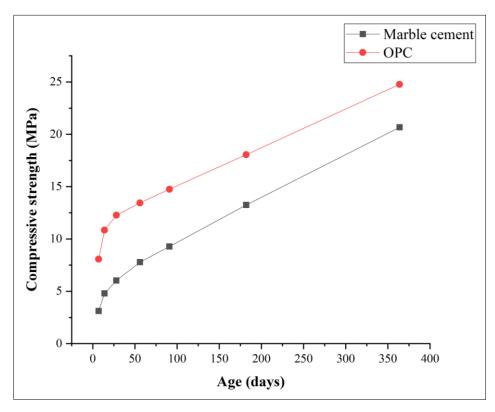


Figure 6. Compressive strength of Marble cement and OPC mortars

The compressive strength of marble cement mortar at 7 days is 3.12 MPa as compared to 8.07 MPa of OPC mortar. Thus, the compressive strength of marble cement mortar at the age of 7 days is 61% less than the strength of OPC mortar. Similarly, after 28 and 364 days, the compressive strength of marble cement mortar is 51% and 17% less than OPC mortar. In other words, the increase in the compressive strength of MC mortar is slow, but it continues for an extended period. Comparing the compressive strength of marble cement mortar at the age of 28 days, with its compressive strength at the age of 7 days, it is 94% more. Similarly, the compressive strength increased by 563% as compared to its 7 days strength. The compressive strength of the marble cement mortar increases slowly. It is because of the higher percentage of  $C_2S$ , which hydrates very slowly as reported by Ghosh et al. [29]. The 28 days compressive strength of marble cement mortar as per building code of Pakistan [30].

#### 3.3. Flexural Strength of Mortar

In order to find flexural strength of marble cement and OPC mortars, specimens were removed from curing tank just before testing. The tests were carried out at the age of 7, 14, 28, 56, 91 and 364 days, respectively. The results are shown in Table 6 and Figure 7.

A (D)	Flexural strength (MPa)		
Age (Days) -	Marble cement mortar	Portland cement mortar	
7	1.03	2.51	
14	1.34	2.99	
28	1.89	3.54	
56	2.28	3.99	
91	2.73	4.24	
364	3.64	4.88	

Table 6. Flexural strength of marble cement and OPC mortar

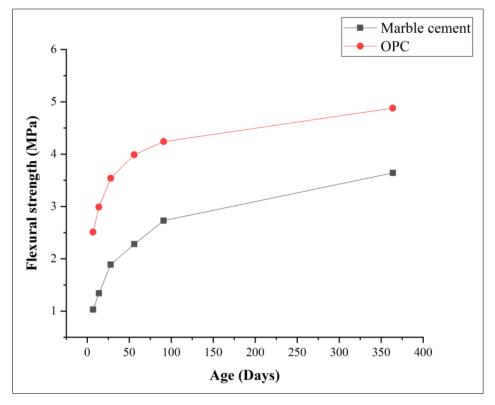


Figure 7. Flexural strength of marble cement and OPC mortars

The flexural strength of marble cement mortar at the age of 7 days was 1.03 MPa as compared to 2.51 MPa of OPC mortar as given in Table 6. Thus the recorded flexural strength of marble cement mortar was 59% lesser than the flexural strength of OPC mortar. Similarly, at the age of 28 and 364 days, the flexural strength of marble cement mortar went up to 1.89 MPa and 3.64 MPa as compared to 3.54 MPa and 4.88 MPa of OPC mortar. Thus, the flexural strength of marble cement mortar was 47% and 25% lesser than the flexural strength of OPC mortar at the age of 28 and 364 days, respectively. Comparing the 28 and 364 days flexural strength of marble cement mortar with its 7 days flexural strength; there was an increase of 83% and 253% respectively. The 28 days flexural strength of marble cement mortar is 1.89 MPa, which is more than the minimum requirement of 0.33 MPa of M1 mortar of building code of Pakistan [30]. The gradual strength enhancement is due to the presence of a large amount of C<sub>2</sub>S, which requires more time for the complete hydration. Slow hydration is the most important property of concrete to be beneficialy used in hot weather. To slow down the hydration of cement in hot weather, various kind of admixtures are used [31]. Therefore, marble cement could be used in hot weather without the addition of any admixture.

