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INCREASING THE CALCIUM SILICATE HYDRATE AMOUNT IN REACTIVE POWDER CONCRETE USING MARBLE POWDER

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ABSTRACT

This research aims to make ultra high strength Reactive Powder Concrete (RPC) with marble powder as one of the components. The use of marble powder can increase Calcium Silicate Hydrate (CSH) and the strength of RPC. The research method used to achieve the objectives is experimental and divided into two steps. The first step is the characterization of a marble and micro silica powder mixture. Stoichiometry calculations are performed to determine the composition of the mixture. The test sample is made in the form of pellets consisting of a mixture of marble powder, micro silica, and water. The water content used is at 30% - 50%. Maintenance is carried out by immersion in water with the temperature of 20°C for 27 days and in steam at temperatures 200°C, 250°C, and 300°C with 2 atm pressure for 4 hours. Material characterization is carried out using X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). The second step in this research is the RPC compressive strength test. The test sample is made in the form of a cube measuring 50 x 50 x 50 mm. This cube is a mixture of water, cement, micro silica, marble powder, sand, and superplasticizer. Material composition is arranged based on the characterization of the sample pellet test, and maintenance of the sample cube test is carried out as in the sample pellet test.

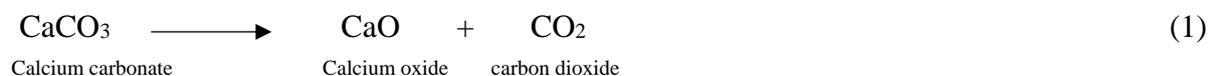
Keywords: CSH, RPC, strength, temperature, characterization

INTRODUCTION

The marble is a rock that resulted from a metamorphosis or a process made from limestone which contains carbonate. As a result of recrystallization of the structure from which the rock originates, it forms a new texture and grain regularity. Indonesian marble is estimated to be around 30-60 million years old. In Indonesia, the usage of marble is quite widespread. The use of marble can be categorized into two motifs: namely ordinario and staturio. Ordinario marble is usually used for manufacturing baths, tables, and walls, while staturio is often used for sculpture.

The processing and utilization of new marble use water, so it produces waste. This waste is accommodated in a pool (reservoir), which gradually settles then hardens. Marble waste can be used so as not to pollute the environment and economic value. Marble waste can be processed with dolomite as a mixture of building materials to reduce environmental pollution. The effects of using dolomite and waste marble powder as partial replacement of cement on the mechanical properties of concrete were investigated. Test results indicate that the optimized assay of dolomite and waste marble powder as a replacement by weight of cement had the best compressive and flexural strengths [1].

Marble has the main content of the element calcium, which binds water to form compounds in the form of calcium hydroxide, chemically written as $\text{Ca}(\text{OH})_2$. Calcium has several unique properties: chemically, calcium is alkaline. So, it quickly forms reactions with strong acids. Calcium in the calcium carbonate compound CaCO_3 releases carbon dioxide on heating to form calcium dioxide, which is usually called lime, and chemically written as follows [2].



Reactive Powder Concrete (RPC) is a concrete composed of materials with microscopic particle sizes, such as cement, micro silica, quartz sand, quartz powder, and superplasticizer. RPC is concrete with excellent mechanical properties: the highest compressive strength ever achieved in making RPC ranges from 120.0 - 150.0 MPa, and the highest flexural strength ever achieved ranges from 20.0 - 30.0 MPa [3]. RPC is characterized by the use of cement and micro-silica in large quantities and does not use coarse aggregate. Because its materials have tiny particle sizes, RPC can be arranged in a compact and has a much better homogeneity when compared to concrete in general. Micro silica in RPC is a material mainly consisting of amorphous SiO_2 . The SiO_2 content in micro silica reaches 98%, far more compared to volcanic ash, flying ash, or other pozzolanic material, which only ranges between 40% - 56% [4]. Micro silica has functioned as an activator for the occurrence of pozzolanic reactions with one of the cement hydration products, namely calcium hydroxide. It also functions to fill small voids in concrete.

