Properties and Microstructural Characteristics of Concrete Made of Marble Powder as a Partial Replacement of Cement

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ABSTRACT
Marble is a metamorphic rock and one of the most important decorative building materials. About 20-30% of a marble block becomes marble powder throughout the cutting process. Marble powder is harmful to human health and to the environment. The main purpose of this research is to study the microstructural characteristics and properties of hardened concrete made with WMP as partial replacement of cement. The main variables in this research are the percentage of marble powder (0, 5, 10, 15, 20, 30, and 40%) as partial replacement of cement in concrete mixes and the age of testing (7, 28, and 56 days). Hardened concrete properties are investigated in terms of compressive, splitting tensile, flexure modulus, and bond strengths. Also, the modulus of elasticity of the mixes was measured and microstructural scanning by an electronic microscope was carried out. The microstructural analysis was conducted on concrete matrix densification, attributed to pores refinement due to both physical and chemical changes in the concrete matrix. The results showed that replacing cement by 10% of marble powder increased the strength and improved the concrete microstructure.

Keywords: Marble Powder; Replacement; Microstructure; mechanical properties.

1. Introduction
Aggregates are the main constituent of concrete representing about 75% of the concrete volume. Aggregates are obtained from natural quarries. The need for artificial aggregates has increased recently due to large and increased construction work all over the world. Industrial waste aggregates can become a lasting alternative to fine aggregates. Researchers have explored the use of various inert waste materials as partial or complete substitution of river sand as fine aggregates or as a partial replacement of cement. 20-30% of a marble block becomes waste marble powder (WMP) throughout the cutting process. Using replacement WMP as recycled aggregates improved the mechanical properties, workability, and the chemical resistance of conventional concrete [1]. Improvement in the durability of concrete made with natural granular granite, silica sand, WMP and basalt as fine aggregates due to low permeability. Also, the compressive strength increased 26% when using WMP additives ratio 10% of cement weight compared to control mix. Using WMP as partial replacement of cement or sand decreased the loss of energy during casting concrete less than other mineral additives such as silica dust which add high cohesion to the concrete mixture. The best compressive strength was occurred when using WMP replacement ratio 10% of sand compared the control mix and the best positive effect of WMP was more pronounced at early ages [3&4]. The improvement in the properties and durability of hardened concrete using WMP as cement or sand replacement to the filling effects [5]. The optimum improvement of the mechanical properties and the of durability hardened concrete were occurred when using 20% and 10% of WMP replacement ratios of sand and cement respectively. Also, using WMP improved the ultrasonic velocity (UPV), carbonation resistance and microstructure of concrete [6]. Using 15% of WMP replacement of sand with using metakaolin additions and Silica Fume exhibited the durability properties of hardened concrete [7]. Sevket concluded that using recycled glass sand (RGS) and WMP gave similar fresh properties; and decreased the compressive strength by 10%-19% compared to control mixes. And using (RGS) and WMP improved other properties such as sound insolation, thermal conductivity, and water permeability of concrete mixes [8]. Using diatomite and WMP replacement of cement affected on the mechanical properties of concrete, the optimum compressive and flexural strength were obtained.
when using diatomite and WMP replacement ratios of 10% and 5% of cement respectively [9]. Using WMP replacement ratios up to 15% as an admixture has better performance in the concrete with using limestone waste as fine aggregate. Using 15% of WMP with limestone waste (LSW) up to 50% improved the compressive strength, indirect tensile and flexural strength by 7%, 17% and 8% respectively [10]. Using WMP replacement ratio of 5% of cement improved fresh, hardened properties of concrete, and the behavior of RC slabs as the initial crack, the ultimate load values, the stiffness, and the ultimate strength were improved compared to the control slabs [11]. The improvement in the mechanical properties and microstructural of concrete were occurred by using WMP or waste marble slurry replacement of cement [12&13&14]. The optimum compressive and thermal strength were obtained when using WMP replacement ratio of 10%. Using WMP improved and controlled of Alkali-Silica Reaction (ASR) extension leading to durable concrete. Scanning microscope electronic (SME) images didn’t display any signs of cracking in mortar mixes cast with WMP compared to the control mixes. However, energy dispersed X-ray spectroscopy had shown that the alkali amounts decreased after using WMP [15]. The mechanical performance of concrete improved when using WMP and silica fume replacement ratio of less than 10% of cement [16]. Using 40% of WMP replacement of sand enhanced of strength and permeation properties, but it decreased the workability, the chloride ion penetration resistance, and water absorption. The microstructural analysis detected that the increased density of concrete matrix refers to decreasing pores as both the physical and chemical properties of concrete matrix change due to using WMP [17]. Successful enhancement of the workability and the mechanical properties of high-performance concrete were detected due to the effect of WMP and glass powder (GP) was reported by Belouadah et al. [18]. Using WMP replacement of cement up to 15% enhanced the long-term strength and durability performance (water permeability, abrasion resistance, and water abrasion). WMP produced more dense mixture for higher replacement ratios, thus enhanced the long-term properties. Extracting relationships between strength and durability properties with compressive strength occurred for concrete with WMP that were concluded by Singh et al. [19]. The effects of using WMP on the properties of different concretes were reported by Zhang et al. When using SF and WMP replacement ratios of 10% and 5–20% respectively from cement in cellular concrete had the optimum mechanical and durability properties with H2SO4 attack. Scanning electron microscopy images showed that using (10% SF) and (10% SF+5% WMP) increased the resistance to MgSO4 and H2SO4 attack [20]. Using Taguchi method, the addition of WMP increased the resistance to high temperature due to modification of aggregate/ cement paste interface zones were investigated by Keleştemur et al. [21]. Especially, using (WMP) for self-compacting concrete has increased the compressive strength at the same workability level by using superplasticizer admixture compared to the control mixes [22]. Increasing the content of natural pozzolana and WMP improved the workability of SCC, but it decreased the compressive strength [23]. Gesoğlu et al. indicated that high replacement of (WMP) and limestone filler (LF) adversely affected the fresh properties of the SCCs, but they were improved by adding fly ash. Using (LF) improved and (WMP) reduced the mechanical and transport properties of the SCCs [24]. When using WMP replacement of cement or aggregate is convenient to enhance some hardened properties of the conventional concrete [25]. Using WMP additives ratio up to 50% by weight of cement could be used successfully as mineral additives in the production of SCC. Using mixed powder improved the performance in SCC. However, silica fume enhanced marble powder performance in SCC [26]. Using additive ratios WMP/C affected on the compressive strength by 15%. The maximum compressive strength of an eco-efficient SCC occurred at w/c-ratio less than 0.55 and the marble powder-to-cement-ratios of up to 0.6 [27]. Using WMP replacement ratios up to 15% had increased the mechanical properties and the durability (chloride ions and oxygen permeability) of HPC. The additive ratios WMP gives very powerful nucleation and contributes successfully to the decrease of chloride ion penetration and oxygen permeability and the durability of concrete [28]. This research aims to studying the microstructural characteristics and properties of hardened concrete made with WMP as partial replacement of cement.

