



Öğütülmüş Diyatomitin Kendiliğinden Yerleşen Harçların Erken Dayanımına Etkisi

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Geliş Tarihi: 14.05.2024
Kabul Tarihi: 20.08.2024

Düzeltilme Tarihi: 01.08.2024

doi: 10.62520/fujece.1484058
Araştırma Makalesi

Alıntı: B. Karabulut, M. Ş. Yön ve M. Karataş, “Öğütülmüş diyatomitin kendiliğinden yerleşen harçların erken dayanımına etkisi”, Fırat Üni. Deny. ve Hes. Müh. Derg., vol. 3, no 3, pp. 350-361, Ekim 2024.

Öz

Çimento üretimi doğaya salınan karbondioksit miktarını arttıran önemli bir etkidir. Bu nedenle çimento yerine puzolanik özellik gösteren doğal ve atık malzemelerin kullanımı oldukça önemlidir. Bu makalede, çimento ile ikame edilebilecek doğal puzolanik bir malzeme olan diyatomit kayacının kendiliğinden yerleşen harç üretiminde kullanılabilirliği araştırılmıştır. Deneysel çalışmada, öğütülmüş diyatomitin kendiliğinden yerleşen harçlarda erken yaşta mekanik özelliklerine olan etkisini incelemek için 40×40×160 mm boyutlarında olan prizmatik numuneler; sırasıyla %0, %5, %10, %15, %20 oranında diyatomitin çimento ile değiştirilmesiyle üretilmiştir. kendiliğinden yerleşen harç elde etmek için çökme-akış testi, Avrupa Uzman Yapı Kimyasalları ve Beton Sistemleri Federasyonu kılavuzuna göre yürütülmüştür. %0, %5, %10, %15 ve %20 diyatomit kullanılarak hazırlanan numuneler 3 gün boyunca 23±2 °C sıcaklıktaki suda kürlenmeye tabi tutulmuştur. Kür süresi tamamlanan numunelerin 3 günlük (erken yaş) eğilme ve basınç dayanım değerleri elde edilmiştir. Bu deneysel çalışmanın sonucunda en yüksek dayanımların referans numuneleri de aşarak, %5 diyatomit içeren serilerde olduğu belirlenmiştir. Ayrıca karışımlardaki diyatomit oranının %5 ten fazla olmasıyla mekanik dayanımların azaldığı tespit edilmiştir.

Anahtar kelimeler: Öğütülmüş diyatomit, Kendiliğinden yerleşen harç, Mekanik özellikler, Erken dayanım

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İntihal Kontrol: Evet – Turnitin

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Effect of Ground Diatomite on Early Strength of Self-Compacting Mortars

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Received: 14.05.2024

Accepted: 20.08.2024

Revision: 01.08.2024

doi: 10.62520/fujece.1484058

Research Article

Citation: B. Karabulut, M. Ş. Yön and M. Karataş, "Effect of ground diatomite on early strength of self-compacting mortars", *Firat Univ. Jour. of Exper. and Comp. Eng.*, vol. 3, no 3, pp. 351-360, October 2024.

Abstract

Portland cement fabrication is a significant factor that increases the amount of carbon dioxide released into nature. For this reason, it is very important to use natural and waste materials with pozzolanic properties instead of portland cement. In this article, the usability of diatomite rock, a natural pozzolanic material that can be substituted with portland cement, in the manufacture of self-compacting mortar was studied. In the experimental study, prismatic specimens with dimensions of 40 × 40 × 160 mm were used to examine the impact of ground diatomite on the early age mechanical properties of self-compacting mortar; it was produced by replacing 0%, 5%, 10%, 15%, 20% of diatomite with portland cement, respectively. The slump-flow test to obtain self-compacting mortar was conducted according to the European Federation of Specialized Construction Chemicals and Concrete Systems guidance. Specimens prepared using 0%, 5%, 10%, 15% and 20% diatomite were cured in water at 23±2 °C temperature for 3 days. 3-day (early age) flexural and compressive strength worths were gained for the samples whose curing period was completed. As a result of this experimental study, it was specified that the highest strengths were in the series containing 5% diatomite, exceeding the reference samples. Additionally, it has been determined that mechanical strength decreases when the diatomite ratio in mixtures is more than 5%.