Research conducted on RPC [3] still shows weaknesses, one of which is covered by quartz powder. Quartz Powder in the making of RPC has a function as a pore filler. In a tiny size, high pressure, and high temperature during RPC maintenance, quartz flour can react with C-S-H to form xonolite so that the compressive strength of RPC can increase significantly [5]. In Indonesia, quartz powder is not easy to get. In order to obtain it, imports from China, India, or Europe are needed, so sustainability in making RPC becomes a huge obstacle. This research will use marble powder instead of silica powder in the manufacturing of RPC. The first reason for using marble powder instead of silica powder in making RPC is that marble powder contains calcium as its main component. This calcium is expected to react with micro silica in the pozzolanic reaction to form calcium silicate hydrate compounds. The higher volume of calcium silicate hydrate is formed, the more the strength of RPC will increase, as explained above. Secondly, marble powder is a readily available material in Indonesia and a waste from marble production plants. Thus, marble powder waste as an RPC maker material can reduce environmental pollution [6].

RESEARCH METHOD AND MATERIALS

To achieve the objectives, the research method used in data collection is experimental research in which several tests, treatments, and characterization methods are carried out to determine the amount of calcium silicate formed based on the reaction between marble powder and micro silica, using the following research steps.

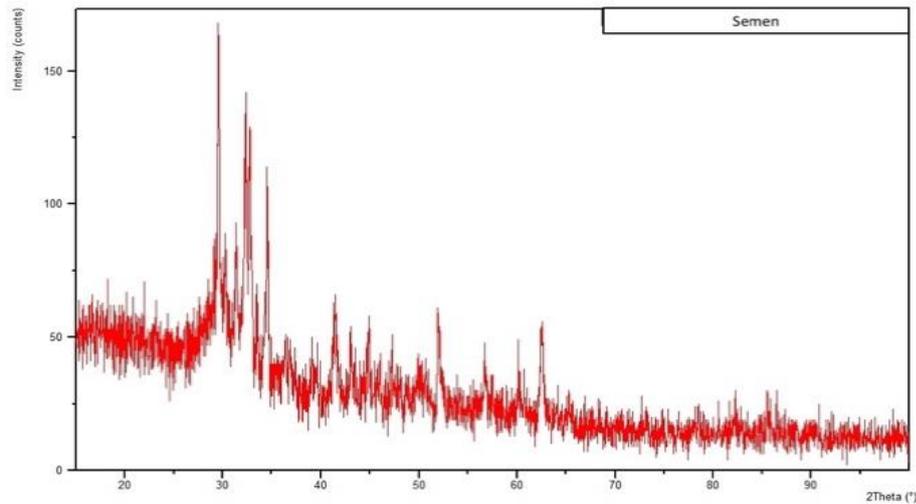
The material used in this research is cement, water, silica fume, marble powder, sand, and superplasticizer with a weight ratio of 1.00 : 0.20 : 0.25 : 0.15 : 1.10 : 0.03. Samples are made by mixing all materials into a mixer which is rotating at a speed of 1500 rpm. Samples were cast on the mold with a size of 5.0 cm x 5.0 cm x 5.0 cm and were treated by soaking in water at a temperature of 20°C for 27 days, followed by treatment at 200°C as mix-1; 250°C as mix-2; and 300°C as mix-3 in an autoclave for 4 hours and with 2 atm pressure. All samples were compared with a typical mix (without marble powder) examined using XRD, SEM, and compressive strength testing was performed.

Cement is used as a binding agent between the constituent RPC constituents. The cement used is portland cement type 1. Silica fume does not have atoms, has no common configuration, or is called amorphous. The size of the granules ranges between 0.1 - 0.2 μm . Silica fume contains 98% of SiO_2 , with fineness of 20,000 m^2 / kg and density of 2,200 kg / m^3 [4]. The sand used is quartz sand, which has SiO_2 as a major composition, 150 μm - 600 μm grain size, zone 4 on grain grading, irregular shape, and hardness of 7 mohs. Marble mainly contains calcium, which binds water to form compounds in calcium hydroxide, chemically written as $\text{Ca}(\text{OH})_2$. Each marble powder granule size ranges between 10 μm - 40 μm [6]. Superplasticizer is used to improve the workability of the mortar so that it can disperse the mortar well. The superplasticizer suitable for this research comes from polycarboxylate-based polymers.