#### 3.4. X-Ray Diffraction (XRD Analysis)

X-ray diffraction analysis of marble cement and OPC powders and pastes were carried out. The results of marble cement powder and paste after 28 days curing are shown in Figure 8. Similarly, the results of OPC powder and paste after 28 days curing are shown in Figure 9.

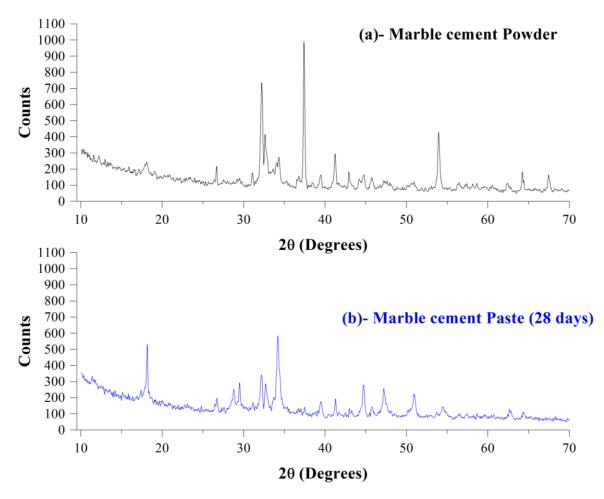
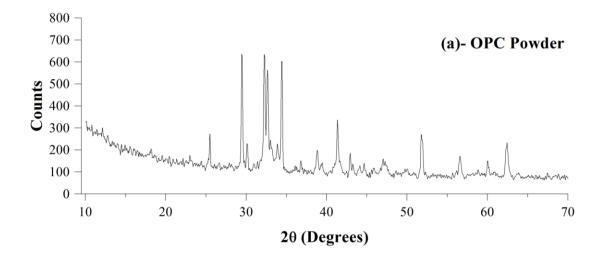


Figure 8. XRD analysis of marble cement powder and paste after 28 days curing

In Figure 8, there are sharp peaks at  $37.36^{\circ}$  and  $53.86^{\circ}$  in marble cement powder, which show 23.09 % free lime (CaO) present in marble cement powder. However, after hydration and 28 days curing these peaks have completely disappeared, because CaO converts to Ca(OH)<sub>2</sub> after hydration. Similarly, there is a sharp peak at  $32.05^{\circ}$  in marble cement powder, which shows di-calcium silicate. However, the peak has significantly decreased after hydration and 28 days curing. After hydration di-calcium silicate results in calcium silicate hydrate gel (C-S-H gel), and Ca(OH)<sub>2</sub>. The peaks in Figure 8(b) at  $17.5^{\circ}$ ,  $34^{\circ}$  and  $47.5^{\circ}$ , show Ca(OH)<sub>2</sub> produced as a result of hydration of CaO and calcium silicates present in the marble cement.



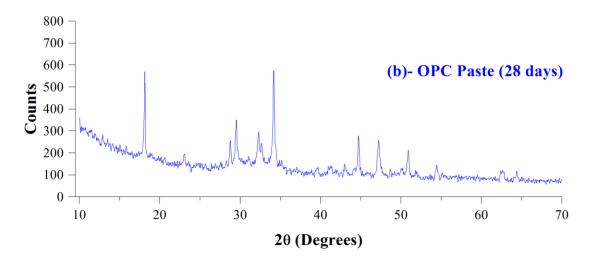


Figure 9. XRD analysis of OPC powder and paste after 28 days curing

In OPC powder the peaks between 29° and 35°, show C<sub>3</sub>S, C<sub>3</sub>A and C<sub>2</sub>S. After hydration, these result in C-S-H, C-A-H and Ca(OH)<sub>2</sub>. The peaks between 29° and 35° reduce significantly due to the formation of C-S-H and C-A-H as evident from Figure 9(b). Similarly, if we look at Figure 9(b), some new peaks have developed at 17.5°, 34° and 47.5°, which show Ca(OH)<sub>2</sub> produced as a result of hydration of OPC powder. It is in agreement with the results of Jadhav and Debnath [32].

#### 3.5. Thermo-Gravimetric Analysis (TGA)

Thermo-gravimetric analysis (TGA) of marble cement and OPC pastes were carried out. The results after 28 days of curing are shown in Figure 10.