Keywords: Ground diatomite, Self-compacting mortar, Mechanical properties, Early strength

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1. Introduction

Portland cement (PC) production, an important building material, is increasing day by day [1]. PC consumption is approximately 1 ton per person per year in the world. PC, one of the basic components of concrete, constitutes 10-12% of concrete [2]. PC has a significant share in the construction cost. High temperatures occur during PC production and a significant amount of CO₂ is released in this process. While there is no need for manufacturing to obtain sand, it requires research and operation of quarries [3]. As the world population increases, the need for infrastructure has increased. In parallel with this, the need for natural aggregates is also increasing. For instance, while sand fabrication in China was 2500 million metric tons (2014), it reached 39.9 million cubic meters in Algeria [3]. Therefore, sand pits need to be protected by using alternative materials. Researchers have produced normal concrete or SCC using marble waste instead of PC or aggregate [4-6]. Also, the use of natural resources with pozzolanic properties that can replace PC is of great importance [7]. Pozzolanic materials that can be used instead of PC, in addition to offering both economic and environmental advantages, can also increase the properties of concrete such as preventing alkaline aggregate reactions and resistance to chemical environments [1].

Diatomite (D), which is among the different pozzolanic materials, is an abundant and cheap material with good quality reserves in Turkey [8]. The impact of D, which is used as an additive in concrete, on concrete/mortar has been the subject of research in recent years. Concrete samples were produced by substituting diatomite and WMP (waste marble powder) with cement by Ergun. As a result of the work, they obtained the best compressive and flexural strength in concrete samples containing 10% diatomite, 5% WPM and 5% WPM + 10% diatomite by weight. He also stated that the use of cement with different proportions of diatomite and WMP, separately and together with superplasticizer, gave positive results [9]. In their study, Zhang et al. investigated the usability of diatomite in recycled aggregate concrete (RAC). They found that the wide capillary pores in the specimens were diminished by using diatomite. They stated that there was an increase in both split tensile and compressive strengths by partially replacing up to 20% by weight OPC with diatomite [10]. Santos and Cordeiro examined the impact of grinding on developing the pozzolanic activity of small-porosity diatomite. They stated that the increase in the specific surface area (SSA) of diatomite increases the silica solubility and pozzolanic activity of diatomite [11]. Sarıdemir et al. In their study, they investigated the mechanical and microstructural characteristics of high-strength mortars (HSMs) produced with calcined diatomite powder (CDP) at ambient and high temperatures. The highest mechanical strength at ambient and high temperatures was obtained in HSM modified with 15% CDP (15CDP-HSM) [12]. Degirmenci and Yılmaz used diatomite as a partial substitute for cement. They conducted mechanical strength, sulfate resistance and freeze-thaw, dry unit weight and water absorption tests. According to the test results, they found a decrease in compressive and flexural strength as the diatomite ratio increased [13]. Xu et al. to produce hydraulic mortars, they used natural hydraulic lime (NHL2) (NHL) as aggregate and diatomite as a partial substitute for masonry waste powder (MWP). For NHL2, they used 0-20% diatomite with a 10% increase in weight. Specimens were produced with diverse water binder ratios (w/b). Diatomite substitution increased the mechanical strength of the hydraulic mortars produced. The change in diatomite ratio affected the compactness, porosity and strength of the mortars. Additionally, adding diatomite to the mortar improved the acid and sulfate resistance of the mortars [14]. Sun et al. used diatomite in their experimental studies to increase frost resistance. In the experimental results, they stated that the use of diatomite instead of silica fume significantly increased the freezing resistance of the mortar [15]. Koçak and Pınarcı conducted research on the hydration systems, compressive strength, physical and microstructural properties of cement pastes and mortars substituted with diatomite in different proportions. In the results, they found that portlandite content decreased with increasing age and diatomite addition. They stated that diatomite added cements had a higher degree of hydration and a higher amount of hydration products were formed in 28 and 90 days. This structure had a positive impact on the compressive strength of the mortars at later ages [16].

Self-compacting (consolidation) concrete (SCC) is a kind of concrete that can reach places where reinforcement placement is frequent and collapse under its own weight. SCC, which has high fluidity, maintains its stable structure without decomposition until it gains strength. It was developed by Okamura in Japan in 1986 to reduce workmanship errors in the compaction of concrete. SCCs have many positive features such as filling ability, good workmanship, construction time, occupational safety, noise reduction, design and

energy efficiency [17]. To produce fresh SCC with good mechanical properties and excellent processability without using vibration, basic parameters such as high passing ability and filling capacity need to be met. To ensure the solidity and combination of the mixture, a mixture that can produce a significant amount of powder material and/or change in viscosity is needed. Pozzolans are siliceous or siliceous aluminous materials that have little or no stringent properties when used alone, but can get stringent properties by reacting with calcium hydroxide $\text{Ca}(\text{OH})_2$ when finely ground [18]. About the use of diverse pozzolanic materials such as silica fume [19, 20], slag [21, 22], metakaolin [23], [24], fly ash [25], marble powder [26], [27], pumice powder [28, 29] in self-compacting mortars (SCMs) important studies have been carried out. In this way, it will minimize cement cost and CO_2 emissions and provide the advantage of achieving high strength.