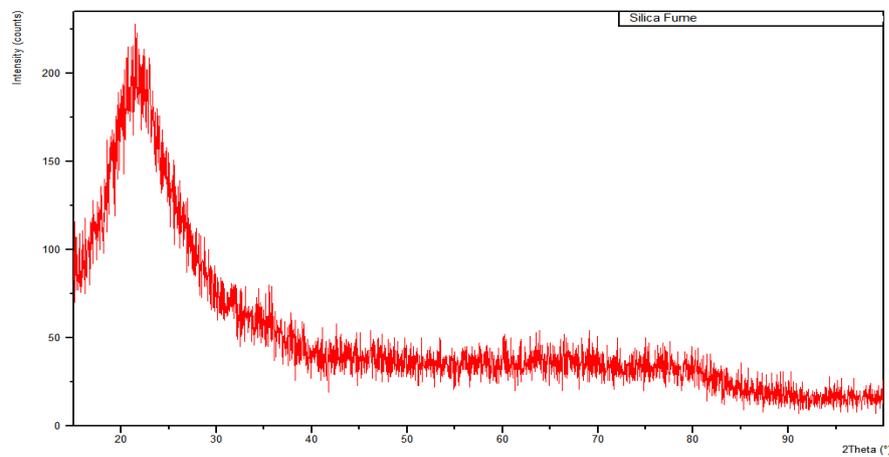
RESULTS AND DISCUSSION

XRD Characterization

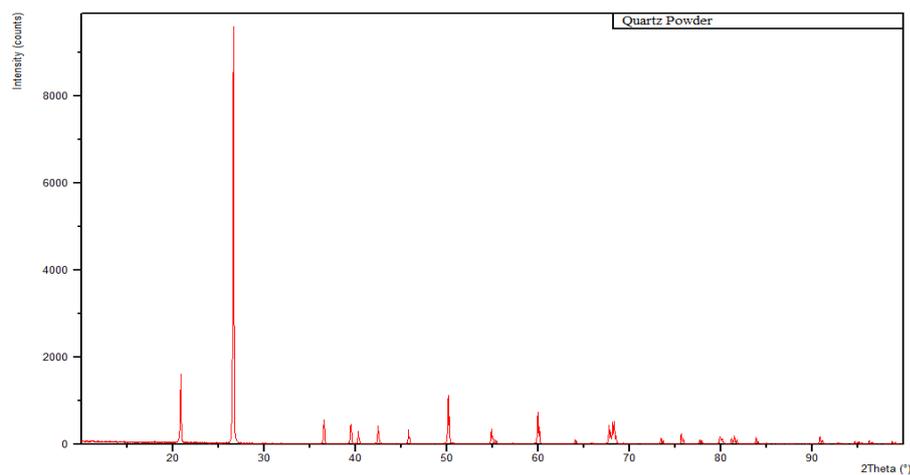
In the XRD spectrum of raw materials for cement, silica fume, and marble powder, as shown in FIGURE 1, the spectrum peaks at certain angles depict the mineral components contained therein. FIGURE 1a shows the mineral component of tricalcium silicate (C_3S) found in the diffraction angle $2\theta = 32.15^\circ, 32, 50^\circ, 34, 37^\circ$ and 41.11° . The minerals in calcium silicate (C_2S) are present at the diffraction angle $2\theta = 32.66^\circ, 33.11^\circ$ and 41.63° . Tricalcium silicate is at the diffraction angle $2\theta = 17.50^\circ, 39.40^\circ$ and 44.57° , while the mineral component of calcium aluminoferrite (C_4AF) is at the diffraction angle $2\theta = 34.38^\circ, 50,56^\circ$ and 50.74° [7]. The four minerals are the main components of cement detected in the XRD spectrum. The XRD spectrum of Silica fume is shown in FIGURE 1b. This spectrum represents amorphous material, meaning that the silica fume of the atoms is not regularly arranged [4]. This amorphous material is very reactive to calcium hydroxide $Ca(OH)_2$ at room temperature, so it is widely used in the concrete industry. The XRD spectrum of quartz is presented in FIGURE 1c, which shows the crystalline spectrum of SiO_2 calcium silica with diffraction angles $2\theta = 20.82^\circ, 26.62^\circ$ and 36.52° [8]. By finding only three diffraction angles 2θ , the type of material can already be described.



1a.



1b.

