Research Article

Improvement of the Brittleness and Drying shrinkage in Very High Strength Concrete Incorporating Micro Metakaolin

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Abstract

Concrete commonly displays substantial brittleness and drying shrinkage, which have a detrimental influence on its performance and durability, especially in combinations with high quantities of cementitious elements, such as extremely high strength concrete. This type of concrete frequently encounters these issues due to its strong compressive strength and comparatively low split tensile strength. Consequently, micro MK was used to mitigate the brittleness and drying shrinkage in concrete. Five mixes were tested using micro metakaolin as a partial cement replacement at 0%, 3.5%, 4%, 4.5%, and 5% in very-high-strength concrete mixes using the wet mixing method. Additionally, each mix's compressive strength, split tensile strength, drying shrinkage, water absorption, and ultrasonic pulse velocity were all examined. The results showed that incorporating 4% micro metakaolin into very-high-strength concrete reduced the brittleness index to 9.101, while drying shrinkage and water absorption were reduced by 18.6% and 19.04%, respectively, due to a reduction in both the quantity and size of pores within the concrete matrix caused by micro-sized metakaolin. Incorporating 4% micro metakaolin increased pulse velocity, reaching a high of 7.493 mm/µs due to shorter distances between micro voids in the concrete matrix.

Keywords: Brittleness Index, Drying Shrinkage, Metakaolin, Ultrasonic Pulse Velocity, Very High-Strength Concrete, Water Absorption

Introduction

The global use of supplementary cementitious materials (SCM) as partial substitutes for cement in the manufacture of ultra-high strength concrete is on the rise, including materials like as fly ash, silica fume, blast-furnace slag, and Metakaolin (MK). The use of MK in the concrete mix enhances the distinctive attributes of very high strength concrete (VHSC), including both mechanical and non-mechanical qualities, due to its filling effect and pozzolanic response. The utilisation of mineral admixtures led to a reduction in carbon dioxide emissions, hence encouraging environmentally benign concrete applications [1,2].

Metakaolin is categorised as natural pozzolan class N in ASTM C618; it is distinctive since it is neither wholly natural nor solely an industrial product. It is obtained from a naturally occurring mineral [3]. Metakaolin is mostly derived from two principal sources: the thermal and mechanical activation of purified kaolinite clay and the calcination of waste paper sludge in an incinerator. The superior quality of metakaolin (MK) is attained by accomplishing near-total dihydroxylation of the kaolinite component of kaolin clay without excessive heating, generally within the range of 650°C to 900°C; the temperature during the calcination process significantly influences the reactivity of the MK [4,5].

When MK is incorporated into concrete, it triggers a pozzolanic reaction, interacting with calcium hydroxide, a byproduct of cement hydration, to produce several hydrates including C-S-H, C2ASH8, and C3AH6. The reaction results in a substantial enhancement in the mechanical characteristics of concrete. Furthermore, the incorporation of Calcium hydroxide by MK alters the microstructure of concrete, enhances the interfacial transition zone (ITZ) between aggregate and cement paste, and increases density while improving the characteristics of concrete [6,7].

Significance of the Research

Drying shrinkage is often greater in VHSC because of the high cement content. In general, a greater brittleness index is associated with a higher compressive strength. This work aims to address these difficulties by incorporating micro MK into VHSC mixes, with the objective of reducing drying shrinkage and brittleness index.

Literature Review

Conducted experimental studies on the use of MK in VHSC to determine how MK affects the characteristics of VHSC mixtures. These studies examined a wide variety of MK replacement ratios in the different grades of concrete, even though the effect of replacement of MK with cement might be more effective in high-cement content concrete for reducing drying shrinkage laiter.

Effect of MK on the Mechanical Properties of Very-high-strength Concrete

Previous studies looked at the influence of MK on the mechanical characteristics of VHSC mixtures. The use of MK as a partial substitute for cement enhanced compressive strength. The optimal MK percentages ranged from 8% to 20% of the cement mass, improving compressive strength by 8.7% to 24.7% at 28 days of age [6, 8, 14].

The effect of MK on split tensile strength of VHSC was explored by [9], the MK replacement ratios ranged from 5-15%. The investigators observed that the splitting tensile strength reached an optimum at 10% MK replacement at which it improved by 9.03% compared to the control mix at 28 days.

A research was conducted by [15] to investigate the effect of partial cement substitution with MK at 5%, 10%, and 15%. The findings revealed that the addition of MK considerably increased the splitting tensile strength of concrete.