There are some studies to determine the early age strength of concrete or mortar [30], [31]. In their study, Shah et al. investigated the fresh and early age strength properties of fly ash/slag-based alkali-activated mortar. The curing temperature contributed to the improvement of the early age strength of the single-component AAM (Alkali-activated mortar). Through microstructural tests, they stated that this effect increased the geopolymerization reaction [22] and thus provided a significant increase in early strength [32]. Ren et al. blended calcium formate and methacrylic acid with PC paste powder and examined the effects on the setting time, fluidity and mechanical properties of the mortar. According to experimental results, 0.5, 1, 3 and 28 days compressive strength increased [33].

The purpose of this experimental work is to investigate the effect of replacing fine particle D (0%, 5%, 10%, 15% and 20%) with PC, which has not been used together before, on fresh and hardened self-compacting mortars. In this article, the fresh properties (mini-slump flow and viscosity experiments) of SCMs made from (D) and (PC) were evaluated. Also, early age strengths of SCMs produced with these substitution rates were determined. The results show that SCM in its fresh state achieved good workability and flow time when 5%-20% D was substituted with PC at a 5% increase in weight.

2. Materials

For this laboratory study, CEM I 42.5 R type PC complying with TS-EN 197-1 was used. PC has specific gravity of 3.09. Locally available river sand with a specific gravity of 2.63, a water absorption rate of 1.91%, a maximum size of 4 mm and a fineness modulus of 2.87 was used. D, as a fine filler pozzolanic binder, was included in the mixture with the PC to increase the viscosity. In addition, D, which is a sedimentary rock, was obtained from NB Global Mining and Chemical Industry and Trade Company. Polycarboxylate based Sika Viscocrete SF 18 super plasticizer (SP) was used as water reducer. Its density is 1.1 g/cm^3 and pH value is 3-7. The aggregate and binder materials used for the experimental study are shown in Figure 1.



Figure 1. Dry ingredients used in mixes a) sand, b) PC, c) D

Characteristic properties and some mineralogical compositions of D and PC are presented in Table 1. Figure 2 was given the granulometry curve of river sand.

Table 1. Characteristics of PC and diatomite

Component (%)	PC	D
SiO ₂	18.36	60.98
Al ₂ O ₃	4.72	9.35
Fe ₂ O ₃	3.63	3.86
MgO	1.58	2.52
CaO	63.06	2.34
SO ₃	3.29	0.38
Na ₂ O	0.18	-
K ₂ O	0.82	-
Loss on ignition (LOI)	4.13	20.31
Specific gravity	3.09	2.41