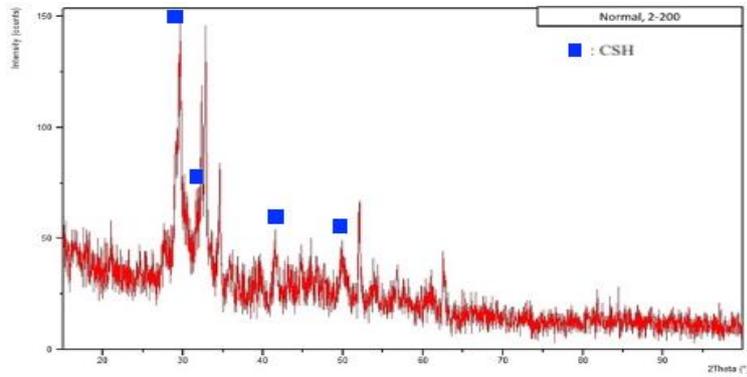


1c.

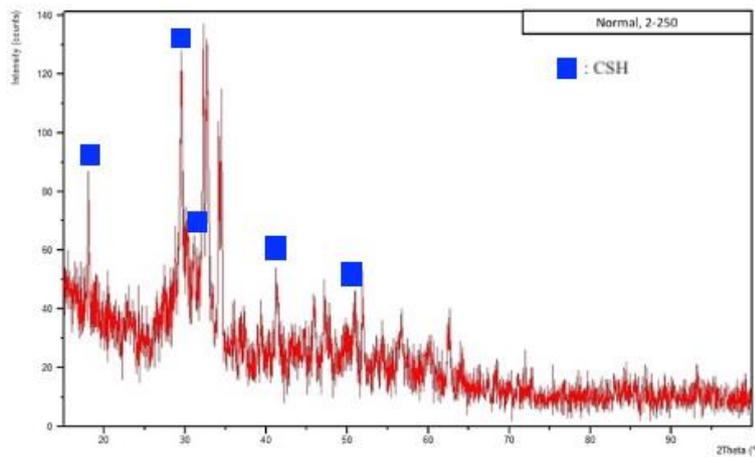
FIGURE 1. XRD Patterns of: a. Cement, b. Silica Fume, c. Silica sand.

This research is conducted on concrete with typical composition, which means that the concrete is only composed of cement, water, silica fume, and superplasticizer. The XRD

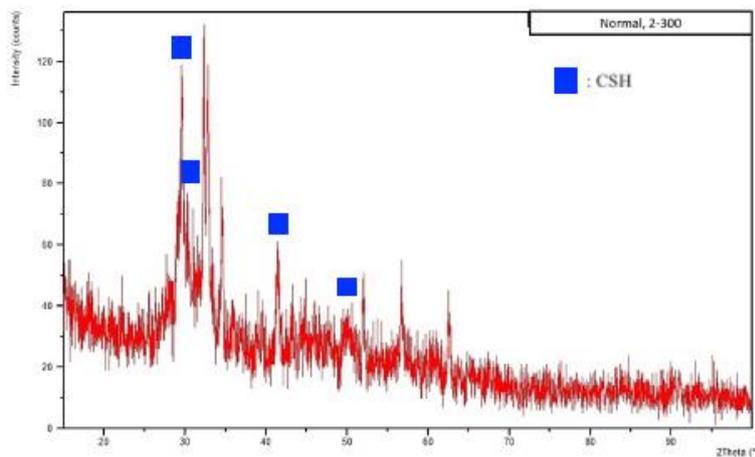
spectrum of concrete with a standard composition is shown in FIGURE 2. This spectrum clearly shows the presence of calcium silicate hydrate or known as (CSH) [9], at a diffraction angle of 2θ 16.9° , 29.2° , 32.0° , 42.7° , 49.8° .



2a.



2b.