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The researchers discovered that at an optimal replacement ratio of 10% MK, the split tensile strength increased by 7.44%.

Effect of MK on the Non-mechanical Properties of Very-high strength Concrete

A study on the drying shrinkage and water absorption of ultra-high strength concrete was performed by Güneyisi et al. [6] employing MK as a partial cement replacement at 10% and 20% ratios. The researchers observed that the drying shrinkage of the concrete mixtures with 10% and 20% MK was approximately 5% and 15% lower than that of the control mixture, respectively. The findings demonstrated MK's effectiveness in reducing drying shrinkage. Moreover, they established that an elevation in the quantity of MK led to a decrease in water absorption.

Güneyisi et al. [14] studied to assess the drying shrinkage of concrete. The investigators determined that the incorporation of MK resulted in a 22% and 26% reduction in the drying shrinkage of concrete compared to the control mix, with partial replacements of 5% and 15% of MK, respectively.

The impact of MK on water absorption was assessed by Nour Eldin et al [16]. They determined that the water absorption is decreased by introducing 15% MK as a partial substitute for cement, in comparison to the control mix. Moreover, researchers disclosed that a decrease of 4.81% and 9.94% was noted for 5% and 10% of MK, respectively, in comparison to the control mix [17].

Experimental Programme

To assess the impact of micro MK on the drying shrinkage and brittleness index of VHSC mixtures, encompassing compressive strength and split tensile strength. Micro MK was integrated into VHSC mixtures as a partial substitute for cement at replacement ratios of 3.5%, 4%, 4.5%, and 5%. Preliminary cube compressive strength tests conducted at 7 days of age across a broad spectrum of micro MK contents indicated that replacement ratios over 5% of micro MK resulted in a substantial decrease in cube compressive strength.

Selection of Materials

This study involved the selection of cement, fine aggregates, and mineral admixtures as materials. The specifications and properties of the cement, aggregates, and auxiliary materials were derived from a prior study now in publication [18]. Nonetheless, micro MK has been freshly presented in this research.

Portland cement

Ordinary Portland cement (Type I) was employed for the research study. The chemical composition and physical properties of the cement are detailed in Tables 1 and 2, respectively. The cement complies with ASTM requirements. [19,20].

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Table 1. Properties of Portland cement and their limits ASTM [19]

Test Name	Results	Allowable limit
Average cube compressive strength, N/mm^2 ,7-days	23.65	19 MPa
Normal consistency ,%	33.2	-
Initial setting time, minute	130	Minimum 45
Final setting time ,minute	246	Maximum 375
Specific gravity	3.1	-

Table 2. Chemical composition of cement and their limits [20]

Composition	Test Results (%)	Allowable limit
C2S	7.67	
C3S	66.33	
C3A	2.19	8 (max)
C4AF	15.5	

Fine aggregates

Three varieties of fine aggregates were included to create the control mix of VHSC: quartz sand, magnetite black sand, and RAP aggregates, as indicated by prior study [18]. Figures 1 and 2 exhibit the three types of sand employed in the research, while Figure 3 presents the sieve analysis, and their attributes are summarised in Table 3. The quartz sand has almost 90% silicon dioxide. Table 4 displays the chemical composition of magnetite BS. The origin of RAP aggregate and the preparation process were derived from [18]. The chemical composition of RAP aggregate is detailed in Table 4.

(a) (b)

Figure 1. Fine aggregate used in the research(a) Quartz sand , (b) Magnetite black sand

Figure 2. Recycled asphalt pavement aggregate

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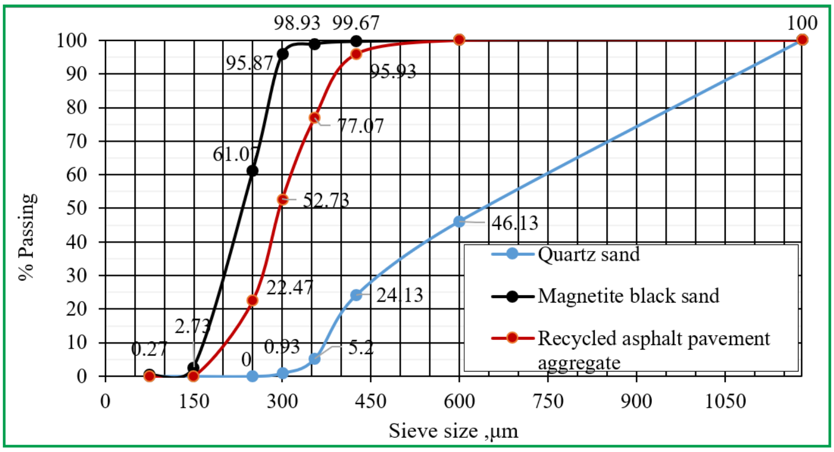

