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The Effect of Clay Type, Fineness and Methylene Blue Value on Mini-Slump Performance of Cementitious Systems

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Abstract: It is known that the fresh and hardened properties of cementitious systems are adversely affected by the increase in aggregate clay content. It was emphasized that the fresh state properties of cementitious systems are affected by the clay type. In this study, the effects of clay type, fineness and methylene blue value on the mini-slump performance of cement paste mixtures were investigated. For this purpose, 3% of the cement weight, bentonite, kaolin and limestone powder passed through a 0.125 mm sieve were used. It was determined that bentonite clay used within the scope of the study had a 5 times higher methylene blue value compared to kaolin. It was determined that the mini-slump value of the mixtures increased with the increase of the water/cement ratio, independent of the clay type, and the said values decreased with the addition of clay. In terms of mini-slump performance, the clay fineness parameter was found to be more dominant than the methylene blue value.

Keywords: Bentonite, Kaolin, Water requirement, Mini slump, Methylene blue value

Introductıon

As a result of facilities such as dams built on rivers where the aggregate used in concrete mixtures is supplied, the supply of aggregate has become difficult (Guneyli et al., 2010; Deşik et al., 2019). For this reason, the use of crushed aggregate, which has a lower cost, has become widespread in concrete (Guneyli et al., 2010). The main usage area of limestone in the world is the construction and building sector with a rate of 40-70% (Bozkurt et al., 2008). Limestone provides a certain skeleton and strength to the concrete mix, especially thanks to its use as aggregate. Limestone deposits are formed in the form of travertine in groundwater, and as a result of chemical, organic or mechanical precipitation in sea or fresh waters (Turan, 2010). Limestone theoretically contains 56% CaO (hydrated Lime) and 44% CO_2, but it is never found in pure form in nature (Turan, 2010, Özbek et al., 2016). Limestone contains at least 90% $CaCO_3$ (Calcium Carbonate) in its chemical composition, as well as compounds such as $MgCO_3$ (magnesium carbonate), clay minerals, iron silicate-oxide and SiO_2 (Turan, 2010). As the clay bands that may be between the sedimentary rock layers are fed directly to the crushers without separating from the broken rocks, harmful substances such as clay and silt in the soiled material can mix with the crushed sand (Ozbebek et al., 2011; Guneyli et al., 2010). In addition, in rainy periods, clay and silt take the form of mud and adhere to the rocks (Desik et al., 2019). If production is not carried out in accordance with the technique in the crusher plant, harmful materials deteriorate the quality of crushed sand (Ozbebek et al., 2011; Guneyli et al., 2010). As a result of the use of the uncleaned aggregate in the concrete, the clay penetrates into the concrete (Muslu, 2019). The clay ratio in the aggregate is determined by the methylene blue and sand equivalence test (Muslu, 2019).

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The presence of clays in cementitious systems leads to various negative situations (Kirthika, et al., 2019). The presence of clay in the concrete aggregate reduces the adherence between cement paste and aggregate (Ozbebek et al., 2011). Due to the high water absorption ability of the clay mineral, it causes volume expansion and as a result, cracks in the concrete and tensile stresses caused by shrinkage (Desik et al., 2019). In addition, clay minerals increase the specific surface area value of the aggregate (Muslu, 2019). As a result, as the amount of mixing water and w/c ratio required for concrete increases, hydration is delayed, setting time increases, and durability and strength values decrease (Ozbebek et al., 2011; Desik et al., 2019). Since extra cement and chemical admixtures will be used to keep the w/c ratio constant in mixtures with higher clay content, the cost of $1m^3$ concrete increases accordingly. Since clay contaminates the aggregate, the consistency losses that occur depending on time in concrete made with dirty aggregate make it difficult to pump (Ozbebek et al., 2011; Desik et al., 2019). In addition, it was declared that due to the layered structure of clays, chemical admixtures are intercalated between these layers (Muslu, 2019). It was emphasized by various researchers that this situation leads to more admixture use (Ozbebek et al., 2011; Desik et al., 2019; Ma et al., 2022). Clay is a layered or fibrous hydrated aluminum or magnesium silicate with a size of less than 0.02 mm (Ozbebek et al., 2011). Depending on the bonding of tetrahedral and octahedral layers, which are the basic structural units in clay minerals, various clay minerals can be formed (Kalpaklı et al., 2022). The quality of the clays and the areas in which they can be used are related to the type of clay minerals, their composition and the amount of these minerals. Clays have properties such as color, particle size, specific surface area, adsorption, cation exchange capacity, cohesion, plasticity. Clay minerals have very small particle sizes and have inner and outer crystal surfaces due to trioctahedral (tetrahedral-octahedral-tetrahedral) or dioctahedral (tetrahedral-octahedral) molecular structures, while increasing the porous structure and surface area of the clays, while increasing the area to be adsorbed (Kalpaklı et al., 2022). The fact that clays have a very large and complex mineral array, the presence of impurities in them, the place of formation and the variation in their properties cause them to be classified in many ways. Clays can be classified as Kaolin group, Illite group, Chlorite group, Smectite group, Mica group.