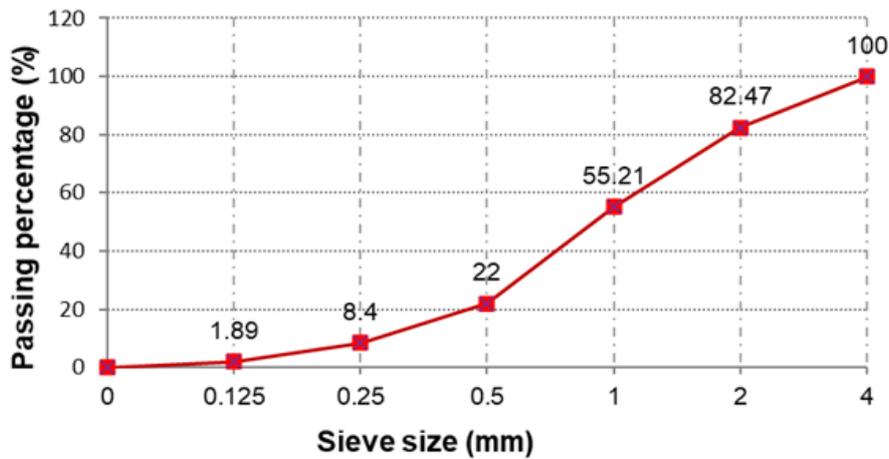


Figure 2. Granulometry curve of river sand

2.1. SCMs Mix Proportions

A control and five mixtures were prepared to determine the fresh and hardened properties of SCMs produced by substituting D with different proportions of cement. The content compositions of SCM are shown in Table 2. Substitution rates by weight were used at 0%, 5%, 10%, 15% and 20%, respectively. After repeated trial mixtures, water/powder (w/p) volume ratios were calculated as 0.36, 0.38, 0.45, 0.57 and 0.69 for control, D5, D10, D15 and D20, respectively. In all prepared mixtures, the binder amount was 630 kg/m³ and the SP amount was chosen as 8 kg/m³. In order provide the EFNARC (European Federation of Specialized Construction Chemicals and Concrete Systems guidance standards) [34] of the spreading diameters of SCMs, the water content was changed depending on the increase in D. Table 2 shows the water/binder (=C + D) (w/b) and water/powder (=C + 0.9D + 0.0191 sand) (w/p) ratios.

Table 2. Mixture proportions for SCM (kg/m³)

Mix name	C	D	Sand	Water	SP	Water/binder	Water/powder
Control	630	0	1367.5	260	8	0.41	0.36
D5	598.5	31.5	1333.4	270	8	0.43	0.38
D10	567	63	1227.0	315	8	0.50	0.45
D15	535.5	94.5	1028.7	380	8	0.60	0.57
D20	504	126	836.8	450	8	0.71	0.69

3. Experimental Method

3.1. Casting and curing

A mixer was used to create SCMs. To create mortar samples, first dry binding materials and river sand were taken into the mixing bowl. Firstly, the dry components (fine aggregate, binders) were mixed until homogeneous. Then, water was added to the SP and poured into the dry mixture and mixed. The prepared mixtures were poured in three stages, taking care to keep the $40 \times 40 \times 160$ mm sized prismatic molds stable. SCMs, which hardened after 24 hours, were removed from the mold. The specimens removed from the mold were left to cure in water at around 23°C until the test day. The fresh mortars poured into molds are given in Figure 3.



Figure 3. Fresh SCM

3.2. Tests

3.2.1. Fresh properties

Mini-slump flow testing and viscosity experiments were conducted to determine the workability and flowability properties of fresh mortars (Figure 4.). Before the strength tests, mini-slump flow test was conducted according to EFNARC criteria to examine the workability properties of SCM to achieve filling and passing ability. Mini slump flow tests were carried out to ensure that the fluidity and spreading diameter of the fresh mortar were compatible with the limit values in accordance with the EFNARC regulation.



Figure 4. a) mini-slump spreading diameter, b) viscometer test machine

Viscometer test is a method used to determine the fluidity of concrete/mortar. It is important to evaluate the workability and plastic properties of concrete/mortar [35]. In the measurement made using the Brookfield DVE brand viscosity device, the mixed mortar was poured into a beaker. The torsional moment response of the mortar in the container to the rotation of the shaft at the end of the device was measured. To determine the rheological properties, the rotational speed of the shaft was measured between 1.0-100 rpm (legs with increasing shear rate-shear stress) and 100-1.0 rpm (legs with decreasing shear rate-shear stress) [28]. The

values obtained for different angular velocities were evaluated by drawing the viscosity-angular velocity graph. The fresh property values used for the prepared SCMs are shown in Table 3.

Table 3. Fresh properties for SCMs

Mix name	Mini-slump flow (mm)
Control	242
D5	243
D10	245
D15	243
D20	250

3.2.2. Hardened properties

Mechanical tests were conducted to decide the early strength of the mixtures whose fresh properties were determined. To measure the flexural strength values of SCMs, three prismatic samples with dimensions of 40 x 40 x 160 mm were cast for a test age of 3 days. After cleaning the surface of the SCM prismatic samples, a three-point flexural test (Figure 5.) was performed at a loading rate of 0.2 kN/s. The 3-day flexural strength test of the mortars was conducted in accordance with ASTM C348 [36]

After the flexural test, the compressive strength of each piece divided into two under a loading speed of 2.4 kN/s was determined according to the ASTM C349 [37] standard.



Figure 5. Three-point flexural test

4. Results and Discussions

This study focused on the usability of grounded D with PC in various proportions (0%, 5%, 10%, 15% and 20%). D and cementitious compositions were conducted fresh mortar tests, and their early strengths were evaluated in experimental study.