Figure 3. Screen analysis of the three types of fine aggregate utilized for research

Table 3. Physical properties of fine aggregate

Composition	Quartz Sand	Magnetite Black Sand	Recycled Asphalt Pavement Aggregate
Appearance/color	Granular / light tan	Granular / black	Granular / brown
Absorption, %	0.32	0.65	2.389
Bulk specific gravity	2.6	4.789	2.513
Apparent specific gravity	2.609	4.953	2.674
Effective specific gravity	2.6045	4.866	2.594
Density, g/cm^3	2.65	5.02	2.68
Average particle size, mm	0.499	0.204	0.267
Fineness modulus	4.29	1.05	2.52
Melting point, C°	1710	-	-
Binder content, %	-	-	0.5

Table 4. Chemical composition of magnetite black sand and RAP aggregate

Composition	Magnetite Black Sand ,%	RAP Aggregate, %
Fe_3O_4	99.450	0.000
Fe_2O_3	0.000	3.120
SiO_2	0.150	34.74
Al_2O_3	0.270	4.634
CaO	0.040	31.98
MgO	0.000	1.612
P_2O_5	0.000	0.073
MnO	0.000	0.070
P	0.007	0.000
S	0.008	0.000
Mn	0.020	0.000
Pb	<0.003	0.000
K_2O	0.0084	0.638
Na_2O	<0.0067	0.263
TiO_2	0.067	0.314
Ca	0.029	0.000
Zn	<0.001	0.000
LOI	0.000	22.41
Others	0.000	0.146

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Metakaolin

Metakaolin was employed as a partial substitute for cement at various replacement ratios, as seen in Figure 4, serving as a supplemental cementitious material (SCM) for the manufacturing of very high-strength concrete (VHSC). The physical parameters of metakaolin are detailed in Table 5.

Table 5. Physical properties of metakaolin

Physical Properties	Value
Appearance / color	Powder/white
Average particle size, micron	2.5-3.5
Density g/cm^3	2.7

Figure 4. Metakaolin powder used in the research

Admixture

A specific Polycarboxylate-based high-range water-reducing additive (Sika Viscocrete 1681) was utilised to improve the workability and strength of VHSC mixtures. Table 6 illustrates the physical parameters of the water-reducing admixture.

Table 6. Physical properties of water reducing admixture

Physical Properties	Quantity
Composition	Aqueous solution of modified polycarboxylates
Appearance and color	Light brownish
Specific gravity, g/cm^3	1.070
pH value	4.5-6.5

The mash cone test was conducted to evaluate the additive dose, achieving the requisite workability and strength of concrete [21,22]. The doses of superplasticizer ranged from 1.2% to 1.6% of the cement weight, with the optimal dosage being 1.53%.

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Mixing water

All concrete mixtures utilised standard warm tap water, devoid of clay and organic or deleterious elements. The mixing water is within permissible parameters for concrete manufacture, with total dissolved solids ranging from 200 to 230 mg/L and electrical conductivity between 400 and 430 µS/cm.

Mix Proportions

The mix proportions of the VHSC were derived from prior study on very-high strength heat-resistant concrete, which examined the mechanical qualities of the concrete through the integration of RAP aggregate. The concrete mixture with the ideal RAP concentration was designated as the control mix. The control mix has an additive to cement ratio of 0.015, a water to cement ratio of 0.21, and a cement to total sand ratio of 0.912, by weight. The entire fine sand mostly consists of quartz sand, with magnetite black sand injected as a partial substitute, resulting in a final ratio of magnetite black sand to quartz sand of 0.31. Subsequently, RAP aggregate was utilised as a partial replacement for the remaining quartz sand. The proportion of RAP aggregate to quartz sand was 0.05.

Properties of Very High Strength Concrete

The efficacy of VHSC is determined by its characteristics in the fresh state and its attributes post-hardening. This section is separated into two subsections: fresh-state qualities, which detail workability, flow characteristics, and setting time post-mixing. The second part delineates the hardened-state features of VHSC, emphasising brittleness, drying shrinkage, water absorption, and the assessment of concrete quality.

Properties of Very High Strength Concrete at Fresh State

This research involved many experiments to assess the performance of VHSC mixes, including the flow table test and setting time at the fresh condition, as referenced in [23, 24] and illustrated in Figures 5. In the flow table test, micro MK was integrated at several replacement ratios, and for all percentages, the flow spread value above 105%, while the setup time exceeded 45 minutes.