Bentonite is the most common of the smectite group clays and is found in nature as rocks composed of mineral mixtures (Al-Hammood et al., 2021). In addition to the predominant montmorillonite mineral, bentonite rocks contain clay minerals such as kaolinite, illite, and paligorskite, as well as non-clay minerals such as gypsum, quartz, calcite (Al-Hammood et al., 2021). Since bentonite clay is formed as a result of bonding an octahedral aluminum layer between two tetrahedral silicon layers, the expression (2:1) is used to describe this lattice number (Kalpaklı et al., 2022; Al-Hammood et al., 2021). When bentonite meets water, it swells 20-30 times (Kalpaklı et al., 2022). It was declared that bentonite has higher methylene blue adsorbing capacity due to the higher specific surface area than kaolin group clays (Mesboua et al., 2018). Bentonite, which is rich in magnesium, is used in the construction industry for impermeable layer and soil stabilization (Mesboua et al., 2018). Raw bentonite is characterized by its relatively low specific gravity and relatively high surface area compared to Portland cement (Al-Hammood et al., 2021). Substitution of cement with raw bentonite has two opposing effects on the consistency of the cement paste. It was reported that bentonite particles cause an increase in the fluidity of paste mixtures with the dispersion effect, while at the same time, it causes a decrease in fluidity due to its high absorption capacity (Al-Hammood et al., 2021). It was observed that sodium bentonite (Na-RB) has high water absorption, while calcium bentonite (Ca-RB) has relatively low absorption (Al-Hammood et al., 2021). In the study conducted by Mesboua et al. (2018), it was reported that the mini-slump diameter values of cementitious mixtures decreased, while the viscosity and shear stress values increased with the increase in the bentonite substitution ratio. The fine bentonite particles provide additional nucleation sites that promote the hydration process and subsequently accelerate the setting time. In addition, it was reported that the high absorption of bentonite accelerates cement paste setting. On the other hand, it was reported that the substitution of Portland cement with raw bentonite causes an increase in the setting time because it reduces the cement components C_3A and C_3S that accelerate the setting (Al- Hammood et al., 2021). It was declared that the compressive strength of concrete decreases due to the substitution of raw bentonite for cement, and high replacement rates (35-50%) cause decreases in the compressive strength of approximately (45-60%) (Al-Hammood et al., 2021). However, Karthikeyan et al. (2015) emphasized that there was a 30% increase in compressive strength with 20% bentonite substitution.

Kaolin clay is a two-layered phyllosilicate group clay (1:1) formed by the combination of tetrahedron and octahedron (Kalpaklı et al., 2022). Since the surfaces of kaolinites with tetrahedrons are covered with an oxygen octahedron and a hydroxide layer, there is a strong attraction force between the two layers and this prevents them from absorbing water and swelling (Kalpaklı et al., 2022). Naganathan et al. (2010), it was reported that the setting time of mixtures without kaolin was 5-7.5 hours, while the setting time of mixtures containing kaolin was 12-32 hours. It was reported that the presence of SiO_2 and Al_2O_3 in kaolin delays hydration and therefore prolongs the initial setting time. In the same study, it was observed that while bleeding was between 1% and

11% in mixtures without kaolin addition, it was 1% or less in all mixtures with kaolin. This was interpreted as the addition of kaolin and the increase in the water-absorbing powder content of the mixture, resulting in decreased bleeding. In addition, it was concluded that the compressive strength values of mixtures with kaolin were lower than the compressive strength values of mixtures without kaolin addition. The decrease in strength was attributed to the reduction of Van der Waals forces due to kaolin. It was reported that water absorption, sorption and initial surface absorption values increase due to the increase in the amount of kaolin added to cements (Naganathan et al., 2010).

As a result, it was emphasized by many researchers that aggregates with a methylene blue value greater than 1.50 g/kg are not suitable for use in concrete production (Kirthika et al., 2019). It was also understood from the literature that the type, fineness and amount of clay affect the fresh and hardened state properties of cementitious systems. In this study, the effect of bentonite, kaolin and limestone powder passing through a 0.125 mm sieve on the mini-slump performance of the cement paste mixture was investigated.