2. Experimental Program

The experimental program in this research was performed in the laboratory of testing of building materials at the Faculty of Engineering, Menoufia University, as well as Housing and Building National Research Center, Egypt. The aim of this research is to spotlight the properties and microstructural characteristics of concrete cast with the marble powder as a partial replacement of cement. Seven concrete mixes were cast and tested to determine the microstructure and the properties of hardened concrete made with marble powder as partial replacement of cement. Cubes (10×10×10 and
15×15×15 cm.), cylinders (10×20 and 15×30 cm.) and prisms (10×10×50 cm.) were cast and tested to determine the compressive, indirect tensile, flexural modulus, bond strength and the modulus of elasticity.

2.1 Materials
Ordinary Portland cement, type (CEMI 42.5 N) produced by the Lafarge Cement Factory was used. Its chemical and physical characteristics satisfied the Egyptian Standard Specification “E.S.S. 4756-1/2013 (2013)”[29]. Natural siliceous sand satisfying the requirements “E.S.S. 1109/2008 (2008)” and “E.C.P. 203/2018 (2018)”[30 & 31], free from impurities with a specific gravity 2.5 t/m$^3$ and a fineness modulus of 2.53 was used. Well graded crushed dolomite was used. Its grading satisfies the limits of “ASTM C33 (2003)”[32]. The particles were irregular and angular with a very low percentage of flat particles. The delivered crushed dolomite size 1 had a maximum nominal size of 12.5 mm. Figures (1) and (2) show the grading of the sand and the dolomite used.