(a) (b)

Figure 5. Testing of very high strength concrete at fresh state (a) Flow table test for concrete (b) Setting time by vicat apparatus

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Properties of Very-high Strength-concrete at Hardened State

In its hardened condition, VHSC displays several physical and mechanical properties that influence its performance and longevity. This study conducted a number of experiments to evaluate the mechanical and non-mechanical characteristics of VHSC. These assessments offer a thorough comprehension of the impact of micro MK on the performance of VHSC mixes.

Mechanical properties of very-high strength concrete

In the hardened condition, two experiments were conducted on VHSC mixes: the cube compressive strength test and the split tensile strength test. The experiments were conducted on concrete mixes at 28 days of water bath curing for both the control mix and those using 3.5%, 4%, 4.5%, and 5% micro MK as a partial cement replacement.

Brittleness index

The brittleness index (BI) is a quantitative metric obtained by dividing compressive strength by split tensile strength, indicating the material's propensity for brittle or ductile behaviour [25-27]. The brittleness index measures the equilibrium between the compressive strength and the split tensile strength of concrete. Cubic concrete samples measuring 50 mm per side were evaluated for compressive strength, whereas conventional cylindrical concrete specimens with a diameter of 50 mm and a height of 100 mm were employed for the split tensile strength test [28,29].

Non-mechanical properties of very-high strength concrete

The non-mechanical characteristics of VHSC offer significant insights on its durability and microstructural assessments with the integration of micro MK. This study included experiments on non-mechanical factors, including drying shrinkage, water absorption, and ultrasonic pulse velocity (UPV).

Figure 6. Drying shrinkage device for measuring the drying shrinkage of concrete

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Drying shrinkage

Standard prisms of 285 mm in length, 25 mm in width, and 25 mm in height were evaluated for drying shrinkage at 7 and 28 days, as seen in Figure 6. The average drying shrinkage for each condition was determined by testing four specimens [30].

Water Absorption

Standard concrete cube specimens measuring 100 mm on each side were utilised to assess the water absorption of VHSC at 28 days of age. Three samples were analysed, and the mean water absorption was established as per [31].

Ultrasonic Pulse Velocity Test for Checking the Quality of Concrete

The ultrasonic pulse velocity of VHSC mixtures was tested using cube specimens with a side length of 100 mm, as illustrated in Figure 7 [32], in accordance with BS EN 12504-4:2021. Figure 8 illustrates the UPV test phenomenon, which involves the recording of the velocity of an ultrasonic pulse that traverses a concrete sample of known thickness. The UPV test is frequently employed to evaluate the quality of concrete by assessing the level of consistency, homogeneity, durability, and presence of fractures [33]. Additionally, the UPV test can be employed to determine the modulus of elasticity of concrete and to indicate the accuracy of the compressive strength test [34]. The average ultrasonic pulse velocity was determined by testing three samples, and the average UPV value was calculated.

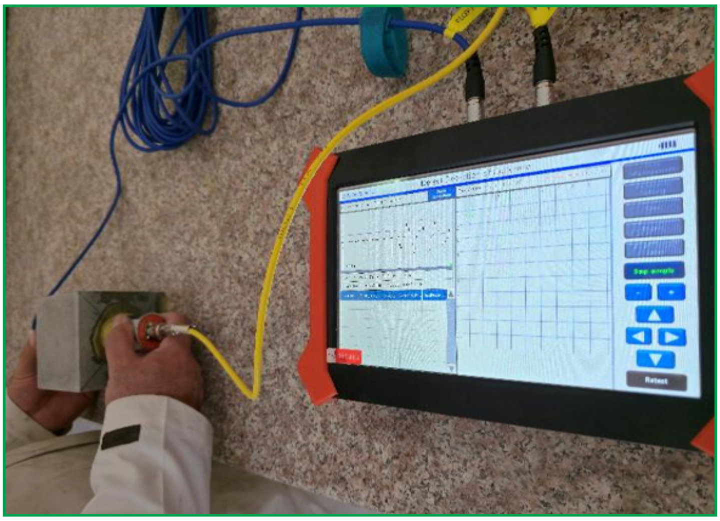

Figure 7. Process of testing concrete via Ultrasonic Pulse Velocity device

Results And Discussion

Brittleness Index

Brittleness is a material property that denotes the material’s propensity to fracture under tension with negligible deformation. Brittle materials are characterised by their high compressive strength and relatively low tensile strength, as well as their sensitivity to load-induced vibrations, feeble impact resistance, and limited flexibility. The degree of brittleness is determined by the ratio of compressive to split tensile strength; a higher ratio indicates a more brittle behaviour [26].