Material and Method

Material

Within the scope of the study, Bursa Cement, Kaolin Company and Technical Mineral Mining Company, CEM I 42.5R type cement, kaolin and bentonite clay were used, respectively. Some chemical, physical and mechanical properties of cement, bentonite, kaolin and sand powder supplied by the manufacturers are presented in Table 1 and Table 2. It was reported by the manufacturer that bentonite clay is composed of a minimum of 85% montmorillonite and a maximum of 15% cristobalite-opal.

Table 1. Chemical properties of cement, kaolin, bentonite and limestone sand powder

Oxide (%)	Cement	Kaolin	Bentonite	Limestone Sand Powder
SiO_2	18.74	46.5	71.00 (± 0.50)	4.60
Al_2O_3	5.37	37.0	12.50 (±0.50)	2.32
Fe_2O_3	3.04	0.5	0.75 (±0.10)	1.33
CaO	64.11	0.15	1.12 (±0.10)	90.50
MgO	1.21	0.15	1.25 (±0.10)	0.782
SO_3	2.68	-	0.02 (±0.01)	0.0562
Na_2O	0.34	0.05	0.04 (±0.01)	-
K_2O	0.62	0.60	0.25 (±0.02)	0.284
Cl^-	0.038	-		-
Free CaO	2.12	-		-
P_2O_5	-	-		0.137
SrO	-	-	-	0.0444
Loss on ignition	3.6	-	-	13.00 (±0.50)
PH	-	6.5-8.5	8.5(±0.5)	-
Maximum moisture (%)	-	1	12	-

Table 2. Physical properties of cement, kaolin, bentonite and limestone sand powder

Properties	Cement	Kaolin	Bentonite	Limestone Sand Powder
Specific gravity	3.15	2.782	2.71	2.73
Sweling (ml/2g)	-	-	30.00 (±2,00)	-
Cation exchange capacity (mg/100g)	-	-	86.00 (5.00)	-
Specific surface area (Blaine, cm^2/g)	3600	10491	1100	1000
Residue in 0.045 mm sieve (%)	7.4	0.60	$D_{97} < 20\mu$ $D_{100} < 30\mu$	-
Residue in 0.090 mm sieve (%)	0.4	0.15	-	-

Preparation of Mixtures

The w/b ratio was kept constant as 0.35 in all mixtures prepared to examine the mini-slump performance of cement paste mixtures. However, in the mixtures produced, the w/b ratio was chosen as 0.39, since spreading did not occur. In addition to the control mixture, which does not contain clay and limestone sand powder, 3% of the weight of the cement, kaolin, bentonite clays and limestone sand powder were added and a total of 4 different series of paste mixtures were prepared. In the preparation of the mixtures, firstly, 800 g of cement and 3% of the cement weight of fine material were mixed homogeneously. 312 g of water was added to the prepared homogeneous dry material and mixed at 20 rpm (slow) for 30 seconds. At the end of 30 seconds, the walls of the container were cleaned with the help of a spatula. It was then mixed at 40 rpm (fast) for 2 minutes. Mini-slump test was performed on the prepared mixtures. The amount of material contained in the paste mixtures produced for the mini-slump test is given in Table 3. The denotation of the mixtures is made according to the w/b ratio and the type of fine material it contains. For example, the mixture containing kaolin clay with a w/b ratio of 0.39 was named Kaolin_0.39.

Table 3. The amount of material contained in the paste mixes produced for the mini-slump test

Mixture	Cement (g)	Kaolin (g)	Bentonite (g)	Limestone sand powder (g)	Water (g)	Water/Binder
Control_0.35	800	-	-	-	280	0.35
Kaolin_0.35	800	24	-	-		
Bentonite_0.35	800	-	24	-		
Limestone_0.35	800	-	-	24		
Control_0.39	800	-	-	-	312	0.39
Kaolin_0.39	800	24	-	-		
Bentonite_0.39	800	-	24	-		
Limestone_0.39	800	-	-	24		

Method

Methylene Blue Value

The methylene blue test was carried out in accordance with the TS 706, EN 12620 Standard. Limestone aggregate sieved through a 2 mm mesh sieve was added to a beaker filled with 500±5 ml distilled water. After stirring at 0 rpm for 5 minutes, 5 ml of methylene blue solution was added to the beaker. The material in the beaker was mixed at 400 rpm for 1 minute and the stain test was carried out on the filter paper with a dropper. Solution addition and staining experiments were continued until the halo appeared.