4.1. Characteristics of fresh SCMs

The workability and fluidity of SCMs containing diatomite were determined by mini-slump test, considering the EFNARC regulation. The results obtained are given in the Figure 6. As seen in the figure, mini-slump results provide limit values between 240-260 mm. These ranges were maintained by changing the amount of water. Accordingly, it has been observed that as the D ratio increases, the water needs increases. Due to the

increase in the D ratio, the increase in the w/b ratio can be associated with the increase in the spreading diameters [38], [39]. As seen in Figure 6, the lower fluidity of the fresh SCMs produced results in a parallel flow time delay [40].

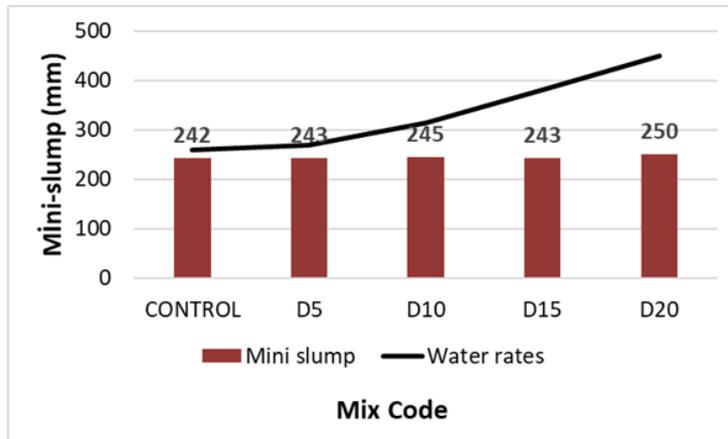


Figure 6. Mini-slump values and the amounts of water

The most important feature of SCMs is sufficient fluidity and resistance to segregation [41]. In order for these parameters to be realized in SCMs, there must be appropriate viscosity, low shear stress and cohesion. For this reason, the viscosity test of mortars containing D was performed to determine their consistency and workability features. Figure 7 shows the viscosity results of the produced samples. This graph shows that as the angular speed of the spindle increased, the viscosity values of the samples decreased asymptotically [18]. As seen in Figure 7, the viscosity value of the samples containing D5 remained above the viscosity value of the control and other mixtures at the first 2 rotation speeds. With increasing speed after 5 rpm, the viscosity of all series decreased and the inclusion of D at this speed reduced the viscosity, resulting in lower results than the control mixture. The lowest viscosity was measured at 100 Rpm for the D15 mixture, while the highest viscosity was measured at low rpm for the D5 mixture.

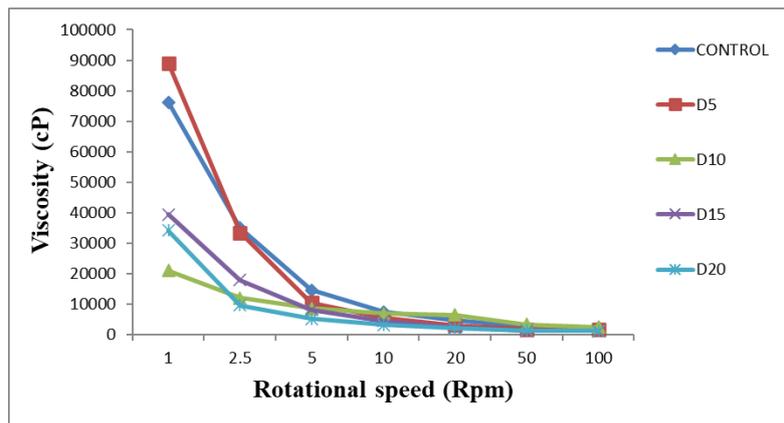


Figure 7. Viscosity values of SCMs

4.2. Characteristics of hardened SCMs

Failure loads for three-point flexural test was given in Table 4. The Figure 8 presents the effect of D on the flexural strength of prismatic samples prepared for 3- days. It was observed that the addition of 5% D increased the flexural strength on compared to the reference samples. When the 3-day flexural strengths of the control sample were compared with the combinations containing 5% D, 10% D, 15% D and 20% D, a 14.6% increase, 13.55%, 44% and 61.6% decrease were detected, respectively. While this shows that the 3-

day strength of the 5% D series was better than the control sample, this situation was reversed due to the increase in the D rate.