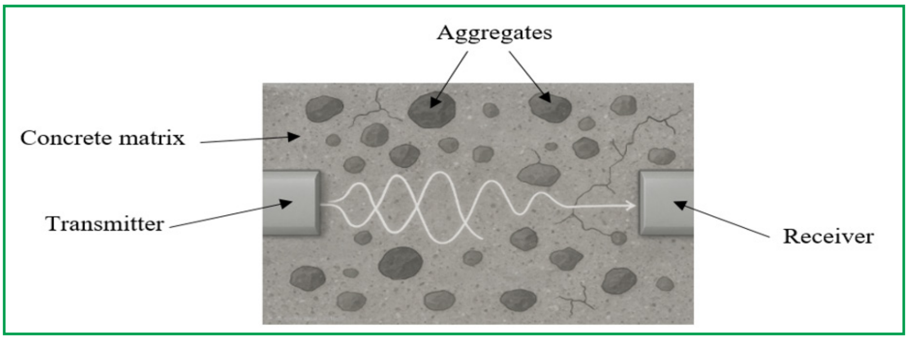

Figure 8. Schematic illustration of the ultrasonic wave transmission through concrete sample for ultrasonic pulse velocity test

Figure 9 illustrates the variation of BI values. The maximal BI value observed for concrete mixes with 3.5% MK content was a 15.37% increase in BI value relative to the control mix, as a result of the reduced compressive strength. This indicates that the brittleness of the VHSC mix has been reduced. Conversely, the BI increased by 1.63% and 3.07% for 4.5% and 5% micro MK, respectively, in comparison to the control blend, as a result of the decrease in compressive strength, beyond 4% micro MK content. The 4% replacement ratio in concrete mixtures containing micro MK resulted in the lowest BI value, with a minor reduction of 0.47% compared to the control mix. This underscores the importance of 4% micro MK in achieving an optimal combination of high compressive strength and low brittleness index.

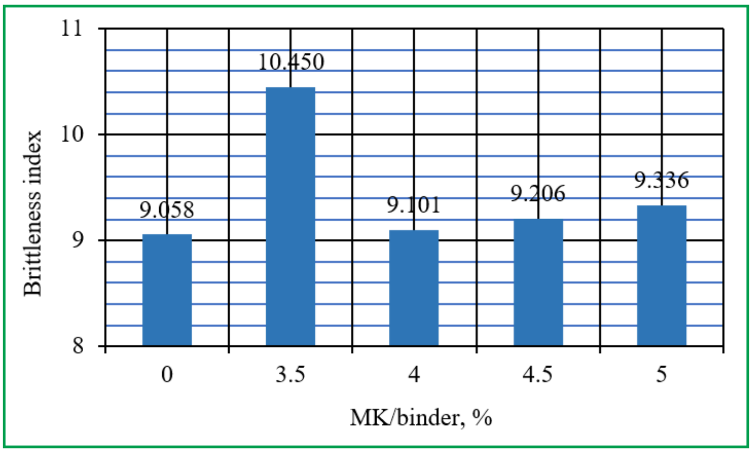

Figure 9. Effect of micro metakaolin replacement ratios on the brittleness index of very high strength concrete

Drying Shrinkage of Concrete

The discharge of water through the capillary openings within the concrete is the cause of drying shrinkage in hardened concrete. However, the drying shrinkage of VHSC mixes at 28 days was generally reduced by the incorporation of micro MK, with the exception of concrete mixes containing 5% of micro

MK, as illustrated in Figure 10. In comparison to the control mix, the drying shrinkage value decreased by 30.23%, 18.6%, and 4.65% at 28 days for MK replacement ratios of 3.5%, 4%, and 4.5%, respectively. Nevertheless, the drying shrinkage of mixtures containing 5% MK exhibited a minor increase of 6.98%, which was attributed to the pore size distribution and the arrangement of the pore structure.