The aggregate passing through a 2 mm sieve was sieved under water in a 0.064 mm sieve and washed sand was obtained. In order to determine the effect of clays on methylene blue value, the experiment was carried out by substituting 5 g of montmorillonite/kaolin clay for sand. The methylene blue value was calculated with the help of equation 1:

$$MB = (V1/M1)x10 \tag{1}$$

In this equation, MB is the methylene blue value, M1 is the mass of the test sample (g), V1 is the total volume of the added dye solution (ml).

Mini-Slump

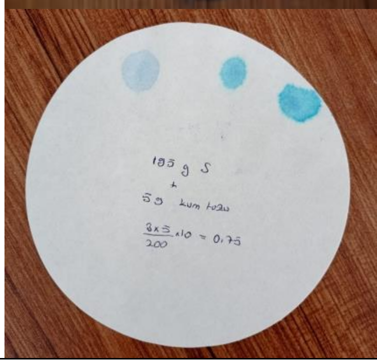
The mini-slump experiment was carried out in accordance with the method proposed by Kantro (1980). The paste mixture prepared in the mini-slump test was filled into a truncated conical apparatus with a lower inner diameter of 38.1 mm, an upper inner diameter of 19 mm and a height of 57.2 mm, placed in the center of a smooth surface. The mini-slump funnel was slowly lifted vertically. Then, with the help of a ruler, the spreading diameter in two perpendicular directions was measured and the average was recorded.

Discussion and Conclusion

Methylene Blue Value

The methylene blue test results are shown in Table 4. As seen in Table 4, the methylene blue value of the sand before washing was 4.25 g/kg, while this value was measured as 0 g/kg after washing under a 0.063 mm sieve. By substituting 5% of bentonite, kaolin and limestone powder for washed sand, the methylene blue value was determined to be 5, 1 and 0.75 g/kg, respectively. Thus, it was understood that bentonite clay is 5 times more aggressive than kaolin. The methylene blue value of the sand powder, on the other hand, took the minimum value with 0.75 g/kg.

Table 4. Methylene blue value test results

Sample	Methylene blue value experiment filter paper	Methylene blue value (g/kg)
Sand		4.25
Washed sand		0
195 g sand+5 g bentonite		5
195 g sand+5 g kaolin		1
195 g sand+5 g limestone sand powder		0.75

Mini-Slump

Mini-slump test results of paste mixes are given in Figure 1. In addition, the images of the mini-slump test of mixtures with 0.35 w/b ratio without any spreading are shown in Table 5. Regardless of the clay type, the mini-slump values of the mixtures increased with the increase of the w/b ratio. With the increase of the w/b ratio from 0.35 to 0.39, the slump values of the mixtures containing control, bentonite, kaolin and limestone powder increased by 56%, 22%, 11% and 33%, respectively. It was determined that the mini-slump values decreased with the addition of fine material to the mixture, regardless of the fine material type. It was understood that the lowest and highest mini-slump values were measured in the mixture containing control and Kaolin, respectively.

As it is known, with the increase in the methylene blue value of the aggregate, its negative effect on cementitious systems increases. In the study conducted by Beixing et al. (2011) it was reported that the workability and compressive-flexural strength of cementitious mixtures decreased with the increase in the methylene blue value of the aggregate (0.35-2 g/kg). On the other hand, Chen et al. (2020) declared that the combined effect of fine material content and methylene blue value has a significant effect on the workability of concrete. It was stated that when the methylene blue value is less than 7 g/kg, sand produced with a high micro-fine content (15 to 20%) can effectively improve the workability of concrete. In this study, it was observed that although bentonite clay had the highest methylene blue value, the mixture containing bentonite was not the mixture with the lowest performance in terms of mini-slump. It was understood that kaolin clay affected the flow performance of the mixture more negatively than bentonite clay. This is thought to be due to the fact that kaolin clay is 10 times thinner than bentonite clay (Table 2). In this context, it was understood that the fineness parameter is more dominant than the methylene blue value in terms of the flow performance of the mixtures. As is known, the binder fineness parameter seriously affects the flow performance of cementitious systems (Mardani Aghabaglou et al., 2016). It was reported by various researchers that the hydration rate increases with the increase of binder fineness, and accordingly, the flow performance of the mixture is adversely affected (Mardani-Aghabaglou et al. 2016, Aydın et al., 2009). Hammat et al. (2021), it was emphasized that the flow performance of the mixture decreased with the increase of the natural pozzolan substitution ratio. It was reported that this is due to the fact that natural pozzolana is thinner and more porous than cement. In the study conducted by Benjeddou et al., (2021), it was stated that the Marsh funnel flow time values increased with the increase of limestone powder fineness. It was emphasized that the cohesion of cementitious mixes increased with the increase of limestone powder fineness (Diederich et al., 2012). Biricik et al. (2022), it was reported that the mixture containing the marble powder with the highest Blaine fineness value had the lowest setting time. However, it was also reported that the flow performance of cementitious mixtures is positively affected by the increase in fly ash fineness, which is another supplementary material (Jamkar et al., 2013; Sahin et al., 2022). Jamkar et al. (2013) concluded that fly ash fineness increases both workability and compressive strength of geopolymer concrete. Şahin et al. (2022) stated that the mini-slump values increase with the increase of fly ash fineness.