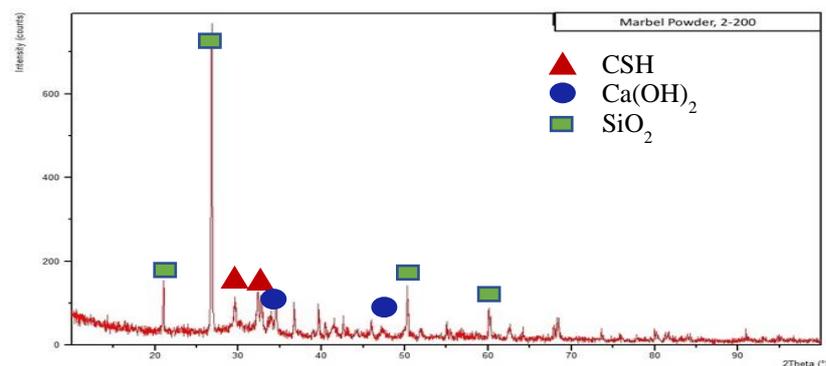


2c.

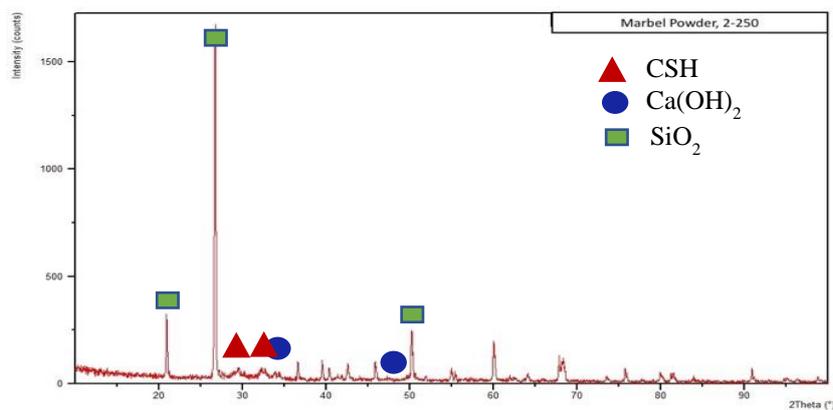
FIGURE 2. XRD Patterns of the standard mix at temperatures: a. 200°C, b. 250°C, c. 300°C.

Examination using X-ray diffraction in mix-1, mix-2, and mix-3 samples is shown in FIGURE 3. In FIGURE 3, the intensity of the quartz spectrum dominates the appearance of other

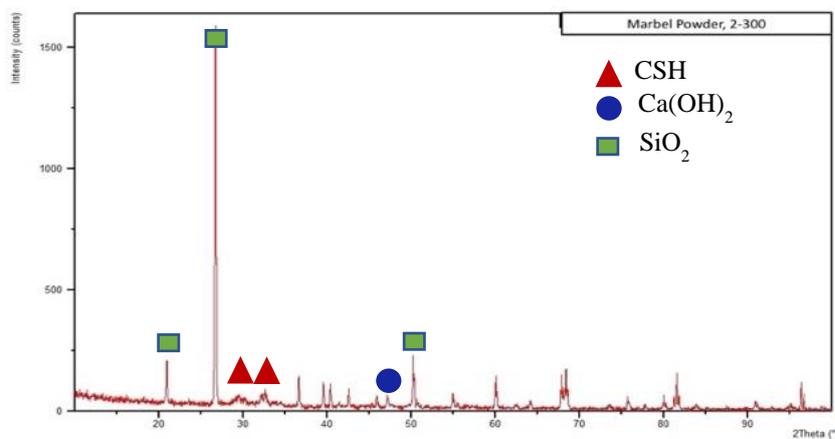
spectra, such as the CSH spectrum. However, the CSH spectrum is still evident in the position of the material. FIGURE 3a shows the CSH spectrum, which is much clearer when compared to the mix-2 spectrum of FIGURE 3b and the mix-3 spectrum of FIGURE 3c. This shows that maintenance using an autoclave at 200°C can increase CSH formation compared to maintenance at 250°C and 300°C. The appearance of the XRD spectrum in FIGURE 3 also shows the use of quartz flour in the process of making reactive powder concrete can increase the formation of CSH in concrete. CSH in concrete has a crucial function, which is to form strength. The CSH spectrum is not the same as the SiO₂ spectrum because CSH is a semi-crystal and not a pure crystal.



3a.



3b.



3c.

FIGURE 3. XRD Patterns of concrete sample: a. mix-1, b. mix-2, c. mix-3.