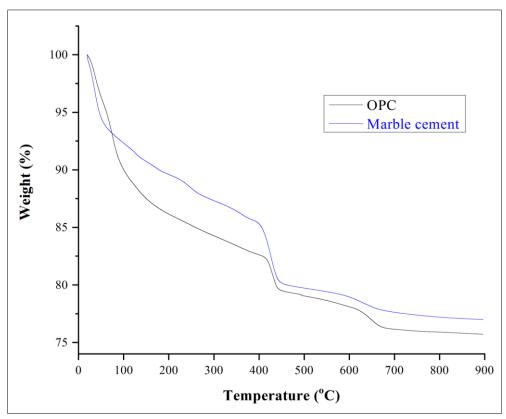


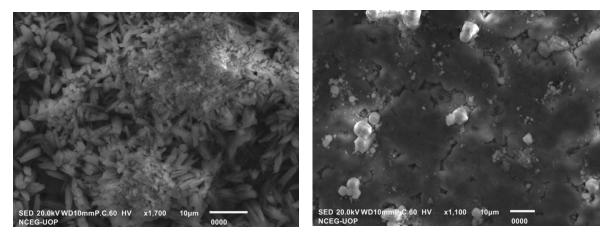
Figure 10. TGA analysis of Marble cement and OPC mortars

As the temperature increases from 0 °C to 100 °C, the surface water present in both materials is driven out. As both the materials are kept in water before test, so both of them have nearly the same quantity of free water and thus the loss of weight is nearly the same. As the temperature goes up from 100 °C to 400°C, the water present in C-S-H gel, ettringite and C-A-H gel is driven out [33]. The OPC paste has a higher quantity of C-S-H and ettringite as compared

to marble cement paste, therefore, in this range, the loss of weight in OPC paste is higher as compared to the marble cement paste. The loss of weight from 400 - 450 °C is due to the dehydration of Ca(OH)<sub>2</sub>. There is higher quantity of Ca(OH)<sub>2</sub> in marble cement paste as compared to the OPC paste. Hence, in this range, the loss of weight in marble cement paste is higher as compared to the OPC paste. Overall the weight loss in OPC paste is 1.3% more than marble cement paste, which shows that the hydration of OPC paste is faster than marble cement paste.

#### 3.6. Scanning Electron Microscopic (SEM) Analysis

Scanning electron microscopic images of marble cement and OPC pastes after 28 days curing are shown in Figure 11. It can be seen that marble cement paste has a higher quantity of  $Ca(OH)_2$  which was produced during the hydration of CaO and calcium silicates. Similarly C-S-H gel can be seen in Figure 11 (a), which was produced as a result of the hydration of calcium silicates. Likewise, OPC paste mostly consists of calcium silicate hydrate, formed due to the hydration of calcium silicates. Figure 11(b) illustrates the traces of portlandite, which were produced by the hydraulycis of C<sub>3</sub>S and C<sub>2</sub>S. The same was reported by Franus et al. [34].



(a) Marble cement paste

(b) OPC paste



## 4. Conclusions

In this study, waste marble powder was utilized in the preparation of marble cement. Various properties of marble cement were determined. In addition, marble cement was used in mortar, and different tests were performed on mortar to find its different properties. Based on the experimental investigations the following conclusions are drawn.

- The overall quantity of C<sub>2</sub>S and C<sub>3</sub>S in the marble cement is 56.23%. These two are very essential in developing binding properties. Hence, it can be used as a binding material.
- The newly developed marble cement can be confidently used in the mortar, as the one-year compressive strength of the mortar is 20.67 MPa. Although, the 28 days compressive strength of mortar is 6.03 MPa, which is higher than M1 mortar of building code of Pakistan.
- Due to the higher quantity of  $C_2S$ , the hydration of marble cement is slower than OPC and is thus highly recommended for hot weather concreting and mass concrete.
- The difference in the strengths of OPC and marble cement mortar was observed to decrease with the increase in time. For instance, the difference in the compressive strengths of marble cement and OPC mortar was 61% at 7 days, which decreased to 17% at 364 days.

## 5. Acknowledgement

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#### 6. Conflicts of Interest

The authors declare no conflict of interest.

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