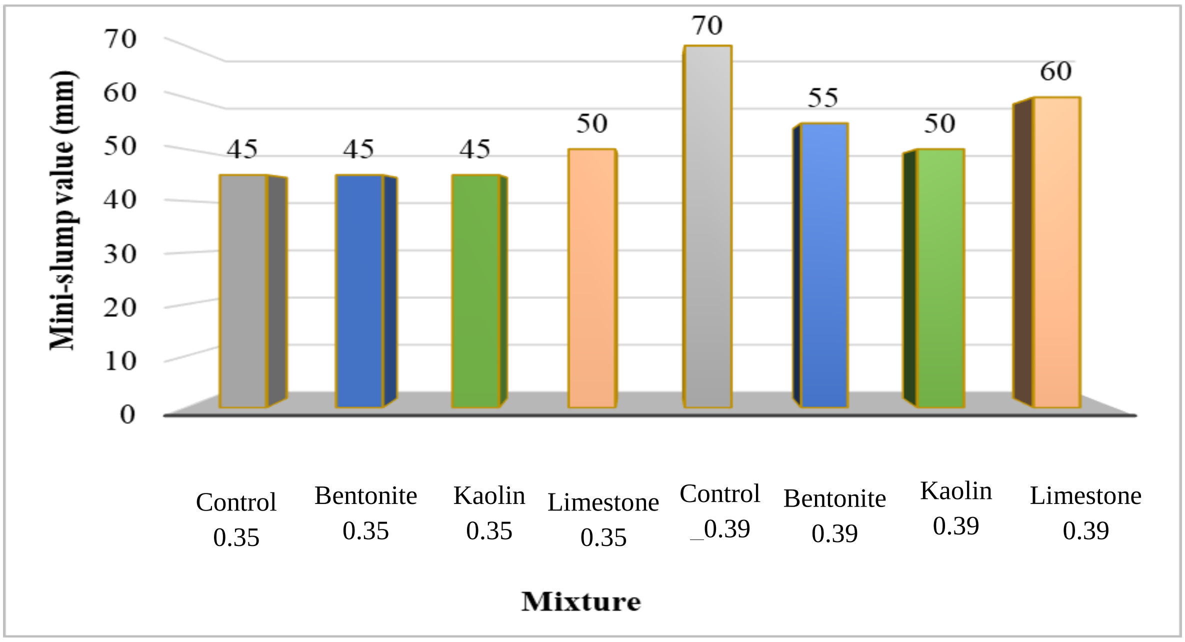
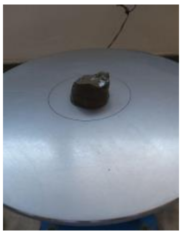
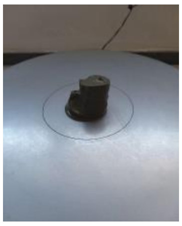


Figure 1. Mini-slump value of mixtures

Table 5. Mini-slump test images of paste mixes with 0.35 w/b ratio

Control_0.35		
Bentonite_0.35		
Kaolin_0.35		
Limestone_0.35		

Conclusion

The results summarized below were obtained in line with the materials used in the study and the experiments applied:

- Bentonite clay has been determined to have 5 times higher methylene blue value compared to kaolin.
- It was observed that the mini-slump values of the mixtures increased with the increase of the w/b ratio, independent of the clay type, and decreased with the addition of clay.
- Mixtures containing control and kaolin without fine material exhibited the highest and lowest performance in terms of mini-slump behavior.
- In terms of flow performance, it was understood that the fineness parameter was more dominant than the methylene blue parameter.

Data Availability Statement

All data, models, and code generated or used during the study appear in the submitted article.

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Conflicts of Interest/Competing Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

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