A high range water reducer (HRWA) as a superplasticizer was used. It is produced by CMB Group under the commercial name of Addicrete BVF. It was used to improve the workability of concrete mixes. Its characteristics satisfy the requirements of “ASTM C494 (2015) (type A and F)”[33]. It is a brown liquid having a density of 1.18 kg/liter at room temperature. The amount of HRWA was 2.0 % of the cement weight in all mixes. Silica fume as a pozzolanic admixture was used (10 % of the cement weight). It contains silica of about 95% in powder form, its specific gravity is 2.3t/m$^3$ as provided by the manufacturer. The marble Powder used was produced by the processing out of sawing and polishing of marble stones. Marble powder from Shaq Al-Thoaban is brought by Egyptian factories of marble companies. Its physical properties and chemical analysis are shown in Table (1).

Table (1): The chemical and physical properties of waste marble powder [26]

<table>
<thead>
<tr>
<th>Composition</th>
<th>Average %</th>
<th>Physical Composition</th>
<th>Average %</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\text{SiO}_2$</td>
<td>2.72</td>
<td>Specific Surface Area (m$^2$/g)</td>
<td>2.42</td>
</tr>
<tr>
<td>CaO</td>
<td>76.3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>MgO</td>
<td>1.11</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\text{O}_2\text{Al}$</td>
<td>0.46</td>
<td>Specific Gravity (t/m$^3$)</td>
<td>2.78</td>
</tr>
<tr>
<td>$\text{O}_2\text{Fe}$</td>
<td>0.39</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\text{SO}_4$</td>
<td>0.32</td>
<td></td>
<td></td>
</tr>
<tr>
<td>O$_2$K</td>
<td>0.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>LOI</td>
<td>17.9</td>
<td>Color</td>
<td>White</td>
</tr>
<tr>
<td>$\text{O}_2$P</td>
<td>0.09</td>
<td></td>
<td></td>
</tr>
<tr>
<td>O$_2$Na</td>
<td>0.34</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

High Tensile Steel Rebars produced from Ezz Al Dekheila Steel Factory, Alexandria was used. The steel bars are high strength steel (steel 52) and 16 mm diameter and 16 cm length were used as embedded rebars in 15 cm concrete cubes to determine the bond strength. Its characteristics satisfy the requirements of "E.S.S. 262/2011 (2011)”[34].

2.2 Concrete Investigation
Seven concrete mixes were designed and cast to investigate the effect of WMP replacement ratios 0, 5, 10, 15, 20, 30 and 40% of cement weight on the hardened properties and the concrete microstructure. The samples were mixed and cast in steel cubes (10×10 ×10 and 15×15×15 cm), cylinders (10×20 and 15×30 cm.) and prisms (50×10×10 cm.) after oiling their surfaces. The concrete samples were placed on the vibration table at a low speed for complete compaction. After casting the specimens, they were covered with wet burlap in the laboratory at 24°C and 68% relative humidity. The proportions of the concrete mixes used are shown in Table (2).
Table (2): The proportion of the concrete mixes