SEM Analysis

The formation and distribution of hydration products of hydrated cement paste with three different mix proportions are pictured below. The microstructure of the three mixes was examined and compared with the normal mix. The microstructure and strength properties of all three mixes were correlated based on the hydration products formed after 28 days. The reason behind the strength of the concrete was analyzed and explained based on the growth of hydration products in the microstructure of concrete mixes.

The SEM micrograph pictures in FIGURE 4 illustrate the microstructural variation during the hydration process and production of hydration products in Mix 1. The rate of hydration was sufficient to achieve the strength of concrete. C-S-H formation was complete. The development of chemical reaction between the portlandite from marble powder $\text{Ca}(\text{OH})_2$ and silica in the silica fume results in the concrete having inadequate strength. In the SEM micrograph, the lower presence of ettringite was clearly evidenced in the micrograph, which leads to the increase of development and distribution of C-S-H gel in the hardened concrete.



FIGURE 4. SEM Micrograph of Mix-1

The microstructure of the hydrated cement paste of Mix-2 was shown in FIGURE 5. The considerable strength obtained in this mix was due to the pozzolanic activity of silica fume. The chemical reaction of silica fume with the portlandite $\text{Ca}(\text{OH})_2$ leads to the production of additional CSH gel, which was the main reason for the strength of this mix [10,11,12]. The rate of hydration process in Mix-2 was similar to the typical concrete mix, but the existence of mineral elements was quite different from Mix-3, which manipulates the strength of concrete curing temperature. Even though the strength of the concrete mix was not up to the anticipated level, it reached a considerable strength, which means it can also be used as a replacement for the normal concrete mix in certain concreting works.

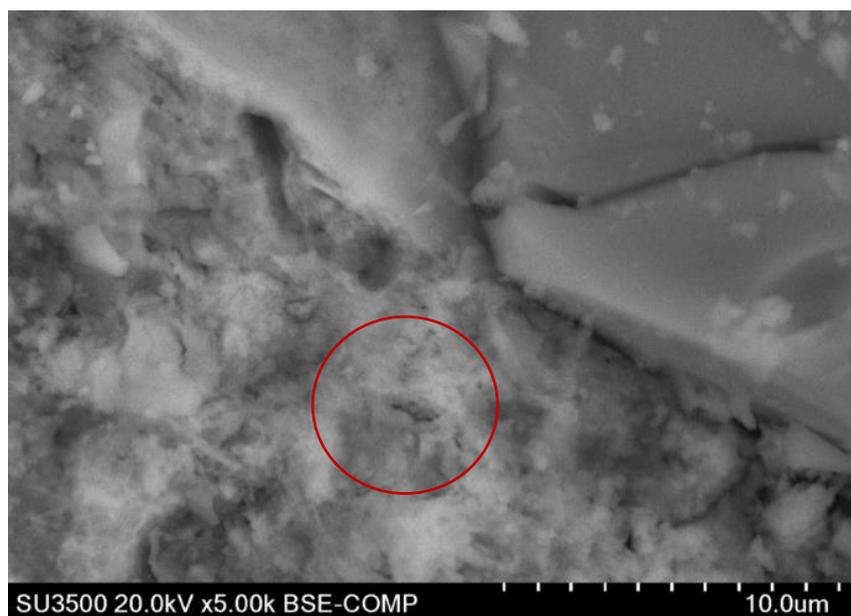


FIGURE 5. SEM Micrograph of Mix-2

In FIGURE 6, The SEM image of Mix-3 visualizes the microstructure of hydrated cement paste. From the SEM micrograph of the hardened concrete, the distribution of CSH was nearly decreased due to elevated curing temperature. In this mix, the range of CSH development was lower, which may be due to unreacted particles of marble powder present in the mix. The accumulation of other major mineral elements, such as Portlandite $\text{Ca}(\text{OH})_2$ and Calcite (CaCO_3) crystals, are found in the microstructure of the mix. The main reason for the decrease in strength was the lack of hydration of particles, which is present in the hardened cement matrix. In other words, the amount of CSH formed is lower than in mix-1. The utilization of supplementary materials such as marble powder in the mix was unsuccessful because of the lack of chemical reaction between marble powder and the silica fume in curing temperature [6]. In this mix, due to the unreacted particles in the hardened cement paste, the strength of concrete mix was affected.