Table 4. Failure loads for flexural tests

Mix name	Failure loads (N)
Control	2181.55
D5	2501.12
D10	1885.87
D15	1221.55
D20	836.70

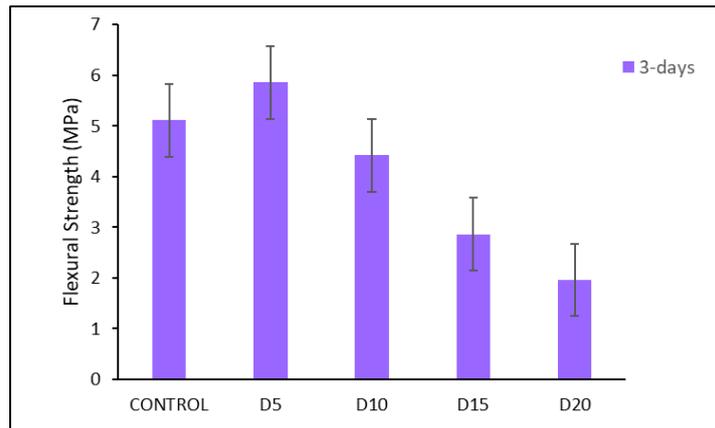


Figure 8. Flexural strength of 3-days SCMs

3-days compressive strengths of SCMs are shown in Figure 9. According to this figure, 3-day strength value of the all the sample, a decrease in compressive strength was observed as the D ratio increased. When the compressive strengths of the D5, D10, D15 and D20 series were compared with the control sample, there was an increase of 11.65%, a decrease of 19.96%, 52.13% and 71.3%, respectively. This situation is similar to the results of the researchers [22].

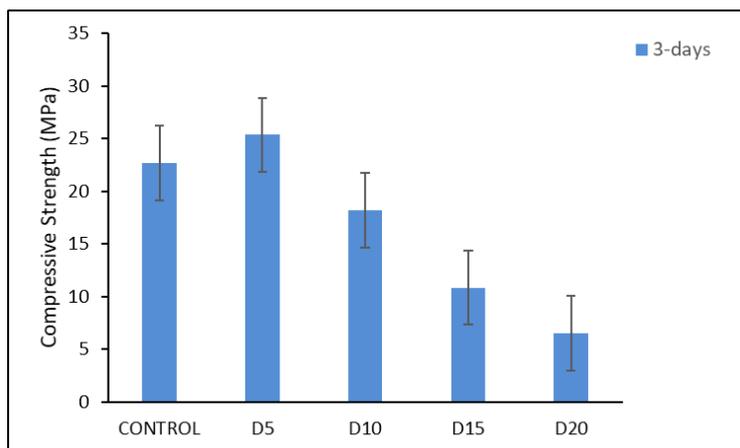


Figure 9. Compressive strength of 3-days SCMs

5. Conclusion

In this study, the fresh and hardened situations of five self-compacting mortars with different replacement rates of diatomite, a sedimentary rock, were examined. For this, rheological and fresh properties were determined by mini-slump flow and viscometer tests on the produced SCMs. Then, flexural and compressive

strength experiments were performed on SCM mixtures containing diatomite. The findings obtained from this experimental research are explained below.

- In order to ensure the self-compacting of mortars containing diatomite, mini-slump values in the range of 240-260 mm were determined in accordance with EFNARC. It has been determined that to achieve this, different amounts of water are required depending on the substitution rates of the binders.
- As the D content increased, the slump flow value increased due to the increase in water need.
- The viscosity readings of the reference sample are higher than all other samples for rotation speeds of 2.5, 5 and 10.
- While the highest flexural strength among all series is measured in the series with 5% D additive, the lowest strength is in the series with 20% D content. This decrease is approximately 67%.
- When the compressive strengths were examined, the highest strength occurred in the mixture with 5% D content, similar to the flexural strength. As the D content increased, the strength gradually decreased. The largest decrease occurred in the D20 series, with a rate of 74% compared to the D5 series.

As a result, it was determined by early strength tests that D could be replaced with PC at a rate of 5%-10%. However, for more detailed examination, 28- and 90-days mechanical strengths should be determined in future studies. In addition, freeze-thaw, sulfate resistance, abrasion, high temperature, etc. tests should be carried out to determine durability properties.

6. Author Contribution Statement

In this study, Author 1 covers the topics of conducting experiments, literature review, and obtaining the materials used; Author 2 under the headings of literature review, preparation of the article and evaluation of the results obtained; Author 3 contributed to the formation of the idea, design, spelling and checking the article for content.

7. Ethics Committee Approval and Conflict of Interest Declaration

There is no need to obtain ethics committee permission for the article prepared. There is no conflict of interest with any person/institution in the prepared article.

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