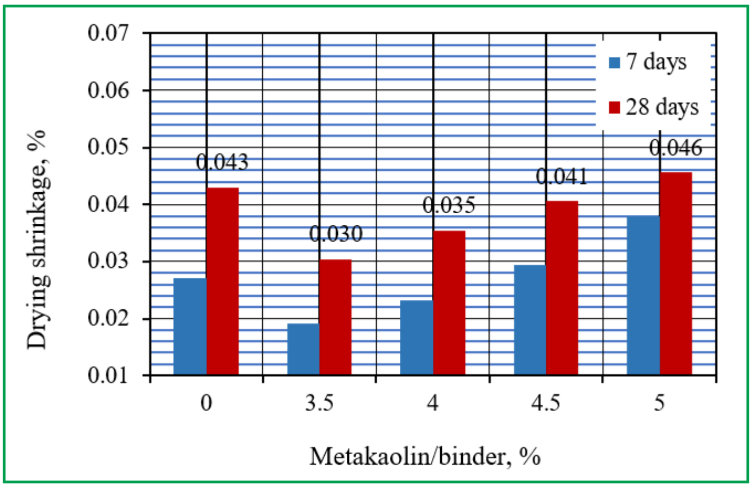

Figure 10. Effect of micro MK on the drying shrinkage of very high strength concrete

The primary reason for the decrease in drying shrinkage of hardened VHSC mixes at low replacements of MK was the reduced capillary porosity caused by ultra-fine micro-sized MK particles. These particles occupied micro voids, refined the pore structure of concrete, and reduced the number and size of pores within the cementitious matrix. Consequently, the drying shrinkage of VHSC mixes was reduced, as moisture loss and migration were limited during the drying process. Another explanation for the reduction of drying shrinkage in VHSC is the reduction in the quantity of cement from partial substitution with MK, where cement is the primary constituent responsible for eliciting drying shrinkage.

At higher replacements of micro MK, the minor increase in drying shrinkage of VHSC mix is due to the high surface area and high water demand of micro MK, which promote self-desiccation. This results in increased autogenous shrinkage and microcrack formation during the hydration process due to capillary tension and internal tensile stresses within the pore structure. In addition, the micro MK accelerates self-desiccation, which in turn facilitates vapour transmission during the drying phase, resulting in a higher drying shrinkage than the control mix. The findings of this study are in accordance with prior research, which reported a decrease in drying shrinkage [6,14].

Water Absorption of Hardened Concrete

As illustrated in Figure 11, the incorporation of micro MK into VHSC mixtures led to a decrease in water absorption by 18.65%, 19.04%, 20.6%, and 32.29% for concrete mixes containing 3.5%, 4%, 4.5%, and 5% micro MK, respectively. The pore structure of the concrete was refined concurrently by the progressive decrease in absorption, which was attributed to the filling effect of ultra-fine micro MK particles. This resulted in the interruption of capillary pores and a reduction in water retention within the voids of the concrete matrix by filling the micro voids and reducing the size of the voids. As a result, the water absorption in VHSC mixtures containing micro MK was substantially reduced as a result of the reduction in pore continuity and permeability. These findings are in accordance with prior research, including that conducted by [6,16,17].

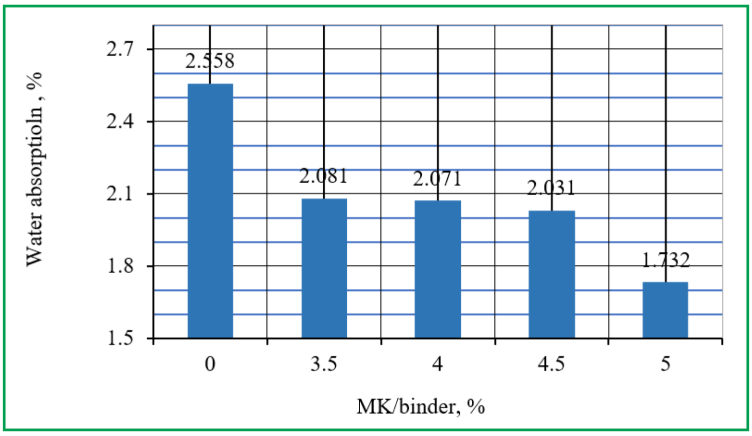

Figure 11. Effect of micro MK on the water absorption of very high strength concrete