<table>
<thead>
<tr>
<th>Mix</th>
<th>WMP/C %</th>
<th>Cement (Kg/m)</th>
<th>WMP (Kg/m)</th>
<th>Dolomite (Kg/m)</th>
<th>Sand (Kg/m)</th>
<th>Water (Kg/m)</th>
<th>Silica Fume (Kg/m)</th>
<th>Ad. (Kg/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>0</td>
<td>400</td>
<td>0</td>
<td>1045.4</td>
<td>696.9</td>
<td>168</td>
<td>40</td>
<td>8</td>
</tr>
<tr>
<td>WMP 5</td>
<td>5</td>
<td>380</td>
<td>20</td>
<td>1044.1</td>
<td>696.1</td>
<td>168</td>
<td>40</td>
<td>8</td>
</tr>
<tr>
<td>WMP 10</td>
<td>10</td>
<td>360</td>
<td>40</td>
<td>1042.8</td>
<td>695.2</td>
<td>168</td>
<td>40</td>
<td>8</td>
</tr>
<tr>
<td>WMP 15</td>
<td>15</td>
<td>340</td>
<td>60</td>
<td>1041.5</td>
<td>694.3</td>
<td>168</td>
<td>40</td>
<td>8</td>
</tr>
<tr>
<td>WMP 20</td>
<td>20</td>
<td>320</td>
<td>80</td>
<td>1040.2</td>
<td>693.5</td>
<td>168</td>
<td>40</td>
<td>8</td>
</tr>
<tr>
<td>WMP 30</td>
<td>30</td>
<td>280</td>
<td>120</td>
<td>1037.6</td>
<td>691.7</td>
<td>168</td>
<td>40</td>
<td>8</td>
</tr>
<tr>
<td>WMP 40</td>
<td>40</td>
<td>240</td>
<td>160</td>
<td>1035.0</td>
<td>690.0</td>
<td>168</td>
<td>40</td>
<td>8</td>
</tr>
</tbody>
</table>

(Scanning Electron Microscope (SEM 2.3)
A scanning electron microscope test was done to examine the microstructure of concrete mixes. The microstructure of the concrete mixes was examined by conducting a scanning electron microscope test on broken specimens after indirect tensile strength tests at 28 days. In this test, the samples are cut into small cubes or slices (1 x 1 cm) or less to fit in the device room. In this test, the samples were enlarged to ×12000 as showed in Fig. (3)

3. Analyses and Discussion of Test Results
Figures (4) to (9) show the results of the effect of using WMP as a partial replacement of cement weight (0, 5, 10, 15, 20, 30, and 40%) on the properties of hardened concrete.

3.1. Compressive Strength
Figure (4) shows the comparison between the compressive strength of different concrete mixes using WMP as partial replacement of cement compared to the control mix at different ages of the test. At 7-day age, the compressive strength increased by 18, 42, 27, 20, and 12% when using WMP replacement ratios of 5, 10, 15, 20, and 30%, respectively compared to the control mix. But, using WMP replacement ratio of 40%, the compressive strength decreased to 17% compared to the control mix. At 28-day age, the compressive strength increased by 14, 32, 17, 12, and 2% when using WMP replacement ratios of 5, 10, 15, 20, and 30%, respectively compared to the control mix. But, using WMP replacement ratio of 40% the compressive strength decreased by 23% compared to the control mix. At 56 days age, the compressive strength increased by 14, 26, 14, 8, and 4% when using WMP replacement ratios of 5, 10, 15, 20, and 30%, respectively compared to the control mix. While, using WMP replacement ratio of 40% the compressive strength decreased by 25% compared to the control mix. The above results shows that the optimum WMP replacement ratios were 10 percent in terms of compressive strength. The strength gain was higher at 7-days and reduced with time. While the strength tended to increase up to 30% replacement ratios, the strength remarkably decreased at a replacement ratio of 40%.

3.2. Indirect Tensile Strength
Figure (5) shows the comparison between the indirect tensile strength of different concrete mixes using WMP as partial replacement of cement compared to the control mix at different ages of the test. At 7-day age, the indirect tensile strength increased by 19, 40, 29, 16, and 10% when using WMP replacement ratios of 5, 10, 15, 20, and 30%, respectively compared to the control mix. But, using WMP replacement ratio of 40%, the indirect tensile strength decreased to 18% compared to the control mix. At 28-day age, the indirect tensile strength increased by 14, 34, 20, 12, and 3% when using WMP replacement ratios of 5, 10, 15, 20, and 30%, respectively compared to the control mix. But, using WMP replacement ratio of 40% the indirect tensile strength decreased by 23% compared to the control mix. At 56 days age, the indirect tensile strength increased by 14, 28, 16, 8, and 3% when using WMP replacement ratios of 5, 10, 15, 20, and 30%, respectively compared to the control mix. While, using WMP replacement ratio of 40% the indirect tensile strength decreased by 25% compared to the control mix. The above results shows that the optimum WMP replacement ratios were 10 percent in terms of indirect tensile strength. The strength gain was higher at 7-days and reduced with time. While the strength tended to increase up to 30% replacement ratios, the strength remarkably decreased at a replacement ratio of 40%.
3.2. Indirect Tensile Strength
Figure (5) shows the comparison between the indirect tensile strength of different concrete mixes using WMP as a partial replacement of cement compared to the control mix at age of 28 days of the test. The indirect tensile strength was increased by 9, 18, 9 and 1% when using WMP replacement ratios of 5, 10, 15 and 20, respectively compared to the control mix. But, using WMP replacement ratios of 30 and 40% the indirect tensile strength decreased by 8 and 48%, respectively compared to the control mix. By evaluating the performance of the mixes in compressive strength and splitting tensile strength, it can be observed that the percentage increasing of compressive strength is higher than the increase in the splitting tensile strength for all mixes at 28 days. After analyzing the results, the ratio between the indirect tensile and compressive strength is 9% and 8% at control mix and for WMP replacement ratio of 10, respectively.