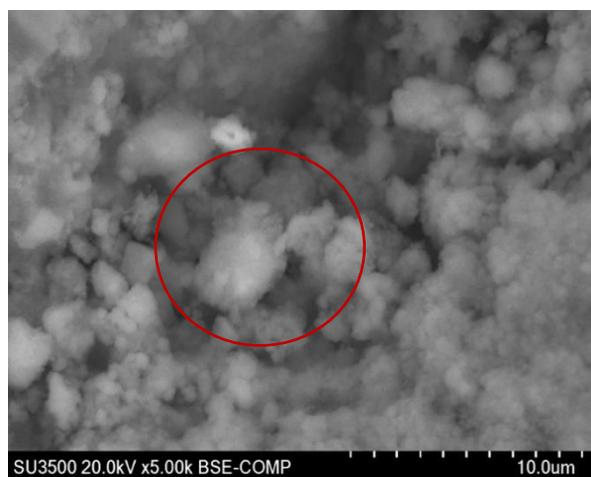


FIGURE 6. SEM Micrograph of Mix-3

Compressive Strength

Compressive strengths of four concrete mixes were determined after 28 days, and their comparison details are pictured in FIGURE 7. The concrete cubes were tested for compressive strength after 28 days of curing, and a higher value of compressive strength was obtained in Mix-1, which was slightly higher than mix-2. Even though Mix-3 did not reach the target strength, 50,0 MPa, it reached enough strength to be used as an alternative for the standard concrete mix in specific concreting works.

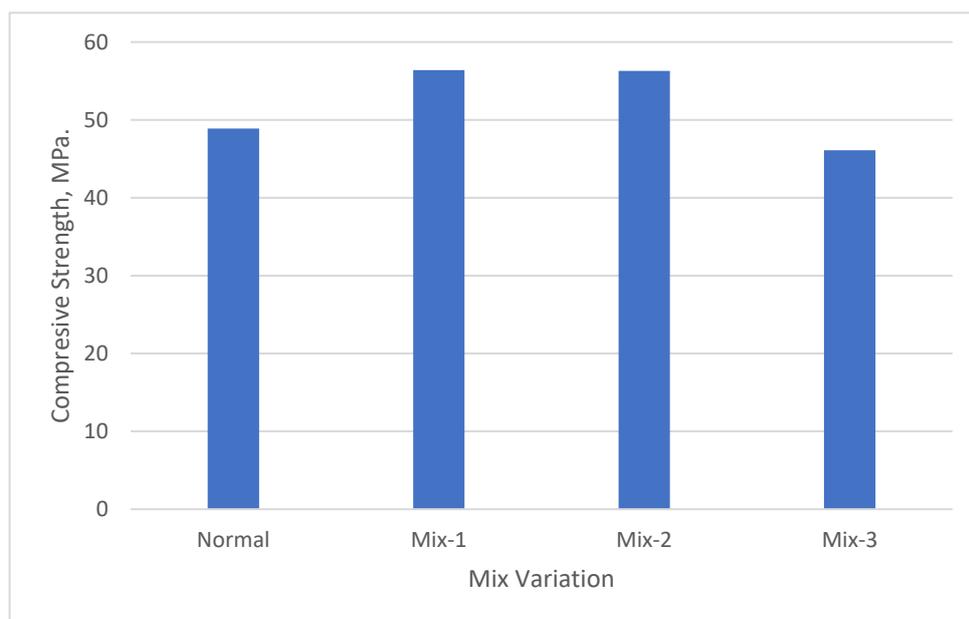


FIGURE 7. Concrete Compressive Strength

CONCLUSION

The conclusion obtained from the experimental study is that the addition of marble powder to reactive powder concrete (RPC) can increase the amount of calcium silicate hydrated (CSH). The increase in the amount of calcium silicate hydrated occurred due to the pozzolanic reaction between silica fume and marble powder. The rate of the pozzolanic reaction can be increased by applying pressure and increasing the temperature during the curing process.

Increasing the amount of hydrated calcium silicate in reactive powder concrete increases the compressive strength of the concrete. The increased amount of hydrated calcium silicate on RPC with the addition of marble trunks can be maximized if RPC is cured using an autoclave at a pressure of 2 atm and a temperature of 250°C for 4 hours.

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