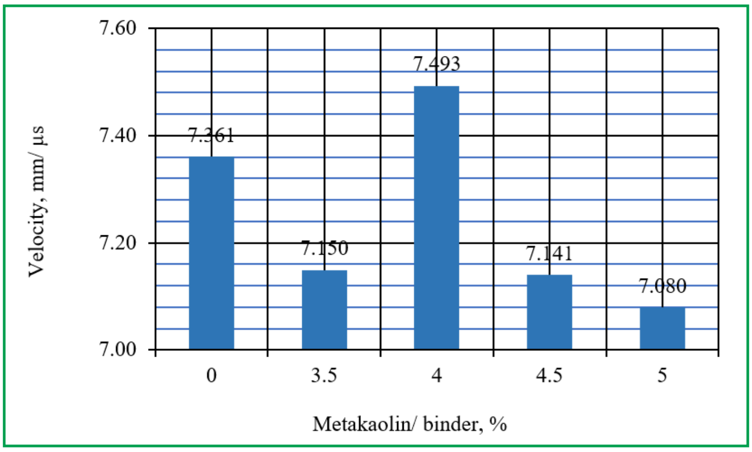

Figure 12. Effect of different replacement ratios of micro Metakaolin on the ultrasonic pulse velocity

Ultrasonic Pulse Velocity for Evaluation the Quality of Concrete

Figure 12 illustrates the impact of including micro MK on the UPV values of VHSC mixtures, with a 4% substitution of cement by micro MK yielding the highest UPV value of 7.493 mm/µs. The maximum UPV value of 4% MK is ascribed to the filling effect of ultra-fine micro-sized MK particles, which significantly improved the microstructure of concrete by reducing the distance between micro voids and filled these spaces. The transmission of ultrasonic waves in concrete is significantly influenced by the internal pore architecture and the level of density of the concrete. When the voids are extensively spread and unevenly distributed, the ultrasonic wavelength interacts with several contacts, resulting in scattering, attenuation, and a subsequent decrease in wavelength, thereby diminishing the UPV value. Conversely, when the void

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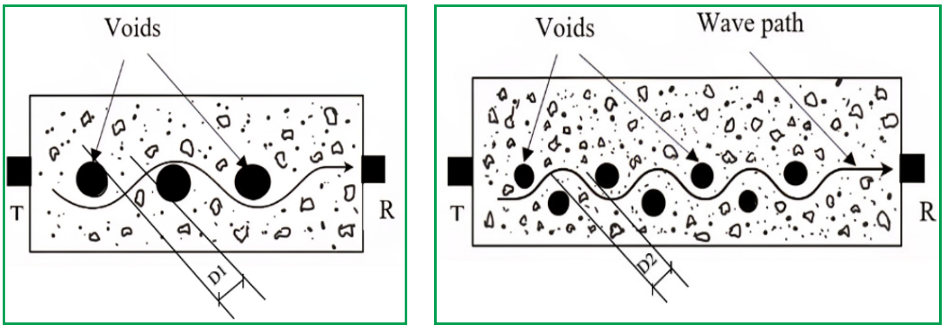
spacing diminishes and the pore structure becomes more uniformly distributed and refined, ultrasonic waves encounter fewer discontinuities, leading to a rise in ultrasonic wavelength and, subsequently, elevated UPV values. This behaviour unequivocally illustrates the robust link between microstructural refinement and ultrasonic wave transmission, as seen in Figure 13.



(a) (b)

Figure 13. Ultrasonic wave propagation through concrete samples for Ultrasonic pulse velocity test (a)Wave path through a large distance between voids (b) Wave path through a short distance between voids

In contrast, the concrete mix with 3.5% MK content exhibited a UPV value of 7.15 mm/μs, attributable to an inadequate quantity of micro MK to effectively fill the gaps and diminish the distance between them inside the concrete matrix. The diminished UPV value with increased micro MK replacement ratios is due to the dilution effect resulting from the excessive substitution of cement with micro MK, which primarily acts as fine aggregate rather than enhancing the binding effect, leading to a greater volume of voids and consequently reducing the compactness and quality of the concrete, thus lowering the UPV value. These results indicate a link between UPV value and both the compressive strength and drying shrinkage of the concrete, providing evidence for the maximum UPV value in the concrete with 4% micro MK content. In the realm of concrete quality evaluation, the UPV values above 4.5 mm/μs for all concrete mixtures, signifying exceptional quality, elevated density, uniformity, and an advanced microstructure (IS, 2018).

Determination of the Quality of Very High Strength Concrete by Incorporating Micro Metakaolin

The relationship between the compressive strength and split tensile strength of VHSC mixes is illustrated in Figure 14. The inclusion of micro MK generally increased the compressive and split tensile strengths of VHSC mixes, reaching an optimum value of 4%, resulting in maximum compressive and split tensile strengths of 111.31 MPa and 12.231 MPa, respectively. Furthermore, the compressive strength and split tensile strength exhibit a direct correlation for all micro MK replacement ratios. As the compressive strength increased, the split tensile strength also increased, with the exception of concrete mixes containing 3.5% micro MK, which experienced an increase in compressive strength but a decrease in split tensile strength.