3.3. Flexural Modulus
Figure (6) shows the comparison between the flexural modulus of different concrete mixes using WMP as a partial replacement of cement compared to the control mix at age of 28 days. The flexural modulus was increased by 17, 40, 20 and 3% when using WMP replacement ratios of 5, 10, 15, 20 and 30% respectively, compared to the control mix. But, using WMP replacement ratio of 40% the flexural modulus decreased by 22% compared to the control mix. The observed reductions were attributed to the increase of the fines content.

3.4. Bond Strength
Figure (7) shows the comparison between the bond strength of different concrete mixes using WMP as a partial replacement of cement compared to control mix at age of 28 days. The bond strength was increased by 5, 20 and 7% at replacement ratios of WMP as 5, 10 and 15%, respectively. As the WMP% increased by 20, 30 and 40%, the bond strength decreased to 4, 17 and 57%, respectively compared to the control mix.

3.5. Modulus of Elasticity
Figure (8) shows the comparison between the modulus of elasticity of different concrete mixes using WMP as partial replacement of cement compared to the control mix at age of 28 days. The modulus of elasticity increased by 7, 15, 8, 6 and 1% when using WMP replacement ratios of 5, 10, 15, 20, and 30%, respectively compared to the control mix. But, using WMP replacement ratio of 40% the modulus of elasticity decreased by 12% compared to the control mix.
3.6. Scanning Electron Microscope (SEM) Test

Scanning electron microscope was used to analyze the microstructural characteristics of the concrete mixes. SEM images of fractured concrete samples from concrete mixes with different percentage of marble waste (0, 5, 10, 15, 20, 30 and 40%) as cement and cured to 28 days have been taken and shown in Figure (9). While, all the micrographs show pore voids, the considerable density of the mix containing waste marble can be observed in SEM images, showing the increase of filler and decreasing the cement content effect caused by the inclusion of waste marble. SEM photos show the microstructure of all concrete mixes. Thus, it is observed that increasing marble powder to 10% decreased the pore voids. Improving the microstructure of mixes improved the hardened properties mixes containing 5 and 10 % of marble powders. Therefore, the improvement in mechanical properties of the mixes can be explained by the filling effect of waste marble and the densification of the concrete matrix up to 10% of marble powder.

Then, it is observed that increasing marble powder up to (10% to 40%) of cement changes the microstructure of concrete mixes by increasing the voids in concrete and thus the strength was reduced. From the test results, it is observed that the hardened properties are directly proportional to the compressive strength of concrete and the concrete mixes microstructure. WMP can fill up the pores in concrete to improve the density of the micro-structure of concrete mixes and consequently the strength.
4. CONCLUSIONS

Based on the obtained test results, the following conclusions can be drawn:

1. The tested concrete strength values in terms of compressive, tensile, and modulus of elasticity increased by increasing WMP ratio as a replacement of cement up to 10% of cement weight at different ages up to 56 days.

2. The strength either increased or remained unchanged up to 30% of Marable powder replacement.

3. The images of scanning electronic microscope showed that partial replacement of cement by WMP up to 10% led to filling of the internal pores yield denser microstructure.

REFERENCES