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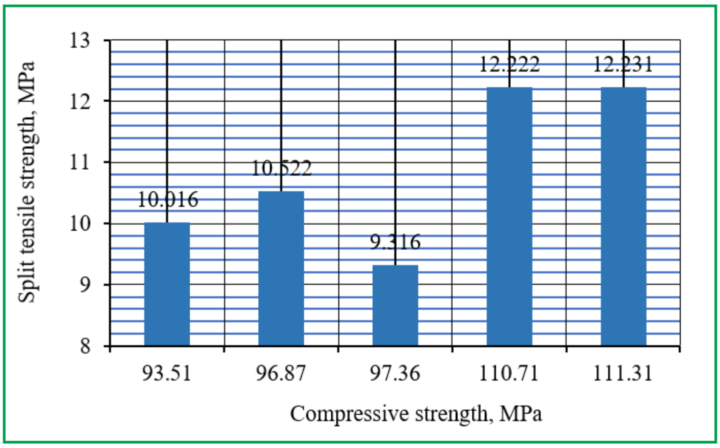

Figure 14. Relation between compressive strength and split tensile strength of very high strength concrete by incorporation of micro MK

The relationship between the compressive strength and UPV of VHSC mixtures is illustrated in Figure 15 by incorporating varying replacement ratios of micro MK. The maximum values of the UPV and compressive strength of VHSC were achieved at a 4% micro MK replacement ratio, with a 7.493 mm/µs and 111.31 MPa, respectively. Additionally, the compressive strength exhibits a consistent trend with respect to the UPV value for all micro MK replacement ratios. As the UPV value increases, the compressive strength also increases.

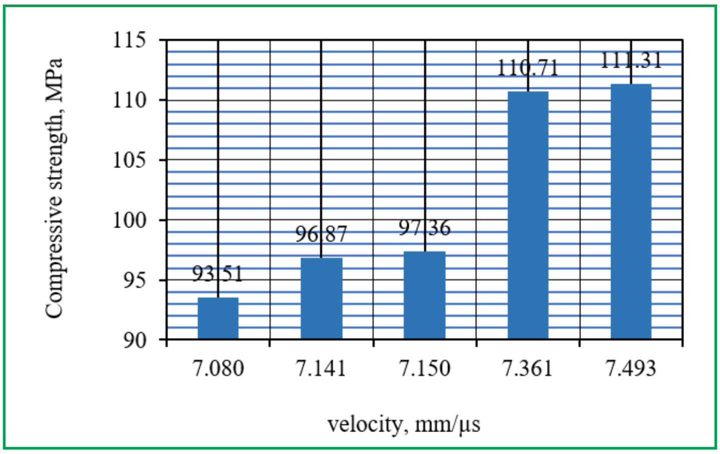

Figure 15. Relation between ultrasonic pulse velocity and compressive strength of very high strength concrete by incorporation of micro metakaolin

Conclusion

This study was conducted to enhance the properties of VHSC incorporating RAP aggregate, utilising micro-sized MK as a partial cement replacement at rates of 3.5%, 4%, 4.5%, and 5%. The study intended to evaluate the influence of micro MK on several categories of mechanical and non-mechanical properties of VHSC mixtures. The following conclusions can be derived from the experimental results:

- The optimal replacement percentage of micro MK in VHSC was 4%, at which point the brittleness index dropped to 9.101 due to the maximum

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split tensile strength value, decreasing the compressive strength which cause the improvement the velocity in UPV test to 7.493 mm/µs. It means that modified concrete has excellent quality concrete with minimal voids in their microstructure, as a result of improved compressive strength.

- The dry shrinkage of VHSC was reduced by the use of low replacement ratios of MK. Specifically, the drying shrinkage value was reduced by 18.6% at 4% micro MK due to the reduction of capillary porosity and the minimisation of both the size and quantity of micro pores, thereby reducing moisture loss during drying. In addition, the incorporating MK in to VHSC led to a 19.04% reduction in water absorption values due to the filling of microvoids and a decrease in their size, which diminished pore continuity and permeability via the micro MK particles.

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Conflicts of Interest

The authors declare no conflicts of interest

Author Contributions

Zhwan Anwar Noori: conceptualization, methodology, , original draft preparation. **Hardy Kamal Karim:** supervision. **Ferhad Rahim Karim:** supervision, writing-reviewing and editing.

Data Availability Statement

The data supporting this study’s findings are available on request from the corresponding author.

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